

ANALYSIS OF BIOPOLYMER FROM CASSAVA PEEL USING CROSSLINKER Cr^{3+} AS AN ALTERNATIVE MATERIAL IN WATER CONING

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Abstract

Polymers play a crucial role in chemical processes for controlling water condensation. This study chose biopolymer materials derived from cassava peels due to their superior environmental sustainability compared to synthetic polymers commonly used in water conditioning. While synthetic polymers are frequently employed for water conditioning, they may pose adverse environmental impacts. The chemical polymer introduced into the water formation involves the injection of a chromium (Cr^{3+})-based agent, enhancing viscosity and impeding water movement. The primary objective of this study is to utilize cellulose from cassava peels as the main component for producing sodium carboxymethyl cellulose, aimed at mitigating water coning. The synthesis and hydrolysis processes involve the use of sulfuric acid and cassava peels as biopolymers. The Fourier-Transform Infrared (FTIR) spectroscopy test revealed the presence of functional groups such as OH, C-H, C-O, and C=O in the cassava epidermis. The cellulose yield ranged from 62.3% to 90%, demonstrating a proportional decrease in yield weight with increased sulfuric acid concentration. Concentrations of 1,000, 2,000, and 3,000 ppm were employed, with salinity set at 5,000, 10,000, and 15,000 ppm, respectively. These conditions were maintained at 60, 70, and 80°C. The compatibility of solutions with concentrations of 1,000 and 2,000 ppm was assessed. The results indicate that solutions at 1,000 and 2,000 ppm concentrations are sui, showing no precipitate and clear characteristics.

Keywords: Biopolymer; chemical polymer; crosslinker; sulfuric acid

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Introduction

Excess water production in oil and gas wells is often attributed to reservoirs with a bottom-water drive mechanism. One of the key factors contributing to water production is the influx of water into the oil zone, a phenomenon known as water coning (Frigrina *et al.*, 2017). The detrimental impact of excessive water production on well productivity cannot be overstated, as it not only affects individual well performance but also introduces new challenges to the overall oil production process.

Water coning is characterized by the breakthrough or movement of formation water into the oil zone, forming a cone that leads to premature and increased water production (Okon *et al.*, 2018). This breakthrough occurs because the mobility of water surpasses that of oil. Wells exceeding the critical production rate, experiencing low well pressure (PWF), leading to high drawdown pressure, and having perforations situated too close to the water-oil contact are conditions conducive to the occurrence of water coning.

Various oil and gas industry methods address water coning and high-water cuts, including reducing production rates, recompletion, water shutoff, chemical polymer, and dual completion. This study uses the chemical polymer method to counter water coning and high water cut.

Chemical techniques effectively manage excess water production without hindering hydrocarbon output (Fang *et al.*, 2019). Modifying relative permeability without isolating layer zones enables bullhead injection into all layers, reducing water permeability through increased water viscosity. Polymer gel solutions are simple and cost-effective, requiring minimal polymer and a crosslinker (Frigrina *et al.*, 2017).

Two primary types of polymers play a pivotal role in this treatment: synthetic polymers, specifically partially hydrolyzed polyacrylamide (HPAM), and biopolymers, exemplified by polysaccharides. Biopolymers from plants, animals, and microorganisms encompass natural compounds like starch, dextran, xanthan, chitosan, and cellulose formed within cells through complex metabolic processes (Gao, 2016). Notably, biopolymers are effective viscosity enhancers, acting as potent thickening agents due to their excellent water-soluble properties. They also exhibit good thermal resistance, are environmentally friendly, and are easily obtainable (Ayoola *et al.*, 2018).

Chromium emerges as the most extensively employed metal crosslinker in this context. Crosslinkers enhance the viscosity of gelling

agents by interconnecting separate gel polymer chains. This interconnection substantially augments the linear gel viscosity and strength by increasing the molecular weight of the polymer. The advantage of employing this crosslinking method lies in its ability to yield biopolymers resistant to both acid and thermal media, preventing their easy degradation when heated.

Cassava (*Manihot esculenta*), a carbohydrate-rich plant, generates various usable parts, producing waste, particularly cassava skin. Despite its potential utility, cassava skin is often discarded by communities. Researchers aspire to harness this waste to prevent environmental pollution (Setiawan *et al.*, 2017).

The cellulose content in cassava peel is approximately 57% (Ningrum, 2019). In this study, researchers plan to utilize the cellulose from the inner cassava peel to produce sodium carboxymethyl cellulose (NaCMC) to counteract water coning. To augment viscosity, a crosslinker is introduced to the polymer material.

Materials and Methods

Materials

The researchers conducted this investigation at the Petroleum Engineering Reservoir Laboratory, Islamic University of Riau. The methodology employed for this experiment is based on synthesis methods aimed at producing biopolymer products in the form of NaCMC through a series of processes, including hydrolysis, delignification, bleaching, carboxymethylation, and alkalization. The components utilized in this synthesis include methanol, isopropyl alcohol, acetic acid, sodium hydroxide (NaOH), sulfuric acid, trichloroacetic acid, hydrogen peroxide (H₂O₂), sodium chloride (NaCl), xanthan gum, and chromium acetate. The cassava peel waste was procured from Kampar District, Siak Hulu, Riau, Indonesia.

Material Preparation

The initial phase of this investigation involves the preparation of samples or materials. The cassava peel is initially separated from the outer skin, followed by a washing process and grinding into powder. The obtained powder is then subjected to sun-drying for 18 h until it attains a constant weight (Figure 1(a)) and is further sieved through a 100-mesh sieve (Figure 1(b)) (Setiawan *et al.*, 2017).



Figure 1. Drying process of cassava peel samples
(a) 100 mesh sieve process (b)



Figure 2. Hydrolysis preparation of cassava peel using H₂SO₄

Cassava Peel Cellulose Powder Production

The process of obtaining cellulose powder from cassava peel begins with the hydrolysis of cassava peel using sulfuric acid at concentrations of 45, 50, and 55%(w/w), followed by neutralization with distilled water (Pratama *et al.*, 2019). The sample undergoes delignification by treating it with 40% NaOH and heating it at 100°C for 3 h, after which it is neutralized with distilled water to facilitate the initial lignin removal process (Basmal *et al.*, 2018). The procedure advances with the bleaching of the sample using a mixture of NaOH and H₂O₂ at 20% for 3 h, with continuous stirring and subsequent filtration (Figure 2). The solid phase is thoroughly rinsed with distilled water until the residual water runs clear. The cellulose samples are then dried in an oven at 55°C for one day (Suriyatem *et al.*, 2020).

Cassava Peel Biopolymer in the Form of Sodium NaCMC (Carboxymethyl Cellulose)

Following the creation of cellulose powder, the next step involves generating a biopolymer. This process includes mixing and stirring the cellulose powder (15 g), 30% NaOH, and isopropanol in a beaker for 3 h at 50°C (Figure 3(a)) (Suriyatem *et al.*, 2020). Gradually, a chloroacetic acid (IPA) is added to the mixture, which is stirred for 30 min before being placed in a 55°C oven for up to 24 h. The solid phase is neutralized with acetic acid (CH₃COOH) after being mixed with methanol. Following filtration, the mixture undergoes rinsing with 70% methanol. Subsequently, the biopolymer is dried in an oven at 55°C for up to 24 h (Figure 3(b)). The equation below, provided by (Penjumras *et al.*,

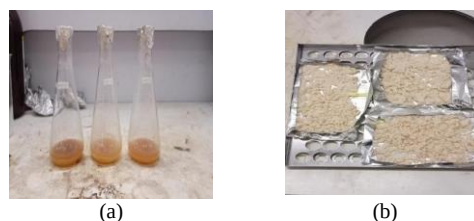


Figure 3. Preparation of carboxymethyl cassava peel (a) and cassava peel biopolymer (b)

2014), can be utilized to calculate the percentage of cellulose:

$$\text{Cellulose (\%)} = \frac{W_2}{W_1} \times 100\% \quad (1)$$

W₁: initial cassava skin weight (gr)

W₂: final cassava skin weight (gr)

Biopolymer Functional Group Analysis Procedure

The method for analyzing functional groups in biopolymers follows the approach outlined by Santoso *et al.* This involves weighing 3 g of the most optimal biopolymer, followed by milling it with potassium bromide (Shella *et al.*, 2012). The resulting mixture is pressed to form a thin plate with a 1 cm diameter. The FTIR spectrum is recorded in the range of 4000-400 cm⁻¹. The sample is exposed to the NaCl window to determine the functional groups of the biopolymer. Two NaCl windows are pressed together to eliminate any air bubbles. The sample is then placed in the demountable cell for quantitative analysis, followed by the actual analysis of the sample.

Biopolymer Mixing Procedure with a Crosslinker

Concentrations of 1,000, 2,000, and 3,000 ppm, along with a 10% crosslinker added to the biopolymer concentration, were introduced to attain the desired gel formation, as per the study by (Frigrina *et al.*, 2017). Additionally, brine salinity levels of 5,000, 10,000, and 15,000 ppm were incorporated, with temperature variations set at 60, 70, and 80°C.

Gelation Time Test

The test utilizes the bottle method, where a biopolymer solution is mixed with a crosslinker in a test tube at different concentrations, temperatures, and salinity levels. The goal is to observe the onset of gel formation (Frigrina *et al.*, 2017). The procedure involves placing the biopolymer solution in a test tube, varying salinity, concentration, and temperature, monitoring physical changes, and initiating gelation time.

Viscosity Test

Viscosity is a fluid property influencing its resistance to deformation at a specific level, commonly denoted as the fluid's viscosity value. The Ostwald viscometer is employed for viscosity determination (Saputra *et al.*, 2017). To conduct the test, prepare the biopolymer solution and the viscometer. Inject 3 ml of the biopolymer solution into the viscometer, drawing the liquid with a rubber ball until it reaches the upper limit. Remove the rubber ball and let the liquid traverse the viscometer's upper limit. Measure the time it takes for the biopolymer solution to move from the top to the bottom line using a stopwatch. The resulting time, denoted as the "t" value, is recorded, and the viscosity is calculated using the following equation:

$$\mu_{kin} = C \times t \quad (2)$$

$$\mu_{din} = \rho \times \mu_{kin} \quad (3)$$

μ_{kin} = kinetic viscosity (cst or centistoke)

C = viscometer constant *Ostwald* (0,4994 cst. S⁻¹)

t = fluid flow time (s)

μ_{din} = dynamic viscosity (cP)

ρ = fluid density (gr/ml)

Compatibility Test

A compatibility test was conducted to assess the extent of compatibility between the biopolymer and the tested salinity levels. The test spanned three days, and the biopolymer passing the compatibility assessment yielded a clear solution without precipitation (Dhafa *et al.*, 2021).

To create a 5,000-ppm brine solution, measure 2.5 g of NaCl salt. Ensure the 500-ml measuring cup is filled with Aquadize and then weigh the salt using a digital scale. Add the salt to a beaker glass and stir with a magnetic stirrer until a homogeneous solution is achieved. Transfer the NaCl solution into the designated container. For a brine solution with a salinity ranging from 10,000-15,000 ppm, refer to the calculation results presented in Table 1. Follow the appropriate proportions to achieve the desired salinity level. To prepare biopolymer solutions at 1,000, 2,000, and 3,000 ppm concentrations, use a solvent (brine)

containing 0.04, 0.08, and 0.12 g of biopolymer, respectively (Alquraishi and Alsewailem, 2012).

Results and Discussion

NaCMC (Sodium Carboxymethyl Cellulose) Cassava Peel

The hydrolysis of cassava peel samples involves blending the peel until smooth and filtering them. This process aims to enhance and influence the hydrolysis process utilizing sulfuric acid, ensuring maximal acid reaction during fiber granule penetration. Hydrolysis, a reaction between reactants and water leading to compound decomposition, is commonly conducted with acids such as sulfuric, acetic, hydrochloric, and phosphoric acids. Among these, sulfuric acid is favored for its effectiveness in hydrolysis. Pratama *et al.* (2019) findings on cellulose hydrolysis using sulfuric acid align with this preference, highlighting sulfuric acid's superiority in cellulose production due to its higher hydronium ion content, allowing for perfect monomer termination. The resulting product's darkness correlates with the sulfuric acid concentration.

In addition to facilitating hydrolysis, this process, coupled with delignification, removes lignin. High-purity cellulose is obtained by further removing lignin using NaOH. Since the reaction produces a blackish-red color, bleaching is necessary to achieve a whiter and cleaner appearance before neutralization. Subsequently, alkalization and carboxymethylation processes are carried out.

Researchers employed a 30% NaOH solution in the alkalization process for 30 min; this step aimed to achieve a uniform and even reaction between the sample and the solution. To ensure thorough wetting, the sample was meticulously treated with NaOH before advancing to the subsequent carboxymethylation process, which involved using trichloroacetic acid for 30 min. During arboxymethylation, cellulose underwent substitution with Na⁺ and reacted with trichloroacetic acid, resulting in the production of CMC and NaCl. This methodological approach ensures a systematic modification of cellulose, yielding the desired products with controlled reactions and processing times.

Cellulose Yield Results

The percentage yield of biopolymer spans from 62.3% to 90%, with the highest value observed in the hydrolysis treatment using sulfuric acid at a concentration of 45%(w/w) and the lowest in the hydrolysis treatment using 55%(w/w).

Table 1. Calculation of the mass of brine (NaCl)

Salinity Concentration (ppm)	Mass of NaCl (g)
5,000	2.5
10,000	5
15,000	7.5

Table 2. Results of the effect of sulfuric acid concentration on the weight of cellulose

Cassava Peel Sample	Yield	Cellulose Yield Percentage
H ₂ SO ₄ 45%	13.5 g	90%
H ₂ SO ₄ 50%	11.5 g	77.3%
H ₂ SO ₄ 55%	9.35 g	62.3%

According to the observations in Table 2, cellulose yields range from 62.3% to 90%. Findings indicate an inverse relationship between sulfuric acid concentration and cellulose yield weight. Higher sulfuric acid concentrations result in lower cellulose powder production, as increased acid concentration leads to more hydrolysis of amorphous cellulose areas into monosaccharides (Pratama *et al.*, 2019). Biopolymers with a sulfuric acid concentration of 45% exhibit the highest cellulose percentage, influenced by the hydrolysis and delignification processes, noted that higher hydronium ion content in sulfuric acid contributes to more complete monomer termination during hydrolysis. Hence, relying on the outcomes of the hydrolysis selection involving 45% sulfuric acid, a candidate was selected for further testing in the subsequent experiment.

The impact of sulfuric acid concentration was explored by Ioelovich (2018). The solubility of the initial sample gradually increased within the acid concentration range of 50%-60%, yielding an insoluble cellulose residue with a relatively low sulfonate group content (Ioelovich, 2018). Complete dissolution of the cellulose sample occurred at an H₂SO₄ concentration of 65 wt%, resulting in cellulose regenerating within the crystalline polymorph CII. However, the yield of regenerated cellulose decreased as the H₂SO₄ concentration exceeded 65 wt%. Treatment with 0 wt.% H₂SO₄ led to the formation of low-molecular-weight oligomers, preventing the regeneration of dissolved cellulose from the acid solution through dilution with water. Ioelovich proposed a method for producing amorphous cellulose by regenerating its solution in a 65 wt% H₂SO₄ solution.

Analysis of Fourier Transformer Infrared (FTIR) Functional Group Results

The wavelength of the carboxymethyl compound is employed to analyze the identification results obtained from the infrared spectrum analysis readings on NaCMC samples derived from cassava peel samples.

The infrared spectrum wave results for the cassava peel biopolymer are illustrated in Figure 4(a). Cassava peel samples exhibit the presence

of functional groups OH, C=O, C-O, and C-H. The hydroxyl group (-OH), a characteristic feature of cellulose (Salimi *et al.*, 2021), is identified in this study at a wave number of 3,250 cm⁻¹, differing slightly from the placement suggested at 3,500-3,200 cm⁻¹. The C-H group, typically within the range of 2,850-3,000 cm⁻¹, according to Sastrohamidjojo (1991), is observed in this study at a wavelength of 2,889 cm⁻¹. The C-O functional group is identified at the 1,380 cm⁻¹ wave, while the suggested wave number range is 1,680-1,750 cm⁻¹, according to Salimi *et al.* (2021). In this study, however, the C-O functional group is observed at the 1,690 cm⁻¹ wave.

The results of the infrared spectrum wave on the NaCMC biopolymer are displayed in Figure 4 (b). The image illustrates the presence of the OH, CO, CO, and C-H functional group spectra peaks in the NaCMC sample. The wave number for the OH functional group is 3,700 cm⁻¹. The C-H functional group is detected at wave number 2,867 cm⁻¹. Additionally, the C-O functional group exhibits a wave number of 1,324 cm⁻¹, while the C-O

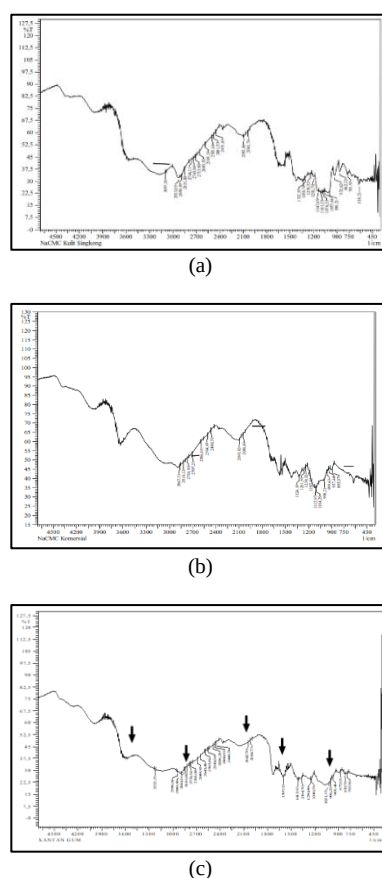


Figure 4. Spectra of FTIR waves on commercial CMC samples (a), NaCMC samples (b), and Xanthan Gum samples (c)

functional group is situated at a wave number of 1,680 cm⁻¹.

The results of the infrared spectrum wave for the xanthan gum biopolymer are illustrated in Figure 4(c). The image showcases peak spectra corresponding to the xanthan gum sample's OH, C=O, C-O, and C-H functional groups. The OH functional group is identified at a wavenumber of 3,685 cm⁻¹, the C-H functional group is observed at wave number 2,844 cm⁻¹, and the C=O functional group is seen at wave number 1,780 cm⁻¹, with the C-O functional group appearing at wave number 1,412 cm⁻¹.

The infrared wave spectra of the cassava peel samples, commercial CMC, and xanthan gum are depicted in Figure 4. Absorption peaks at strong wave numbers of 3,750 and 3,350 cm⁻¹ are evident in the cassava peel sample and indicate the OH functional group. Similarly, peaks at 3,700 cm⁻¹ in the commercial CMC sample and the xanthan gum sample denote the OH functional group. The OH functional group, represented by the peak at wave number 3,685 cm⁻¹, indicates the formation of hydrogen bonds in a hydroxyl group in a glucose monomer within the cellulose polymer chain or the presence of cellulose in the NaCMC polymer chain, emphasizing its importance in NaCMC formation. Additionally, the cassava peel sample exhibits a spectrum peak ranging from 2,889 to 2,950 cm⁻¹, forming a C-H group; the commercial CMC sample displays a peak ranging from 2,950 to 2,870 cm⁻¹, forming a C-H group; and the xanthan gum sample shows a peak at 2,844 cm⁻¹, forming a C-H group. According to Megawati *et al.* (2017), the vibration peak at C-H links glucose units found in cellulose.

Table 3 illustrates variations in functional groups across NaCMC cassava peel, commercial CMC, and xanthan gum samples, highlighting the impact of different wave numbers on NaCMC product yield and quality. This test was conducted to showcase the formation of NaCMC by observing the absorption of functional groups in NaCMC. The infrared spectrum of NaCMC comprises functional groups such as OH, C-H, C-O, and C=O. The presence of OH, C-H, C-O, and C-O clusters in the cassava peel samples aligns with the requirements for NaCMC formation, as detailed in Table 3.

Table 3. Biopolymer functional group test

Biopolymer	Functional Groups	Wave number (cm ⁻¹)
Cassava Peel (NaCMC)	O-H	3,250
	C-H	2,889
	C-O	1,380
	C=O	1,690
Commercial CMC	O-H	3,700
	C-H	2,867
	C-O	1,324
	C=O	1,680
Xanthan Gum	O-H	3,685
	C-H	2,844
	C-O	1,412
	C=O	1,780

Gelation Time Result Analysis

Gelation time testing on a biopolymer is crucial for determining the speed at which a biopolymer can form a gel and function effectively after injection into a reservoir with specific polymer concentration and temperature conditions. The gelation time test analysis on a cassava peel and

Table 4. Gelation time biopolymer with and without crosslinker

Biopolymer Type	Salinity (ppm)	Concentration (ppm)	Gelation time (h) (no crosslinker)			Gelation time (h) (with crosslinker)		
			60°C	70°C	80°C	60°C	70°C	80°C
Cassava Peels (NaCMC)	5,000	1,000	2.5	2.5	2.5	2.5	2.5	2.5
		2,000	2.5	2.5	2	2.5	2.5	2
		3,000	2.5	2.5	2	2.5	2.5	2
	10,000	1,000	2.5	2.5	2.5	2.5	2.5	2.5
		2,000	2.5	2	2	2.5	2	2
		3,000	2.5	2	2	2.5	2	2
	15,000	1,000	2.5	2.5	2	2.5	2.5	2
		2,000	2.5	2	2	2.5	2	2
		3,000	2.5	2	2	2.5	2	2
Xanthan Gum	5,000	1,000	2.5	2	2	3	2.5	2.5
		2,000	2.5	2.5	2	3.5	3	3
		3,000	3	2.5	2.5	3.5	3	3
	10,000	1,000	2.5	2.5	2	3.5	3	3
		2,000	2.5	2	2	3.5	3.5	3
		3,000	2.5	2	2	3.5	3.5	3
	15,000	1,000	2.5	2.5	2	3	3	3
		2,000	2.5	2	2	3.5	3	3
		3,000	2.5	2	2	3.5	3.5	3

xanthan gum biopolymer solution is presented in Table 4.

According to the gelation time biopolymer test results in Table 4, which involves cassava peel and xanthan gum biopolymers, the gelation times for both solutions increase with higher test temperatures. This aligns with the findings of Frigrina *et al.* (2017) who noted that the gelation time in polymer solutions accelerates with higher test temperature due to the kinetics of the solution's temperature reaction. The results indicate that the xanthan gum takes substantially longer to form a gel. The cassava peel biopolymer and xanthan gum, when combined with a chromium (Cr^{3+}) crosslinker, exhibit a longer time for gel formation compared to the cassava peel and xanthan gum biopolymer without the addition of a crosslinker (Jia and Chen, 2018). Gelation time is observed to increase with viscosity. Researchers conduct observations every 30 min to ensure the accuracy of the results. The testing of these two types of biopolymers revealed that higher concentrations

of biopolymers and increased water salinity lead to extended gelation times (Lenji *et al.*, 2018).

Overestimating gelation time can lead to premature gelation in the wellbore, while underestimation can result in insufficient closing time. The crosslinking reaction takes longer at low than high crosslinker concentrations. Consequently, even at low polymer and crosslinker concentrations, gel penetration to greater depths is feasible (Sengupta *et al.*, 2012).

As indicated in a previous study Shriwal and Lane (2012), the crosslinking mechanism for HPAM polymer gels crosslinked with Cr^{3+} involves a substantial component of ionic bonds between negatively charged carboxylic groups in the polymer chain and multivalent cations (Shriwal and Lane 2012). The bond is intricate in the case of Cr because Cr^{3+} is a triple ion group of Cr^{3+} bridged by a carboxylate group. HPAM/Cr gels exhibit consistent gelation times at specific temperatures, and extensive field use has demonstrated their stability well above 200°F.

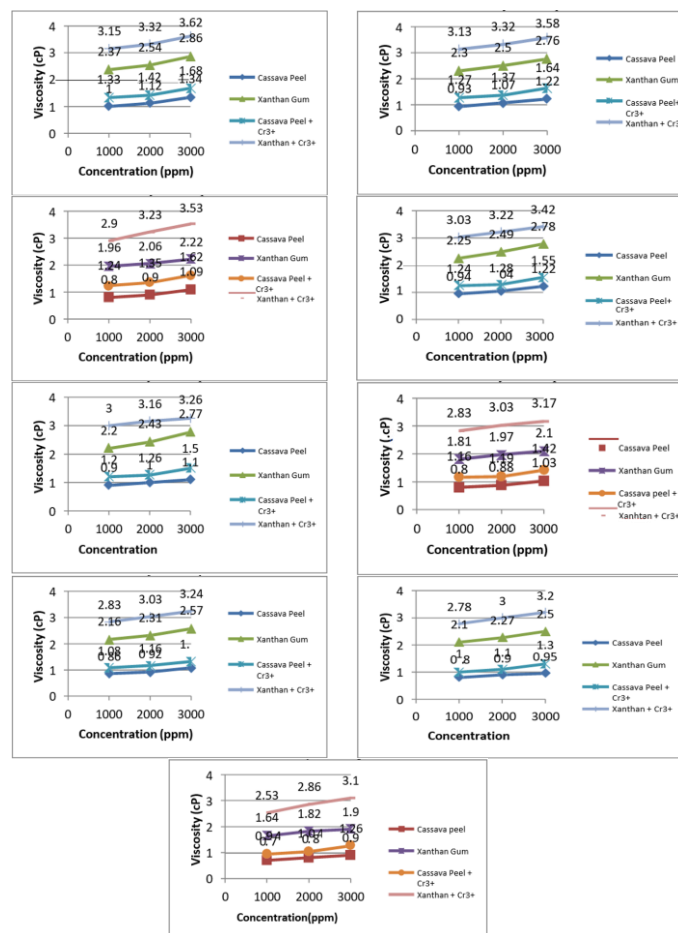


Figure 5. Effect of concentration on viscosity at temperatures of 60, 70, and 80°C, with salinity levels set at 5000, 10,000, and 15,000 ppm

As depicted in Figure 5, the viscosity values increase with the concentration of cassava peel biopolymer and xanthan gum, with xanthan gum demonstrating superior viscosity enhancement. Adding a crosslinker (Cr^{3+}) to cassava peel biopolymer did not yield a substantial increase in viscosity compared to the sample without the crosslinker. In contrast, when combined with a crosslinker, xanthan gum exhibited a substantial increase in viscosity compared to xanthan gum without a crosslinker.

The test results indicate that the brine salinity (NaCl) influences the viscosity of the biopolymer solution. A decrease in viscosity is attributed to metal ions in the brine solution pulling the branch chains from the polymer, causing a shortening of the polymer chain's shape. Salinity is a parameter that induces a decrease in polymer viscosity, and the addition of brine NaCl has an electrolyte resistance effect on NaCl ions and polymers, leading to polymer rolling. The neutralization of the negative charge in the polymer molecule by salt contributes to the decrease in polymer solution viscosity as the brine NaCl salinity increases.

The rheological characterization of the gel in the study (Simjoo *et al.*, 2009) indicates that increased polymer chain crosslinking in the presence of water formation results in higher gel viscosity and modulus of elasticity (Simjoo *et al.*, 2009). This effect is attributed to the presence of divalent cations in water formation.

This study used concentrations of 1,000, 2,000, and 3,000 ppm of biopolymers, and a 10% crosslinker was added to obtain a viscosity value suitable for water shutoff. Jia and Chen (2018) employed a polymer gel developed with 0.4%-0.6% by weight of KYPAM HPAM and 0.02%-0.04% (5:1), (10:1), and (20:1) by weight of chromium acetate in their research. According to the findings of this study, gelation time ranged between 2 and 4 h; considering pumping ability, the viscosity of the crosslinker gel solution should not be excessively high (Jia and Chen, 2018). When the crosslinker reaction occurs, target gel viscosities can reach 343, 545, 543, and 875 cP, and low gel viscosity benefits downhill pumping. Figure 6(a) compares the effect of temperature on the viscosity of cassava peel biopolymer with the addition of a crosslinker (Cr^{3+}), while Figure 6(b) compares the effect of temperature on the viscosity of xanthan gum biopolymer with the addition of a crosslinker (Cr^{3+}).

The viscosity of the polymer changes with increasing temperature. Figure 6 illustrates that as the temperature increases, the viscosity of the biopolymer sample decreases. This decrease is attributed to the thermal energy possessed by polymer molecules; the stronger thermal

vibration, compared to water molecules, can impede the association effect at elevated temperatures, leading to more effortless molecular movement and a subsequent decrease in viscosity (Mohd *et al.*, 2018).

At 70°C and 80°C, viscosity decreased in cassava peel biopolymer and xanthan gum samples at all concentrations. However, unlike the cassava peel biopolymer and xanthan gum with the addition of a crosslinker, the introduction of a crosslinker in the form of chromium (Cr^{3+}) in the cassava peel

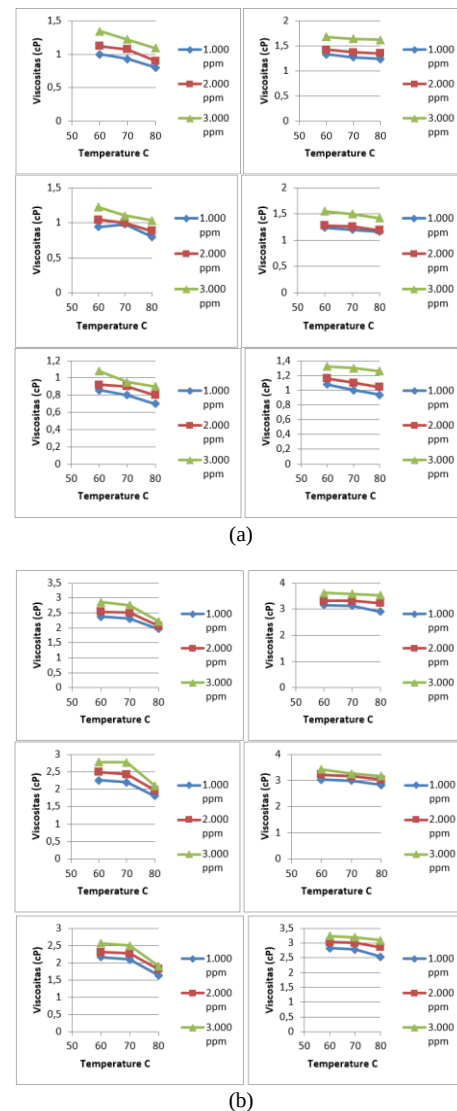


Figure 6. Effect of temperature on the viscosity of cassava peel biopolymer at salinities of 5,000, 10,000, and 15,000 ppm with an additional crosslinker (a). Effect of temperature on the viscosity of the xanthan gum biopolymer at salinities of 5,000, 10,000, and 15,000 ppm with an additional crosslinker (b)

biopolymer sample and xanthan gum stabilized the decrease in viscosity at 70°C and 80°C. This stabilization indicates that these two samples exhibit good thermal stability. The advantage of employing this crosslinking method is its ability to produce biopolymers resistant to acid and thermal conditions, preventing them from breaking when heated.

Biopolymer Compatibility Testing Analysis

A compatibility test assesses the suitability of a polymer solution by examining its interaction with the concentration of formation water (brine). The salinity of the formation water influences polymer adsorption, as the presence of salt can lead to the precipitation of the polymer. Successful compatibility testing is indicated by a fully dissolved solution that is clear and free of deposits. This outcome indicates that the solution, when injected, will not clog the pores of the rock.

The compatibility test results for the cassava peel biopolymer were obtained at temperatures of 60, 70, and 80°C. The outcomes revealed that the cassava peel biopolymer demonstrated compatibility at 1,000 and 2,000 ppm concentrations under salinity conditions of 5,000, 10,000, and 15,000 ppm across all temperatures (60, 70, and 80°C). However, at a concentration of 3,000 ppm, slight precipitate was observed in the solution at all salinity levels and temperatures, indicating that the solution was not entirely compatible. This precipitation could be attributed to the speed and duration of the biopolymer dissolution process with a stirrer. Additionally, variations in the testing time during the three-day compatibility test may have contributed to this observation (Figure 7) (Dhafa *et al.*, 2021). Compatibility testing for xanthan gum was conducted, and the findings revealed that xanthan gum exhibited compatibility across all concentrations and temperatures.

Adding a crosslinker, chromium (Cr^{3+}), to the cassava peel biopolymer at concentrations of 1,000

and 2,000 ppm, with salinity levels of 5,000, 10,000, and 15,000 ppm, resulted in a clear solution with no precipitate. However, at a concentration of 3,000 ppm, a slight precipitate was observed in all salinity levels and temperatures. This minor precipitate may be attributed to the residual solution that did not dissolve completely during stirring. In contrast, xanthan gum, with the addition of a crosslinker, showed compatibility at all concentrations and salinities, exhibiting no precipitate. The clarity of the solution and the absence of precipitate confirm the success of the compatibility test. The study indicates that higher polymer concentrations and increased salinity levels may decrease solution compatibility.

Conclusions

From this study, the following conclusions can be drawn:

1. The FTIR test results revealed that the cassava peel biopolymer contains OH, C=O, C-O, and C-H functional groups, with the OH group being particularly effective.
2. The FTIR test results for the three biopolymers in this study showed that the cassava peel biopolymer had a higher OH wave number than the other two polymers.
3. The cellulose's percentage yield ranged from 62.3% to 90%, with the highest value obtained in the hydrolysis treatment using sulfuric acid at a concentration of 45%(v/v).
4. With the addition of a crosslinker, the cassava peel biopolymer and xanthan gum exhibited longer gelation times than biopolymers without a crosslinker.
5. In viscosity measurement, it was observed that the biopolymer with the addition of a crosslinker (Cr^{3+}) could increase viscosity from its normal level but was not suitable for water shutoff due to its low viscosity.
6. Compatibility test results at 1,000 and 2,000 ppm concentrations across all salinity levels and temperatures passed successfully as the solutions were clear with no precipitate.

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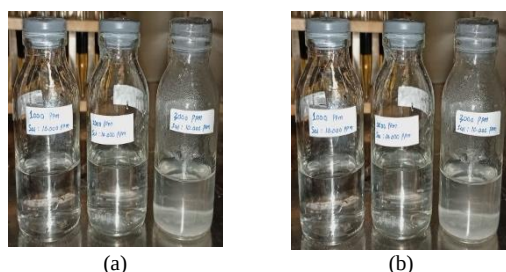


Figure 7. Sample cassava peel biopolymer compatibility: Day 1 (a) and Day 3 (b)

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