

## The experimental investigation on the geo-polymerization of water-based filtercake at the second interface of the oil-gas well

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### ABSTRACT

The quality of bond along the second interface of sandstone oil-gas well is of an utmost important in order to ensure long-term zonal isolation throughout the lifecycle of the well by preventing the potential leakage of hydrocarbon fluids. Attaining effective long-term zonal isolation for the lifetime of oil-gas wells depends on the sealing capacity at second interface (SI). Existence of untreated filtercake at SI acts as weak point of potential leakage of hydrocarbon. Different techniques were developed to improve the situation; however, it is still difficult to cleaned up completely by chemical washes and spacers. Therefore, it is imperious to develop a method that can make a filtercake which can provide an effective bonding between the formation and the cement sheath. The experimental work introduces a novel KN solution developed in the laboratory, simulated wellbore as sandstone formation under the wellbore simulated conditions.

The results revealed that the samples treated with KN solution had higher shear bond strength over untreated samples. The bond quality increased with the increase of curing time (3, 7, 14, and 28 days). The improvement was due to geopolymerization of remained water-based filtercake took place which led to formation of cementitious material such as Calcium-Silicate-Hydrate (C-S-H) and FTIR analysis revealed the formation of cementitious material (Calcium-Silicate-Hydrate (C-S-H)) and other geopolymers like Calcium-Aluminium-Silicate-Hydrate (C-A-S-H) and Sodium-Aluminium-Silicate-Hydrate (N-A-S-H) along the interface which filled the existed pores hence reducing porosity hence high strength of bond at high concentration to treated samples. The untreated samples were observed at low transmittance revealing the low content of inorganic geopolymers hence poor bond quality. Thick water-based filtercake films at second interface, which prevented the complete reaction of cement hydration between rock grains and cement slurry material to form a chemical binder at the interface.

This technique is of an utmost important as compared to the other means of improving shear bond strength. This chemical solution tends to convert the clay material into cementitious product (geopolymers) which will enhance the bonding strength. In addition, the formed products are high durable as it can withstand harsh environmental conditions such as higher elevated temperature and salinity condition hence leading to remedial cost reduction.

### 1. Introduction

In exploration and petroleum industry, the integrity of oil and gas wells is the key issue to ensuring the long-term productivity of the

hydrocarbon fluids (oil and gas). According to API-RP90 standard (API recommended practice 90, 2006) the integrity of oil and gas wells is the “application of technical, operational, and organizational solutions to reduce risk of uncontrolled release of formation fluids throughout the lifecycle of a well” (Summerhayes, 2011). It is an important attribute for

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### Nomenclature

API	American Petroleum Institute
Fig	Figure
FTIR	Fourier Transform Infrared Spectroscopy
HOAP	Hydroxyapatite
KN-I	Formulated chemical solution
KN-II	Formulated chemical solution
OBM	Oil Based Mud
SI	Second Interface
SWB	Simulated Wellbore
TIRDO	Tanzania Industrial Research and Development Organization
WBM	Water Based Mud
WCFI	Wellbore Cement-Formation Interface

oil and gas wells to resist structural damage and maintain operational performance.

The existence of filtercake material at the second interface (SI) (Fig. 1) is said to be one of the critical causes of weak bond as results hence leading to loss of wellbore integrity. In addition, when the well is subjected to reservoir temperature-pressure fluctuations and axial loads and during oil-gas production hence weakening the bond at the wellbore second interface (SI) (Plank et al., 2014; Chougnet et al., 2009; Foroushan et al., 2021). During the lifecycle of an oil and gas well, the loss of well integrity would cause serious and even disastrous consequences such as leaks and blowouts. The inspection report published by the Norwegian Petroleum Safety Authority on integrity revealed that 18% of injection and production wells are subject to integrity failure, and 7% of production wells have to be shut down (Li et al., 2020; Haga et al., 2009). The formation fluids tend to migrate through micro-annuli existing at interface to the wellhead leading to formation of sustained casing pressure (SCP) and contamination of nearby layer and aquifer (water bearing rock) and thus, there is raising of several issues including costs for remediation, production and abandonment as well as destruction of environment (water aquifer) whereby more than 26,000 oil-gas wells have been exhibiting have leakage associated problem (Lichinga et al., 2022; Federal register, 2010; Kiran et al., 2017).

The formation of strong bonding along the SI is essential for maintaining effective zonal isolation and wellbore integrity. Bonding associated with cement-interlayers include hydraulic bond (prevents fluid path), tensile bond (prevents debonding caused by tectonic lateral stresses) and shear bond strength (resists movement due to shear force)

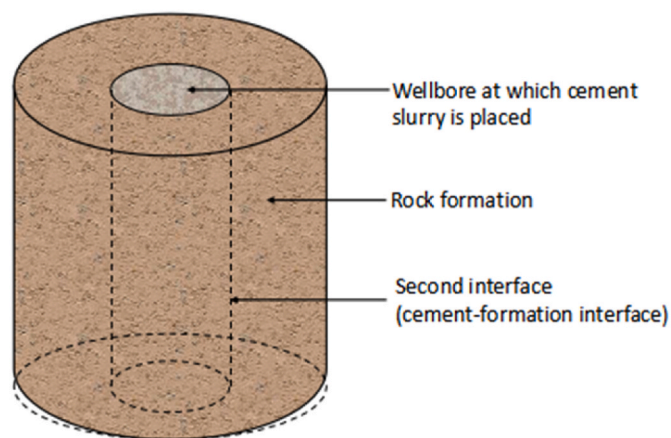


Fig. 1. Schematic of simulated wellbore (SWB) indicating the second interface (SI).

(Khalifeh and Saasen., 2020). However, the quality of shear bond strength between interfaces usually depends on the surface nature of wellbore wall and the reaction behavior of the materials (rock and cement materials) (Lichinga et al., 2020). Thus, critical analysis of bonding nature at the SI, is a criterion in order to lessen the possible leakage of formation fluids along the SI (Lavrov et al., 2015).

Drilling fluid is a material any liquid or gas which is circulated in the borehole which act as coolant, lubricant, cleaner, carrier of debris, formation pressure control, and enhance the function of the drill string and tools in the hole (Drilling mud, 2015). A number of drilling fluid be rheopectic or thixotropic have been used for a number of years. The fluids include Synthetic-Based Muds, Oil-Based Mud, Water-Based Mud, and Pneumatic or Air Muds as due to economic factor, WBMs are the most commonly used whereby the nature of formation to be drilled influences the choice of drilling mud system to be used (Broni-Bediako and Amorin, 2019; Gandhi and Sarkar, 2016; Zoveidavianpoor et al et al., 2016). In addition, new technology on water-based mud have recently emerged whereby Jiang et al. (2021) developed a Novel Water-based drilling and completion fluid technology to improve wellbore quality during drilling and protect unconventional reservoirs as well as being an ecologically friendly drilling fluid. Most of the oil-gas wells have been drilled by water-based mud, however, oil-based mud has critically been applied in the areas where WBMs are unable to tolerate certain conditions. Such harsh conditions include Deep depth, high temperature, and directional wells are drilled with OBMs whereby WBMs tend to undergo thermal degradation or increase of viscosity (Simpson, 2012).

In oil and gas industry, a filtercake is an impermeable, thin layer formed by the filtration of drilling fluid during the oil-gas well drilling operations (Carrasquilla et al., 2012; Lichinga et al., 2020), whereby the size of the filtercake normally ranges from 2 to 5 mm. However, the typical thickness of oil-based filtercake ranges from 0.5 to 1.5 mm depending on the type of formation (Ladva et al., 2005). This layer is of utmost important when the borehole stability is required in order to protect the formation and wellbore from damage. However, the filtercake becomes an undesirable weakness to provide a good zonal isolation (Hao et al., 2016; Gu and Wenzheng, 2010). The existence of filtercake at the interface leads to failure of cement to form a strong bond with the formation (Opedal et al., 2014). A number of studies have been conducted to indicate the negative effect of filtercake (Lichinga et al., 2022), while different researchers presented various technical approaches on filtercake removal along the oil-gas wellbore, Table 1.

The interfacial transition zone (ITZ) is also known as SI which is characterized by increased porosity created by wall effect due to the differences in size between cement and formation grains (Brady et al., 2000; Hewlett et al., 2004). Typical cement grain size is in the range of 5–60  $\mu\text{m}$  while sand grain size is in the range of 70–200  $\mu\text{m}$  (Lichinga et al., 2022). In civil engineering, this zone refers to a water-cement ratio gradient in fresh concrete that develops around the aggregate particles during casting, resulting in a different microstructure of the surrounding hydrated cement paste.

A number of studies (Soares et al., 2017; Chatterjee and Wasan, 1998; Touhami et al., 1998; Rudin and Wasan, T1992; Agbasimalo and Radonjic, 2014; Anugrah et al., 2014; Chan and Yen, 1981) have reported different chemical techniques for the filtercake removal (Oxidizing Breakers (Peroxides) Hydrochloric Acid (HCl), Chelating Agents) which have been employed to remove the filtercake in the wellbore prior to cementing job in order to attain strong bond along the SI. However, there have been some shortcomings to such methods which could influencing well productivity, for instance, Enzymes may poorly destroying starch and xanthan polymers under certain harsh environment (geological heterogeneous with high permeability streaks), hence resulting to high consumption of chemical fluid. In addition, enzyme has a difficult time dissolving calcium carbonate particle. Oxidants and acidic materials are very reactive, that may improperly clean the filtercake residue, as mineral contents such as illites in sandstone reservoir have high affinity to acids, hence leading to formation damage near the

**Table 1**  
Different studies on filtercake removal techniques.

Author	Title	Approach	Contribution
Rui et al. (2021)	A Novel Experimental Method for Mudcake Removal Efficiency of Spacer	Experimental	Provision of the method that can evaluate the flushing efficiency quantitatively and provide guidance for designing of spacer
Opedal et al. (2014)	Experimental Study on the Cement-Formation Bonding	Experimental	Gives insights on the effects of drilling fluid in bonding strength at different rock formation
Griffith and Osisanya, 1995	Thickness optimization of drilling fluid filter cakes for cement slurry filtrate control and long-term zonal isolation.	Experimental	It suggests that complete removal of the filter cake provides the greatest resistance to fluid communication in most of the cases studied.
Barbosa et al. (2000)	Synthesis and characterization of materials based on inorganic polymers of alumina and silica: sodium polysialate polymers.	Experimental	The synthesis of Inorganic polymers based on alumina and silica polysialate units from dehydroxylated aluminosilicate clay (metakaolinite) under highly alkaline environment.
Zain and Sharma, 1999	Cleanup of Wall-Building Filter Cakes	Experimental	Presentation of different methods used to cleanup the wall building filtercake
Rostami and Nasr-El-Din, 2010	A New Technology for Filter Cake Removal	Experimental	Introduction of a novel cleaning approach for mud-cake using a self-destructing water-based fluid to drill long horizontal and maximum reservoir contact (MRC) wells, which results in higher productivity
Lichinga et al., 2022	A novel alkali-surfactant for optimization of filtercake removal in oil-gas well	Experimental	Introduction of a novel Alkali-Surfactant (KV-MA) solution developed to optimize the filtercake removal of oil-gas wellbore

wellbore (Yong et al., 2007).

Advancements in well cementing and wellbore stability are not comprehensive enough. Several contributions have been given by different authors on quantifying the impact of filtercake physically and chemically on the bonding quality at the wellbore first and second interfaces (casing-cement-formation interfaces).

Attaining effective long-term zonal isolation for the lifetime of oil-gas wells depends on the sealing capacity of the second interface (SI). Apart from various techniques developed to remove the filtercake, it is still difficult to be cleaned up completely by chemical washes and spacers (Perná et al., 2017, 2020). Therefore, it is imperious to develop a method that can make a filtercake which can provide an effective bonding between the formation and the cement sheath. The related studies conducted by different researchers are presented in Table 2. These materials are also related geopolymers with cementitious properties to improve the bonding quality.

The geopolymer is an inorganic polymeric material formed through

the reaction between aluminosilicate sources and highly alkaline silicate solution, followed by curing at ambient or slightly higher temperature (Davidovits J. 2002; Liew et al., 2016; Reddy et al., 2016). The formation process is termed as geopolymerization reaction. It involves chemical reaction that transforms partially or totally amorphous aluminosilicates into three-dimensional polymeric networks. The geopolymerization reaction is an exothermic. Under strong alkaline medium, the aluminosilicate sources dissolve into SiO<sub>4</sub> and AlO<sub>4</sub> tetrahedral units which later on participate in the polycondensation process (Davidovits and Metha, 1994; Yip et al., 2005; Davidovits, 2015; Provis, 2018). Moreover, the heat resistance of geopolymers makes it possible to increase the resistance of concrete structures at high temperatures.

The main purpose of this study is to enhance the bonding strength along the SI interface of the oil-gas well drilled with Water-Based Mud (WBM) by converting the remained filtercake to make the geopolymer materials that have cementitious properties hence improving the bond quality along the interface to ensure the long-term zonal isolation. The alkali activated reactions have been employed to convert the water-based filtercake whose main composition is bentonite clay. The performances of this novel method were measured by calculating shear bonding strength and hydraulic bonding strength tests. FTIR analysis was used to illustrate the differences of microstructure between the treated and untreated filtercake. This method can enhance the shear bonding strength of the cement-formation interface (second interface) significantly.

## 2. Methods and materials

### 2.1. Materials

The materials for experiment were taken from different areas including oilfields and some were prepared in the laboratory. The materials used were Water-Based Mud (1.32 g/cm<sup>3</sup>) from Daniudi Oilfield, API Class G Oil-well Cement (from Jiahua cement plant), NaOH, CaCl<sub>2</sub>, Na<sub>2</sub>SiO<sub>3</sub>, Alkyl glycosides (1214 and 0810), 3 wt% fluid loss agent, 0.1% defoamer (from Shandong Obot Petroleum Technology Co., Ltd. In China), 0.3 wt% retarder (from Qinshui basin in china), and 44% tap water. Simulated wellbore (SWB) with porosity (17.3%) and permeability (140 × 10<sup>-3</sup> μm<sup>2</sup>) were made in the laboratory by using available quartz sand, cement and water.

### 2.2. Equipments

The following equipment were used to accomplish this study, these include ZNN-D6 six kinds of speed rotary viscometer (Qingdao hisense optical communication co., ltd, China), JJ-I Accurate Strengthen Electronic Stirrer (Changzhou Guo Hua Electrical Equipment Co., Ltd. In China), Accurate Strengthen Electronic Stirrer (Changzhou Guo Hua Electrical Equipment Co., Ltd. In China), The liquid density meter – YM-2 (Qingdao Hisense optical communication co LTD, specialized factory), Atmospheric Pressure Curing Chamber, TG-1280 (Shenyang tiger petroleum equipment manufacturing co LTD), WDW Universal testing machine (Jinan Hensgrand Instrument Co., Ltd China), TG-3060 A Constant Speed Agitator (Shenyang tiger petroleum equipment manufacturing co LTD), 600 Series Servo-hydraulic Test Machines (Novatq Pte Ltd, Singapore) were used in the experiment. ( ).

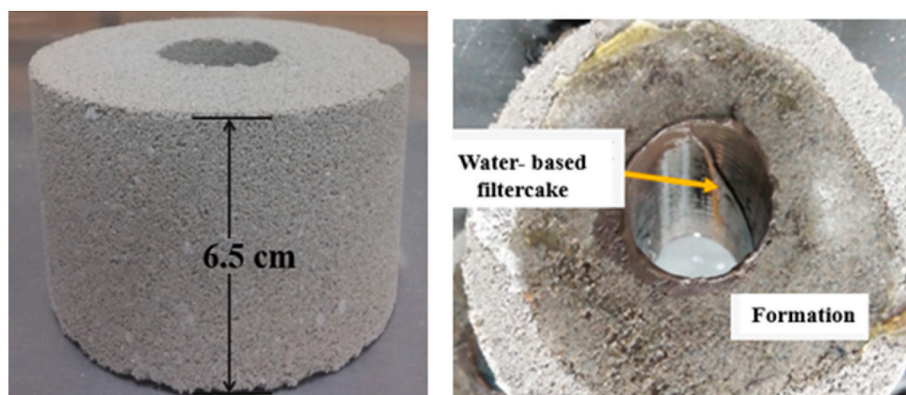
### 2.3. Methodology

#### 2.3.1. Simulation of water-based filtercake and simulated wellbore

The simulated wellbores (SWBs) (Fig. 2 Left). Were prepared as according to Gu et al. (2015) and Lichinga et al. (2019). The simulation of water-based filtercake into the SWBs was performed by using a glass plate put at their bottom. The water-based Mud (WBM) was poured into the SWB uniformly to reach the top of SWB and left for about 4 h under

**Table 2**  
Summary of the recent trends in well integrity.

Authors (S)	Title	Approach	Contributions
Bu et al. (2020)	Utilization of metakaolin-based geopolymer as a mud-cake solidification agent to enhance the bonding strength of oil well cement–formation interface.	Experimental	Enhancement of shear bonding strength and hydraulic bonding strength of the cement– formation interface significantly.
Hao et al. (2016)	Comparative study on cementation of cement-mudcake interface with and without mud-cake-solidification-agents application in oil & gas wells	Experimental	It provides a new insight into the interfacial-transition zone, which was efficiently sealed by Mudcake Solidification Agents
Cowan et al. (1992)	Conversion of drilling fluids to cements with blast furnace Slag: performance properties and applications for well cementing	Experimental	provides the proper combination of fluid and solid properties, simplicity of design and application, improved zonal isolation, and broad applicability to bring drilling fluid solidification technology into widespread use
Gu and Wenzheng, 2010	Experiments on integrated solidification and cementation of the cement-formation interface using the Mud Cake to Agglomerated Cake (MTA) method	Experimental	Provides alternative method to improve the bonding quality at the cement-formation interface hence enhancing the zonal isolation of shallow wells.
Bai et al. (2021)	Enhancing the Solidification Between Mud Cake and Wall Rock for Cementing Applications: Experimental Investigation and Mechanisms	Experimental	Developed methods to enhance the cementation strength of the mud cake and rock interface.
Achang and Radonjic (2021)	Adding olivine micro particles to Portland cement-based wellbore cement slurry as a sacrificial material: A quest for the solution in mitigating corrosion of wellbore cement. Cement and Concrete Composites	Experimental	Olivine as additive serves as a micro-sacrificial material within hydrated Portland cement exposed to low pH fluids to form carbonates, mitigating potential CSH and CH leaching, enhancing wellbore cement resistance to leakage
Ritchie et al. (2019)	An Unconventional Study on the Bond Strength of the Casing/ Cement Interface and the Benefits of Nanoparticle Additives	Experimental	If implemented correctly, the addition of nanoparticle barite or magnetite could yield a significantly improved seal to primary or P&A cement even at elevated pressures
Wu et al. (2022)	Characterization of the mud displacement in an enlarged wellbore: An integrated rock-fluid model	Computational Fluid Dynamic (CFD)	Geopolymer has a better sealing performance and can resist more water-based mud (WBM) contaminations than neat class G cement
Duguid et al. (2011)	Degradation of cement at the reservoir/cement interface from exposure to carbonated brine	Experimental and Numerical model	Cements in sandstone are more vulnerable to degradation (alteration that damages the cements' strength of zonal isolation properties, e.g. permeability) than cements surrounded by limestone.
Massion et al. (2022)	Geomimicry-Inspired Micro-Nano Concrete as Subsurface Hydraulic Barrier Materials: Learning from Shale Rocks as Best Geological Seals	Experimental	Micro-nano aggregates (Olivine, Zeolite and Graphene) contribute to the enhanced performance of the cement matrix as all can have positive impact on cement properties that enable effective and resilient zonal isolation
Vissa and Radonjic (2022)	Restoration of Reservoir Caprock: Long-Term Wellbore Sealing and Abandonment through Zeolite Enhanced Cement	Experimental	Additional of Zeolite in class H cement leads to maintenance of chemical stability of cement matrix even after hydration process hence increasing bonding strength as well
Didier et al. (2018)	The Wellbore Cement Additive, Gilsonite, a Solution to Leaky Gas Wells	Experimental	The exposure to Natural Gas resulted in a decrease of the permeability of the Gilsonite-enriched cores, which implicates potential gas migration mitigation.
Li and Radonjic (2019)	Mechanism for the promotional effect of a novel solidifiable spacer fluid system on the cementation quality of cement sheath/ formation interface	Experimental	The presence of acid environment and drilling fluid cake would produce leaching of elements and fracture formation, which would result in compromised zonal isolation and wellbore integrity issues.



**Fig. 2.** Simulated wellbore (Left) and Simulation of water-based filtercake in a SWB (Right).

atmospheric conditions before spilled out. The weak filtercake was removed to get a thickness of 0.7 mm (Fig. 2 Right).

### 2.3.2. Preparation of KN solution

The formulated KN solution was split into two parts; KN-I and KN-II, where. KN-I was prepared by mixing of 0.9 wt% Alkyl Polyglucoside (APG-1216) and 14 wt%  $\text{CaCl}_2$  and water to produce a

uniform solution with clouding while KN solution was prepared by mixing 0.9 wt% Alkyl Polyglucoside (APG-0810), 12 wt% NaOH, 15 wt%  $\text{Na}_2\text{SiO}_3$ .

### 2.3.3. Application of KN solution

The chemical solutions (KN-I and KN-II) were placed into two beakers then heated to 55–65 °C in a water bath. Then KN-I solution was

poured into the simulated wellbores to be treated and allowed to imingle with the filtercake materials for 135 s before poured off (Fig. 3a). Then KN-II solution from water bath was injected into the borehole and left for 150 s (Fig. 3b). After pouring off the chemical the SWB were ready cement slurry placement.

#### 2.3.4. Cement slurry placement

Preparation of cement slurry used for this study, 600 g of class G oil well cement was added to 261 ml of fresh water to make a water-to-cement ratio of 0.44 and blended using Model TG-3060 A Constant Speed Agitator for blending cement slurry in compliance with the API's 10A/B at high speed for 35 s. The cement additives identified in this study include Fluid loss agent, Defoamer, and Retarder as shown in Table 3. The slurry was then injected into the SWB blank samples without KN solution treatment Fig. 4a, and SWB samples treated with KN solution Fig. 4b. Cement slurry was gently stirred to make desirable consistency.

The simulated wellbores were then put into Atmospheric Pressure Curing Chamber containing hot water Fig. 5, and left for 3, 7, 15 and 28 days. The temperature of water was kept changing from 75 to 81 °C to imitate the wellbore conditions.

#### 2.3.5. Bonding quality test

The bond strength of both treated and untreated samples was measured by using the WDW Universal testing machine (Fig. 6). equation (1) below was used to compute bond strength of the samples.

$$P = \frac{10F}{\pi hD} \quad (1)$$

$D$ , SWB inward diameter, (cm).  $P$ , shear force, (KN).  $h$ , bond strength, (MPa).  $F$ , SWB height, (cm). In this study ' $D$ ' was 3.3 cm same to all samples, as the same iron rod was used to simulate the wellbore diameter.

#### 2.3.6. Percentage increase of bond strength (PI)

The percentage increase for the measured bond strength between which have been treated by KN solution and that which have not been treated with the KN solution sample amount of bond strength was calculated as percentage increase by using equation (2) below.

$$PI = \frac{\text{Increase}}{\text{original value}} \times 100\% \quad (2)$$

Where,  $\text{increase} = \text{new value} - \text{original value}$ .

#### 2.3.7. Material characterization

The Fourier-transform infrared spectroscopy (FT-IR) was used to analyze the material existed at the interface of the blank and WBM treated simulated wellbores. FT-IR spectra of the pellets were obtained using Nicolet 6700 FTIR between  $400 \text{ cm}^{-1}$  and  $4000 \text{ cm}^{-1}$ . The background spectrum was collected using a pure KBr pellet and the spectra of

**Table 3**

The Class G Cement slurry formulation design.

Materials	Quantity (g)
Class G Cement	600
Water	261
Fluid loss agent	3
Defoamer	1
Retarder	3

samples were corrected with a linear baseline.

### 3. Results and discussion

#### 3.1. Bonding quality test

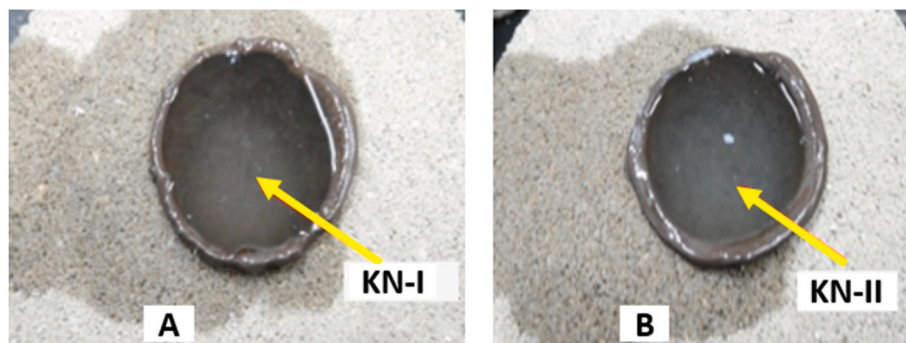
The test was performed for treated and untreated samples and the results are shown in Tables 4 and 5 below.

The bonding strength of the SWB samples at SI was measured and analyzed. From the experimental results it was observed the samples which were treated with KN solution indicated higher bond strength over the untreated samples. The strength was generally increasing with curing time as for the treated samples, 0.150, 0.318, 0.471 and 0.522 MPa for 3, 7, 14, and 28 days correspondingly while for the untreated samples had the strength of 0.050, 0.070, 0.81 and 0.100 MPa for 3, 7, 14, and 28 days respectively (Fig. 7). The Improvement of bond strength to the treated sample was due to filtercake solidification as a result of formation of cementitious material such as Calcium-Silicate-Hydrate (C-S-H) and other geopolymers like Calcium-Aluminium-Silicate-Hydrate (C-A-S-H) and Sodium-Aluminium-Silicate-Hydrate (N-A-S-H) along the interface which filled the existed pores hence reducing porosity while increasing the strength of the bond (Fig. 8b) (Wu et al., 2022). The lower/poor bond strength for the untreated samples is due to existence of untreated, thick water-based filtercake films at SI (Fig. 8a) which prevented the complete hydration process between rock grains and cement slurry components to make chemical binder of cement slurry material and formation.

#### 3.2. Percentage increase (PI) of bonding strength

The percentage increase of bond strength between the treated and untreated (blank) samples was calculated by using equation (2) above and the results are presented in Table 6 below.

The bonding strength percentage increase of the tested samples between treated and untreated were 241%, 397%, 441% and 417.0% for 3, 7, 14, and 28 days respectively, indicating that the water-based filtercake solidification techniques had significantly enhanced the bonding strength of SI by increasing rates above 241%.



**Fig. 3.** Injection of KN-I (A) and KN-II (B) solution into SWBs.

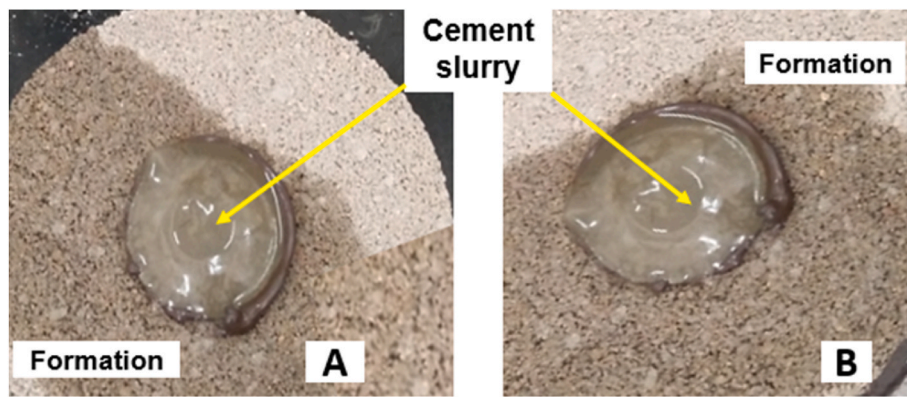


Fig. 4. The injection of cement slurry into the Blank Sample (A) and KN Treated Sample (B).



Fig. 5. The atmospheric pressure curing chamber.

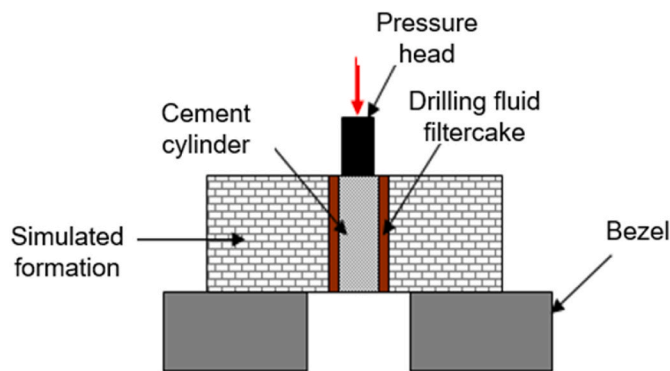


Fig. 6. Schematic diagram for shear bond strength measurement.

Table 4  
Bond strength test results for the untreated SWBs samples.

Curing Temperature (°C)	Filtercake Thickness (mm)	Curing period (Days)	SWB (MPa)			Average SWB (MPa)
			1	2	3	
81	0.6	3	0.150	0.138	0.145	0.044
		7	0.055	0.069	0.067	0.064
		14	0.084	0.087	0.090	0.087
		28	0.101	0.102	0.101	0.101

### 3.3. Material characterization

The FTIR analysis was conducted by using powdered material from the SI of the cured samples so as to identify the existing materials that influenced the bond strength as shown in IR Spectrum below (Fig. 9).

Table 5  
Shear bond strengths test results for the Treated SWBs samples.

Curing Temperature (°C)	Filtercake Thickness (mm)	Curing period (Days)	SWB (MPa)			Average SWB (MPa)
			1	2	3	
81	0.6	3	0.148	0.146	0.157	0.150
		7	0.398	0.279	0.278	0.318
		14	0.361	0.493	0.560	0.471
		28	0.588	0.424	0.554	0.522

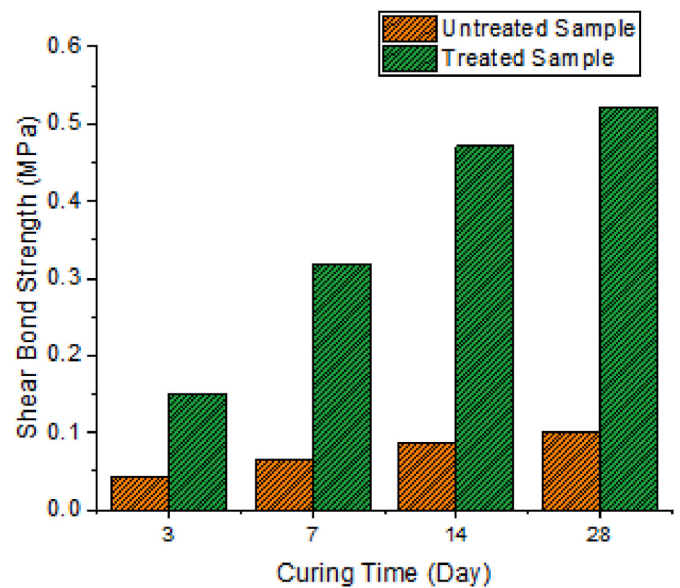


Fig. 7. Shear bond strengths for Untreated and Treated samples.

The sample taken from SI of the 15 days cured were analyzed using FTIR analysis. In the sample spectrum, the bands at 3622, 3440 and 3296  $\text{cm}^{-1}$  were attributed to the stretching vibration of structural hydroxyls (Si-OH, Al-OH) and the physical absorption of water (H-OH), respectively (Pan et al., 2008; Wolters and Emmerich, 2007) with a corresponding deformation band (H-O-H) at 1636  $\text{cm}^{-1}$  (Oztrk et al., 2008). The medium sharp bands at 1430 and 1428  $\text{cm}^{-1}$  for both treated and untreated samples were attributable to an asymmetric extending vibration of  $\text{CO}_3^{2-}$  due to assigned  $\text{CaCO}_3$ . The observed significant band 470  $\text{cm}^{-1}$  which was designated to the zeolitic framework having tetrahedral structural elements and mostly consisted of  $\text{SiO}_4$  (Mei et al., 2016).

The high-pitched bands at 990–1018  $\text{cm}^{-1}$ , indicates the presence of

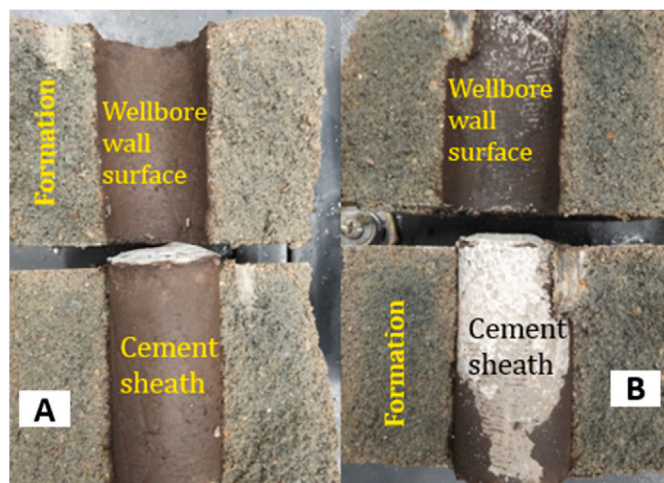


Fig. 8. The SI of untreated simulated wellbore samples (a) and treated simulated wellbore sample (b).

Table 6  
The percentage increase (%) of bond strength.

Curing temperature (°C)	Filtercake thickness (mm)	Curing period (days)	Bond Strength (MPa)	Percentage Increase (%)	
81	0.6		Untreated samples		
		3	0.044	0.150	241
		7	0.064	0.318	397
		15	0.087	0.471	441
		30	0.101	0.522	417
			Treated samples		

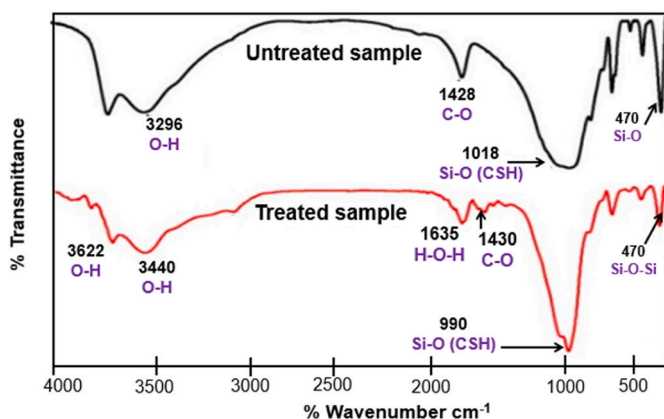


Fig. 9. The SI The full range of FTIR spectra of SI material sample obtained from treated and untreated samples.

Si-O-(Al, Si in tetrahedra coordination) asymmetric stretching development of C-S-H (Król et al., 2016). In addition, the geopolymer structure was clearly observed the same range of band where, it showed an amorphization of aluminate mineral structures in bentonite clay (Govindarajan1 and Govindarajan and Gopalakrishnan, 2011; Fernández-Jiménez and Palomo, 2005). The low value transmittance for the KN treated samples indicated that there were more cementitious materials existing at the SI which led to enhancement of high sealing quality as compared to the untreated samples.

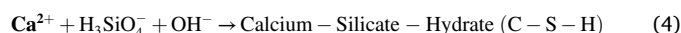
### 3.4. Reaction mechanism of KN solution

The KN solution is a formulated filtercake converter which divided

into two parts, KN-I and KN-II basing on their composition as described below.

#### 3.4.1. KN-I solution

This solution consists of Ca<sup>2+</sup> and Cl<sup>-</sup> ions and non-ionic surfactant (Alkyl polyglycosides). The additional of Ca<sup>2+</sup> ions (equation (3)) as divalent from salt (CaCl<sub>2</sub>) to the system lead to slow down the gelation time through charge screening as result quickening gelling kinetics (Jurinak and Summers, 1991). They also facilitate the formation of chemical binder (C-H-S) by reacting with other silicates ions released from KN-II Solution and cement slurry material to form a chemical binder (C-S-H) at SI (equation (4)). The oily part material was eliminated by nonionic surfactant whereby the water-based filtercake becoming water-wet which later ease the hydration process.



#### 3.4.2. KN-II solution

This solution is a blend of sodium hydroxide (NaOH), and Sodium Silicate (Na<sub>2</sub>SiO<sub>3</sub>) and Nonionic Surfactant (APG-0810). NaOH works as alkaline activator to disintegrate Si-O-Si and Al-O-Si bonds into monomeric ions (H<sub>3</sub>SiO<sub>4</sub><sup>-</sup>, H<sub>3</sub>AlO<sub>4</sub><sup>-</sup>). Additional of Na<sub>2</sub>SiO<sub>3</sub> was done to facilitate the geopolymerization reaction at the SI in presence of water-based filtercake (montmorillonite ((M<sub>v</sub>+•H<sub>2</sub>O) (Al<sup>3+</sup>+2-yMg<sup>2+</sup>) Si<sub>4</sub><sup>4+</sup> O<sub>10</sub>(OH)<sub>2</sub>)). As source of aluminosilicate material which later lead to formation of a chemical binder (Calcium-Silicate-Hydrate) hence strengthening the bond at SI. The nonionic surfactant helped to evenly spread the sodium silicate over the water-based filtercake. On the other hand, an alkali is applied with surface-active agent to stabilize the foaming liquid. This makes it as an important chemical in the oil industry to be applied in enhance oil recovery (Wang et al., 2022) and oil based mudcake removal (Lichinga et al., 2022).

#### 3.4.3. Partaking chemical reactions

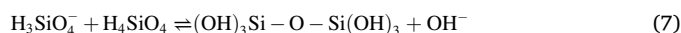
- Hydrolysis of sodium silicate

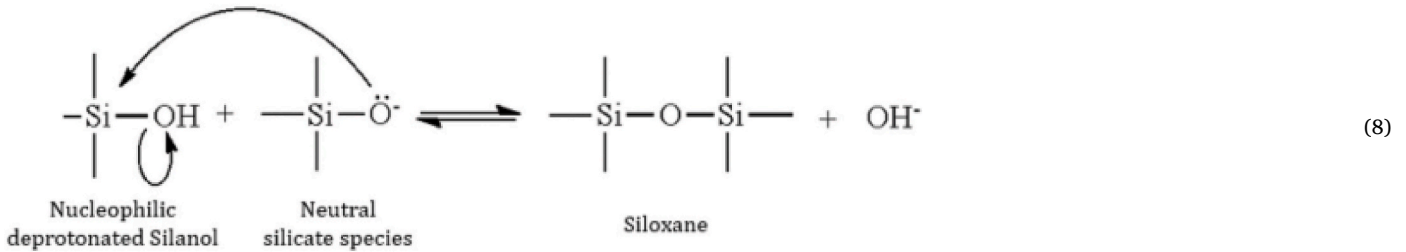
The utilized water-based mud consisted of bentonite clay (phyllosilicates group - Smectite) as base material. It contains montmorillonite which has high base affinity to sodium silicate in contrast to other minerals like kaolinite. This reaction starts with the hydrolysis of the sodium silicate to produce hydrated silica (H<sub>3</sub>SiO<sub>4</sub><sup>-</sup>) known as silanol (equation (5)) whereby as the pH decreases, the silicic acid is produced (equation (6)).



- Condensation of Silanol from hydrolysis of Na<sub>2</sub>SiO<sub>3</sub>

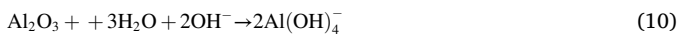
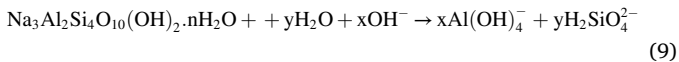
As at that high pH (10–12), H<sub>2</sub>SiO<sub>4</sub><sup>2-</sup> and H<sub>3</sub>SiO<sub>4</sub><sup>-</sup> species are mainly dominating the system, while at low pH (2–4) H<sub>3</sub>SiO<sub>4</sub><sup>2-</sup> and H<sub>4</sub>SiO<sub>4</sub> are main dominant ions. Thus, the condensation reaction of dominating material under alkaline condition is given by equation (7) below. Since it is condensation reaction, liberation of water is indicated by hydroxyl ion (OH<sup>-</sup>). The dimer (siloxane) is produced by nucleophilic attack reaction of two silanol groups linked by oxygen atom (Iler, 1979). As shown in equation (8).





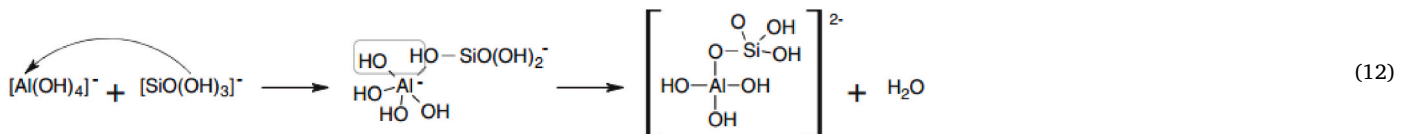
- Dissolution, and Hydrolysis of  $\text{Al}^{3+}$  and  $\text{Si}^{4+}$  from water-based filtercake

At high alkaline condition, the water-based filtercake tends to breakdown by the existing Al-O-Si and Si-O-Si bonds from aluminosilicate precursor (montmorillonite) (equation (9)) to make silicate ( $\text{AlO}_4$ ) and aluminate ( $\text{AlO}_4$ ) tetrahedral oligomers (Gartner and Macphree, 2011) through hydrolysis as shown in equations (10) and (11) below.



- Condensation of  $\text{Al}^{3+}$  and  $\text{Si}^{4+}$  from water-based filtercake

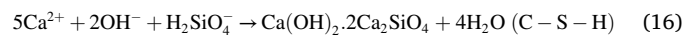
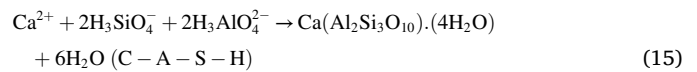
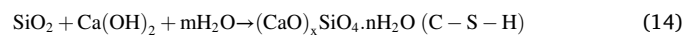
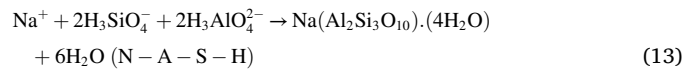
The nucleophilic substitution reaction between ( $\text{AlO}_4$ ) and aluminate ( $\text{AlO}_4$ ) species took place to form new aluminosilicate (intermediate complex) by the attraction between one of the OH groups (Wolters and Emmerich, 2007). As shown in equation (12) below.



- Polycondensation process

This is the process of gelation at which under acidic condition, a poor crystalline gel material was reported to form as compared to under alkaline condition which has been widely used method (Guo et al., 2016). The  $\text{Ca}^{2+}$ , from KN-I and  $\text{H}_4\text{SiO}_4$  ions from the KN-II Solution, and their corresponding species with  $\text{Ca}^{2+}$  and  $\text{OH}^-$  from the cement slurry to form C-S-H gels. Monomeric species like  $\text{H}_3\text{SiO}_4^-$  and  $\text{H}_3\text{AlO}_4^-$  from the alkali-activated reaction a nucleation and development process to form crystalline zeolite ( $\text{Al}_x\text{Si}_{(2-x)}\text{O}_4$ ) phases or low calcium alkali aluminosilicate (sodium aluminosilicate) hydrate (N-A-S-H) gel framework which is an amorphous matrix with higher strength cementitious properties participating in filling up micro-fractures at SI (equations (13)–(16)) (Davidovits and Metha, 1994; Yip et al., 2005). Zeolite materials have also been used as an additive for improvement of cement

strength properties so as to prevent the potential wellbore leakage (Radonjic and Vissa 2020; 2022).



#### 4. Conclusions

1. The existence of untreated water-based filtercake at the SI of simulated wellbores acted as a weak point that hence causing poor formation of bond between cement slurry material with the rock grains.
2. The water-based filtercake modification techniques had significantly enhanced the bonding strength of SI by increasing rates above 241%. The bonding strength of treated samples cured for 3, 7, 14, and 28 days were observed to be higher than that of untreated samples which were cured for same curing time.

3. KN solution as filtercake modification which led to formation of cementitious material at SI. Materials such as Calcium-Silicate-Hydrate (C-S-H).
4. FTIR analysis revealed the formation of cementitious material (Calcium-Silicate-Hydrate (C-S-H)) and other geopolymers like Calcium-Aluminium-Silicate-Hydrate (C-A-S-H) and Sodium-Aluminium-Silicate-Hydrate (N-A-S-H)) along the interface which filled the existed pores hence reducing porosity hence high strength of bond at high concentration to treated samples.
5. The low transmittance value for the KN treated samples revealed that there were more cementitious materials existing at the SI which led to improvement of bonding quality as compared to the untreated samples.

#### Author statement

Conception and design of study: K.N Lichinga and Ntelo Seraphin Arthur Brunel, Acquisition of data: Ntelo Seraphin Arthur Brunel, K.N



Lichinga, Pan Lin analysis and/or interpretation of data: Ntelo Seraphin Arthur Brunel, K.N Lichinga, Pan Lin, Fanomezza Jennabbanou Johnson. Drafting the manuscript: Ntelo Seraphin Arthur Brunel, K.N Lichinga, Fanomezza Jennabbanou Johnson. Revising the manuscript critically for important intellectual content: Ntelo Seraphin Arthur Brunel, K.N Lichinga, Pan Lin, Ntelo Eugody Chrisman, Fanomezza Jennabbanou Johnson. Approval of the version of the manuscript to be published (the names of all authors must be listed): Ntelo Seraphin Arthur Brunel, K.N Lichinga, Pan Lin, Ntelo Eugody Chrisman, Fanomezza Jennabbanou Johnson.

### Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

### Data availability

Data will be made available on request.

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