



Utilization of Pineapple Leaf Fiber-Derived CMC to Reduce Filtration Loss and Extend Thickening Time in Oil Well Cementing

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Manuscript received: October 31th, 2025; Revised: November 20th, 2025

Approved: December 19th, 2025; Available online: December 22th, 2025; Published: December 22th, 2025.

ABSTRACT - Cementing operations in oil and gas wells require precise control of cement slurry properties to ensure successful zonal isolation. Two critical parameters—fluid loss control and setting time—significantly influence cement performance. Carboxymethyl cellulose (CMC) has been widely employed to regulate these properties; however, commercial CMC presents cost challenges for large-scale operations. This investigation evaluates the effectiveness of CMC which is synthesized from the waste of pineapple leaf fiber as an alternative additive for Class G drilling cement. The high cellulose content (69.5-71.5%) in pineapple leaf fibers indicates its potential as a cost-effective source of CMC that is compared to conventional agricultural wastes. CMC was extracted from pineapple leaves through alkaline delignification and chemical modification. The slurry of class G cement was formulated with varying CMC concentrations ranging from 0% to 0.4% by weight of cement (BWOC). Fluid loss was measured by using LPLT filter press following API standards, while setting characteristics were evaluated at 40°C and 60°C by using an atmospheric consistometer. Filtrate volumes decreased from 214.69 ml to 153.94 ml as CMC concentration increased from 0.1% to 0.3% BWOC, with all values conforming to API specifications (150-250 ml) for primary cementing. Commercial CMC from literature demonstrates comparable filtrate volumes of 160-180 ml at similar concentrations. Setting time was extended from 329 to 362 minutes at 40°C and from 188 to 266 minutes at 60°C with 0% to 0.4% CMC addition. Temperature significantly influenced hydration kinetics, with elevated temperatures accelerating cement setting regardless of CMC concentration. Pineapple leaf-derived CMC demonstrates comparable performance to commercial additives in controlling fluid loss and extending setting time in the systems of Class G cement. The optimal concentration of 0.3% BWOC provides adequate fluid loss control while maintaining acceptable setting characteristics. Further validation under high-pressure, high-temperature (HPHT) conditions and field-scale implementations are recommended.

Keywords: carboxymethyl cellulose, filtration loss, thickening time, pineapple leaf fiber, cement.

How to cite this article:

Fitrianti, Muhammad Alwi Pranata, and Owwen Tri Handoko, 2025, Utilization of Pineapple Leaf Fiber-Derived CMC to Reduce Filtration Loss and Extend Thickening Time in Oil Well Cementing, *Scientific Contributions Oil and Gas*, 48 (4) pp. 423-432. DOI.org/10.29017/scog.v48i4.1950.

INTRODUCTION

The formulation of cement slurries represents a fundamental aspect of well construction in petroleum engineering. Successful cementing provides multiple functions including maintaining wellbore structural integrity, preventing inter-formation fluid communication, and supporting casing strings throughout their operational lifetime (Pikłowska 2017; Bahrami et al., 2019). Nevertheless, Cementing operations often face technical challenges that can compromise the success of the operation and the well's integrity.

Two predominant challenges have been documented in cementing literature: uncontrolled fluid loss and inadequate setting time management. The phenomenon of fluid loss occurs when the liquid component of cement slurry migrates into adjacent permeable geological layers, which potentially cause premature slurry dehydration, inferior bonding characteristics, and compromised cement sheath integrity (Abu-Jdayil & Ghannam 2014; John 2017). Concurrently, insufficient control of setting time may result in premature solidification before the annular filling completes, therefore it generates flow channels and voids spaces that undermine isolation effectiveness (Hamid 2018; Huda et al., 2018).

To address these challenges, the oil and gas industry have extensively used chemical additives, particularly carboxymethyl cellulose (CMC), as a fluid loss control agent and thickening time modifier (Wijayani et al., 2010; Samura & Zabidi 2018). CMC works by increasing slurry viscosity, improving water retention, and controlling cement setting characteristics. However, commercially available industrial-grade CMC is relatively expensive, which increases operational costs, particularly in large-scale cementing operations. Contemporary investigations have examined

cellulose extraction from agricultural residues as potential CMC precursors. Various biomass sources including corn starch, water hyacinth (Mahendra 2017; Wijayani et al., 2010), sugarcane bagasse, and rattan bark have been evaluated as feedstock materials. Pineapple leaf fiber has demonstrated particular promise due to its superior cellulose concentration (69.5-71.5%), surpassing alternative agricultural wastes (Ari Setiawan et al., 2017). The widespread availability of pineapple processing residues which was combined with elevated cellulose levels presents an economically viable CMC production pathway.

While existing research has characterized cellulose-based additives from various sources, limited systematic evaluation exists for pineapple leaf-derived CMC specifically in oil well cementing under varying thermal conditions. Previous work by Arif (2015) investigated commercial retarder additives (lignosulfonates, Halad 22A, R-21LS) for Class G cement, which primarily focuses on compressive strength and rheological properties at elevated temperatures. The present study is differentiated fundamentally by: 1). Synthesizing CMC from agricultural waste rather than using commercial additives; 2). Emphasizing fluid loss control mechanisms alongside setting time, and; 3). Evaluating performance across operational temperature ranges representative of shallow to intermediate well depths.

Furthermore, direct comparison with commercial CMC performance remains limited in published literature. Studies by Plank et al. (2009) reported commercial CMC achieving 165-180 ml fluid loss at 0.3% concentration, while Bensted & Smith (2008) documented setting time extensions of 280-320 minutes at 40°C. These benchmarks provide essential context for evaluating alternative CMC sources.

This study addresses this gap by investigating the effectiveness of pineapple leaf fiber-derived CMC as an additive for Class G drilling cement. The specific objectives are:

- To synthesize CMC from pineapple leaf fiber waste through alkaline processing and evaluate its applicability as a cement additive
- To assess the effect of varying CMC concentrations (0%, 0.1%, 0.2%, 0.3%, and 0.4% BWOC) on filtration loss in Class G cement slurries
- To evaluate the influence of CMC concentration on thickening time at two operational temperatures (40°C and 60°C) representative of downhole conditions
- To determine optimal CMC dosage that meets API specifications for primary cementing operations

This lab study helps create eco-friendly and cost effective cementing solutions by using agricultural waste, while ensuring the cement slurry still performs well for successful well construction.

Determining an inappropriate and unsuitable cement slurry mixture is one of the problems that often occurs in cementing activity planning (Huda et al., 2018). The cementing process also experiences failures, resulting in material, time, and cost losses. One of the causes of cementing failure is the loss of fluid from the cement suspension into the permeable formation (filtration loss). In addition, an inappropriate thickening time causes the annulus to not be fully filled and results in the cement hardening prematurely (Hamid, 2018). Carboxymethyl cellulose (CMC) is used as an additive such as a thickener, emulsion stabilizer, adhesive stabilizer, and binder (Wijayani et al., 2010). It can also reduce filtration loss and fluid loss in the cement slurry. CMC in cement also serves to slow down the setting time, increase viscosity, and reduce the loss of cement circulation (Samura & Zabidi 2018). Pineapple leaves contain 69.5 – 71.5% cellulose fiber, which has the potential to be used as an additive (Ari Setiawan et al., 2017). Therefore, the researchers conducted a laboratory study to test the filtration loss and thickening time of G – class drilling cement with

varying concentrations of Carboxymethyl Cellulose (CMC) concentrations of 0%, 0.1%, 0.2%, 0.3%, and 0.4%.

METHODOLOGY

This experimental research was conducted under controlled laboratory conditions to assess fluid loss and setting characteristics of Class G drilling cement incorporating carboxymethyl cellulose (CMC) at concentrations of 0%, 0.1%, 0.2%, 0.3%, and 0.4% BWOC. Setting behavior was evaluated at two temperature levels (40°C and 60°C) to elucidate the combined effects of additive concentration and thermal conditions on cement performance.

Equipment and materials used

The equipment used included a digital scale, cement mixer, LPLT filter press, atmospheric consistometer, beakers, measuring cups, oven, stopwatch, cups, jars, sieves, and blender. The materials used were pineapple leaves, G-class cement, water, filter paper, methanol, ethanol, 15% HCL, NaOH (caustic soda), CH₃COOH (acetic acid), and H₂O₂ (hydrogen peroxide).

Production of CMC from pineapple leaf fibers

The following is a method for separating lignin from cellulose that is processed from alkaline into CMC (Ge & Chen, 2013; Mahendra, 2017). First, prepare pineapple leaf waste and wash it thoroughly, then cut it into pieces measuring approximately 1 cm and dry it in the oven at a temperature of 1200°C for 3 hours. The next step is delignification using 15% NaOH in 350 ml of water for 2 hours at a temperature of 1200°C for 50 grams of pineapple leaves in the oven. Next is the bleaching stage by using H₂O₂ by slowly dripping it onto 350 ml of the sample and soaking it at room temperature for 2 hours. Next, purify the cellulose using 9% NaOH and soak the sample for 2 hours at room temperature, then proceed to the monochloride acetic acid stage by mixing 100 ml of acetic acid and 100 ml of 15% HCl and soaking the sample for 2 hours at room temperature. Next,

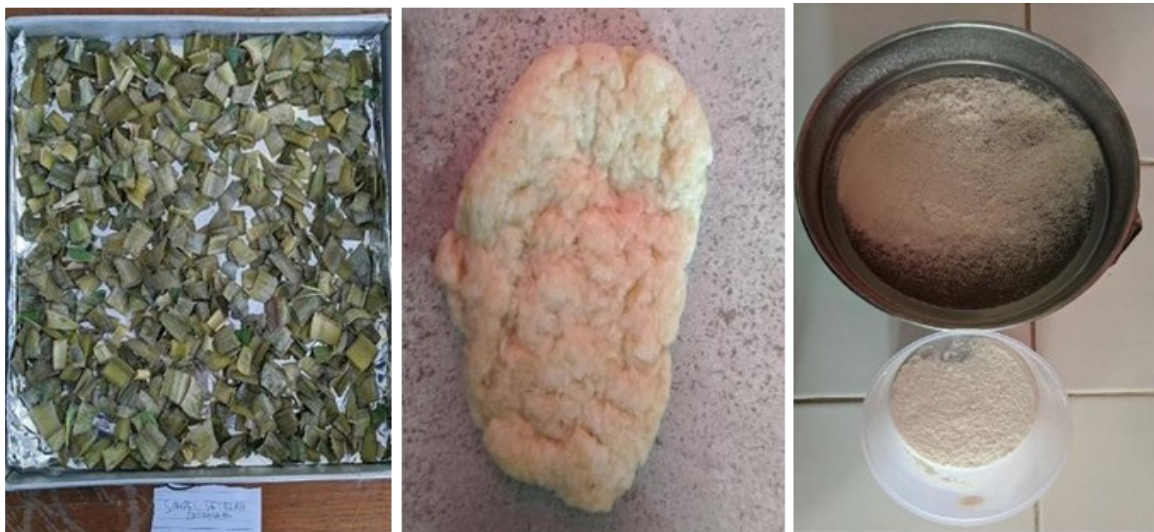


Figure 1. a. Pineapple Leaf Fiber, b. Pineapple leaf fiber that has been delignification, c. Pineapple leaf powder from delignification

enter the neutralizer stage by using 100 ml of ethanol, 100 ml of methanol, and 100 ml of acetic acid, then soak the sample for 2 hours. After finishing, wash the sample with distilled water until clean, then dry it again in an oven for 2 hours at a temperature of 120°C. The final stage is grinding and sieving, which involves sifting the sample using a 100-mesh sieve to separate the fine CMC from the fine lignin residues which is still attached to the cellulose.

Filtration loss testing

Mix cement slurry with a predetermined composition; 600 grams of G – cement, 264 ml of water (44% WCR), and adding CMC 0%, 0.1%, 0.2%, 0.3%, 0.4% BWOC (By Weight of Cement) using a mixer (Arif, 2015). Install filter paper and place a measuring cup under the cylinder, pour the cement suspension into the cylinder and immediately close the valve. Then flow N₂ gas at a pressure of 100 psi. Measure the volume of filtrate as a function of time with a stopwatch. The filtration loss is determined from the volume of filtrate which is collected in the measuring cup after 30 minutes of testing. Stop the flow of N₂ gas, release the pressure in the cylinder, and pour the remaining cement suspension in the cylinder into a breaker.

Thickening time testing

Mix the cement slurry with the specified composition; 700 grams of G cement, 308 ml of water (44% WCR), and add CMC 0%, 0.1%, 0.2%, 0.3%, 0.4% BWOC (By Weight of Cement) by using a mixer (Arif, 2015). Prepare stopwatch and calibrate the equipment to be used beforehand. Turn on the malster switch and set the temperature to the desired level, namely 40°C and 60°C (Arif, 2015). Pour the cement slurry into the slurry container up to the height which is indicated by the white line. Put the paddle coated with grease on the lid, then place the lid that has been placed on the paddle on the slurry container and insert it into the Atmospheric Consistometer. Turn on the motor and record the time when the device shows a scale of 70 UC.

RESULT AND DISCUSSION

Filtration loss

Figure 2 demonstrates a consistent decline in filtrate volume with increasing CMC concentration across samples 2-5. This trend results from the water-binding capacity of pineapple leaf fiber CMC, which effectively retains aqueous phase within the cement suspension, thereby replacing industrial additives in fluid loss control applications

(Abu-Jdayil & Ghannam 2014). Concentrations of 0.1%, 0.2%, and 0.3% BWOC yielded filtrate volumes of 195.34 ml, 174.18 ml, and 153.94 ml respectively, all conforming to API Spec. 10A requirements (150-250 ml range) for primary cementing operations. However, at 0.4% BWOC concentration, fluid loss decreased to 132.18 ml, falling 17.82 ml (11.6%) below the minimum acceptable threshold. This deviation, while indicating enhanced fluid retention, suggests over-stabilization that may adversely affect other cement properties such as pump-ability and placement efficiency.

Comparison with commercial CMC:

Published data for commercial CMC performance shows: 1). Plank et al. (2009): 165-180 ml at 0.3% concentration; 2). Bensted & Smith (2008): 170-190 ml at 0.3% concentration; 3). Present study: 153.94 ml at 0.3% concentration.

The pineapple leaf-derived CMC demonstrates slightly superior fluid loss control (9-17% reduction) compared to commercial products at equivalent concentrations, which potentially attributable to higher degree of substitution or molecular weight distribution.

The observed relationship between CMC concentration and fluid loss confirms that CMC functions as an effective fluid loss control agent (John, 2017). The CMC forms a filter cake at the cement-formation boundary, reducing permeability and limiting fluid invasion. The 0.3% BWOC concentration optimally balances fluid loss control and operational needs.

Thickening time

Figure 3 illustrates the combined effects of temperature and CMC concentration on setting characteristics. The incorporation of pineapple leaf fiber CMC demonstrates clear retardation effects on cement hydration kinetics (Samura & Zabidi, 2018). At 40°C, progressive CMC addition from 0% to 0.4% BWOC extended setting time from 329 to 362 minutes, which represents a 10% increase. This temperature yielded the maximum observed setting time of 362 minutes at 0.4% concentration.

At elevated temperature (60°C), setting times ranged from 188 minutes (0% CMC) to 266 minutes (0.4% CMC), demonstrating a 41% extension. However, all values at 60°C remained substantially lower than corresponding 40°C measurements. It confirms that elevated temperatures accelerate cement hydration rates regardless of CMC presence. This temperature sensitivity results from increased reaction kinetics at higher thermal energy levels (Arif 2015).

Comparison with Commercial CMC and Acceptable Ranges:

Published setting time data shows: 1). Bensted & Smith (2008): Commercial CMC at 0.3% yields 280-320 minutes at 40°C; 2). Nelson & Guillot (2006): Commercial CMC at 0.3% yields 195-230 minutes at 60°C; 3). Present study: Pineapple CMC at 0.3% yields 356 minutes at 40°C and 219 minutes at 60°C.

API Spec. 10A recommends setting times between 90-360 minutes depending on well depth and placement requirements. For shallow wells (1000-2000m), 180-240 minutes is typical; for intermediate depths (2000-3500m), 240-360 minutes is preferred. The pineapple-derived CMC provides longer setting times than commercial products (11-27% extension at 40°C), beneficial for complex well geometries requiring extended placement time. At 60°C, performance is comparable to commercial products, indicating thermal stability of the additive. The CMC molecules adsorb onto cement particles, creating steric barriers that block water access and slow hydration. Hydroxyl and carboxyl groups in CMC bind calcium ions, further slowing calcium silicate hydrate formation (Plank et al., 2009).

Temperature effect analysis

Temperature has dominant influence on setting behavior, with 60°C conditions reducing setting times by approximately 42-47% which is compared to 40°C across all CMC concentrations. This demonstrates that elevated circulation temperatures substantially increase hydration rates, yielding shorter setting times (Arif 2015). The temperature effect was consistent across CMC dosages,

showing that thermal activation of cement hydration overrides chemical retardation at high temperatures.

The Arrhenius relationship governs this temperature dependency, where reaction rate doubles approximately every 10°C increase. The 20°C differential between test conditions resulted in accelerated hydration kinetics that shortened setting times by an average of 44%, consistent with

theoretical predictions and field observations (Nelson & Guillot 2006).

Optimal concentration determination

Based on the comprehensive evaluation of both fluid loss and setting time parameters, 0.3% BWOC emerges as the optimal CMC concentration for primary cementing applications. This concentration provides:

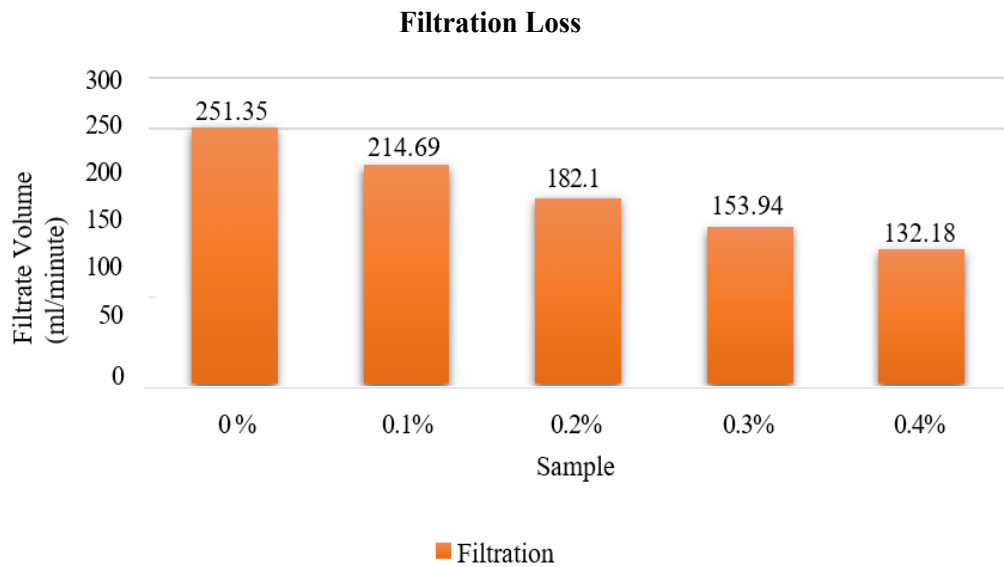


Figure 2. Filtration loss test results

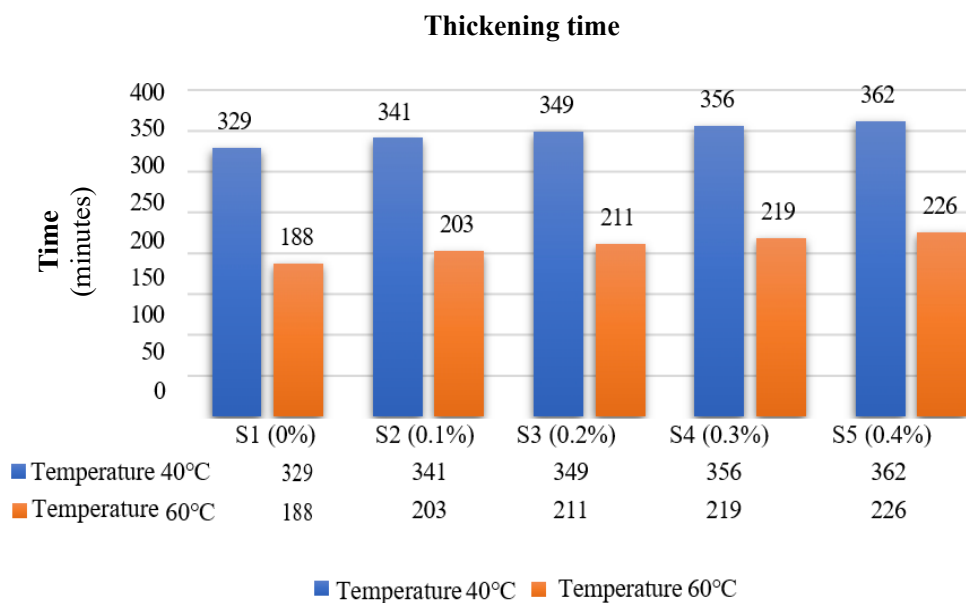


Figure 3. Thickening time test results

- Fluid loss control: 153.94 ml (within API range of 150-250 ml, representing 28% reduction from baseline).
- Setting time at 40°C: 356 minutes (within acceptable range for intermediate-depth wells, 8.2% extension from baseline).
- Setting time at 60°C: 219 minutes (appropriate for shallow to intermediate wells, 16.5% extension from baseline).
- Cost-effectiveness: Lower concentration minimizes additive consumption while meeting performance requirements.

The 0.4% concentration, while demonstrating superior retardation effects, falls below the minimum fluid loss threshold and may introduce excessive viscosity that compromises pump-ability and placement efficiency.

CONCLUSION

This laboratory investigation demonstrates that carboxymethyl cellulose which is synthesized from pineapple leaf fiber waste functions effectively as an additive for Class G drilling cement, providing comparable or superior performance to commercial CMC products in controlling fluid loss and extending setting time. The following conclusions are drawn:

- Progressive CMC addition systematically reduces filtrate volume, with concentrations of 0.1%, 0.2%, and 0.3% BWOC yielding values of 195.34 ml, 174.18 ml, and 153.94 ml respectively—all conforming to API Spec. 10A requirements (150-250 ml range). The 0.3% concentration demonstrates optimal performance, which achieves 28% fluid loss reduction while maintaining compliance. Comparison with commercial CMC (165-180 ml at 0.3% per Plank et al., 2009) indicates that pineapple-derived CMC provides 9-17% superior fluid loss control, which potentially due to higher molecular weight or degree of substitution.

- 2. CMC incorporation extends setting time at both evaluated temperatures. At 40°C, setting times increased from 329 minutes (baseline) to 362 minutes (0.4% CMC), with the 0.3% concentration yielding 356 minutes—within the recommended range for intermediate-depth wells (240-360 minutes) and comparable to commercial CMC performance (280-320 minutes) (Bensted & Smith, 2008). At 60°C, setting times ranged from 188 to 266 minutes, with 0.3% concentration producing 219 minutes, suitable for shallow to intermediate applications (180-240 minutes typical range).
- 3. Elevated temperature has dominant influence on hydration kinetics, reducing setting times by 42-47% when increasing from 40°C to 60°C across all CMC concentrations. This confirms that thermal activation of cement hydration outweighs chemical retardation, requiring temperature-specific adjustments for field applications.
- 4. The 0.3% BWOC concentration represents the optimal balance between fluid loss control and setting time management, meeting API specifications while providing performance advantages over baseline formulations. This concentration minimizes additive consumption while maximizing operational benefits.

Research limitations

Several limitations should be acknowledged: 1). Testing was conducted at atmospheric pressure; high-pressure, high-temperature (HPHT) conditions representative of deep well environments were not evaluated; 2). Long-term cement sheath integrity and compressive strength development were not assessed; 3). Only two temperature levels were investigated; continuous temperature profiling would provide more comprehensive understanding; 4). Rheological properties and pump-ability characteristics were not systematically evaluated; 5). Field-scale validation and compatibility with various formation

types remain to be demonstrated; 6). Direct side-by-side comparison with commercial CMC in identical testing conditions was not performed.

Recommendations for future research

Future investigations should validate these findings under high-pressure, high-temperature (HPHT) conditions which simulates deep well environments (temperatures exceeding 150°C and pressures above 10,000 psi). Field-scale implementation studies are necessary to evaluate performance in actual wellbore conditions with formation-specific compatibility testing.

Additional research directions include: 1). A Comprehensive characterization of CMC molecular structure, degree of substitution, and molecular weight distribution; 2). A Long-term mechanical property evaluation, including compressive, tensile, and bond strength development over 7, 14, and 28 days; 3). Rheological characterization across temperature and shear rate ranges representative of mixing, pumping, and placement operations; 4). Compatibility assessment with other commonly used cement additives (dispersants, weighting agents, and extenders); 5). An economic analysis which compares the production costs of pineapple-derived CMC versus commercial products at industrial scale; 6). An environmental impact assessment that measures the sustainability benefits of utilizing agricultural waste; 7). Optimization of synthesis parameters to maximize yield and functional properties; 8). A direct comparative evaluation of performance against multiple commercial CMC products under identical experimental conditions.

The successful utilization of pineapple leaf fiber waste as a CMC precursor demonstrates the viability of agricultural residue valorization for petroleum industry applications. This approach supports circular economy principles and has the potential to reduce operational costs as well as the environmental impacts of cementing operations.

ACKNOWLEDGEMENT

The authors are grateful to the Drilling Laboratory, Universitas Islam Riau, for assisting and supporting this research.

GLOSAARY OF TERM

Unit	Definition	Symbol
API	American Petroleum Institute (Standard for testing)	
BWOC	By Weight of Cement	%
CMC	Carboxymethyl Cellulose	
HPHT	High-Pressure, High-Temperature	
LPLT	Low Pressure Low Temperature	
WCR	Water Cement Ratio	%
UC	Unit of Consistency (Atmospheric Consistometer scale)	
NaOH	Sodium Hydroxide (Caustic Soda)	
HCl	Hydrochloric Acid	
H ₂ O ₂	Hydrogen Peroxide	
CH ₃ COOH	Acetic Acid	
Filtration Loss	Loss of fluid from cement suspension into permeable formation	ml
Thickening Time	Time required for cement slurry to reach specific consistency (70 UC)	minutes
Class G	Specific classification of oil well drilling cement	

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