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Research Article

Investigation on guar gum and chitosan based polymer composite for oilfield water shut off fluid

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ABSTRACT

The present study highlights the performance of guar gum and chitosan based polymer composite (GG-CH-g-PAN/AA) as water shut off polymer solution for oilfield application. Study on salinity percentage, type of salts, pH and temperature for the salt present in formulation and salts contact with the polymer were investigated. The results of physical-chemicals parameters show that the composite polymer gave positive feedback as water shut off fluid at 60◦C and strength of fully developed polymer was obtained at 0.68 bar. The alteration in the appearance of GG-CH-g-PAN/AA from liquid gel to rigid polymer was achieved at the time difference between 120 minutes and 720 minutes, respectively. Expansion of polymer was noticed at high temperature due to excessive imbibition, meanwhile at low temperature for the same polymer showed shrinking behavior due to syneresis. Polymer GG-CH-g-PAN/AA exhibited 99.98% water permeability reduction and resulting to a significant characteristic as polymeric water shut off fluid.

Keywords: *Guar Gum, Chitosan, Water Shut Off Fluid, Water Control*

INTRODUCTION

Recovery of oil in petroleum industries plays essential roles to maintain oil production in petrochemical industries. Most field operators followed three stages of oil recovery, which are primary recovery, secondary recovery, and tertiary recovery. Primary recovery is commonly known as the natural force of water that helps to extrude oil out into channels bores. However, once the oil production is getting lesser, natural water force depletes. At a timeframe, the secondary recovery process has been introduced. Water injected to create a force for oil to elevate into bores channels. It is called water flooding process. However, for the time being, water and oil mixed aggregately and loss its heterogeneity. Water flooding process can cause damage via water oil hetero-aggregation, fractures or channels result from high water permeability and high water ratio to oil. Thus losses are common and gave negative impacts to oil producers.

At that juncture, tertiary recovery was introduced by using the injection technique to tackle small channels, fractures or pores with high water permeability. Usually, the type of polymers was selected to accomplish this recovery. Polymer injection technique provides a better solution to improve water-oil heterogeneity and minimize water production. The oil recovery injection method includes gel, particle, resin, or foam system were used worldwide. Among them, the polymer-based gel was the preferred method due to its effectiveness, promising result, flexible solidification time and controlled strength (Sydansk and Southwell, 1998). Emphasizing on polymer gelation system, the gel was engineered by the idea of grafting biopolymer with the hydrophobic polymer by using an oxidizing agent. Biopolymer such as guar gum, lignin, and cellulose have been used widely in oil gas industries for years (Place and Thomas, 2015). However, the field test indicated the degradation of macromolecules as results of the free radical attack (Mittal *et al.*, 2014). Biodegradation upon bacteria attack problems become the next concerned (Thombare *et al.*, 2016).

Therefore, industries are well known to dismiss or at least to avoid the addition of problems in polymer solution system. These are due to cost efficiency such as workforce, time and material consumption were premeditated. Guar gum is a long polysaccharide that widely used in oil and gas industries due to its potential as backbone polymer. As guar gum by itself cannot maintain long-term polymer durability, thus give an idea to combine guar gum to a synthetic polymer to obtain composite polymer for better stability, durability, molar change which could meet one's requirement (Kalia and Sabaa, 2013). It has been found that synthetic polymer-based gel has potential to work at in low or high reservoir temperature. Guar gum grafted acrylonitrile (GG-g-PAN) has been synthesized by the various combination of polymerizations, for example okra grafted acrylonitrile (Mishra and Pal, 2007), chitosan grafted acrylonitrile (Singh *et al.*, 2004), guar gum grafted acrylic acid (Shruthi *et al.*, 2016) and guar gum grafted acrylonitrile (Thimma *et al.*, 2003).

In the present study, guar gum was blended with chitosan and combined with acrylonitrile and acrylic acid with the presence of oxidizing agent to improve the composite polymer to become water shut off solution. The composite polymer was improvised by the addition of acrylic acid as polymer filler and chitosan solution as time delay agent. The physical-chemical parameters, namely the effect of temperature, gel ageing effect, and salinity and pH on different composition, were investigated.

MATERIAL AND METHOD

Guar gum in the form of powder having the range of molecular weight of 50,000-8,000,000 was used as the water-soluble polymer for carrying experimental progress. Acrylonitrile (AN) and Cerium Ammonium Nitrate (CAN) were obtained from a Malaysian's manufacturer (RandM Chemical Sdn. Bhd.). Acrylic Acid, the polymer strengthener while Chitosan was received in flakes form. Hydrochloric acid which was also purchased from the same local supplier.

Polymer Preparation Method

Three types of stock solution were prepared prior to combination blends process. The stock solution for Guar Gum (GG) was obtained by dissolving GG powder with 1000ml the required solvent to get 1,000ppm. A stock solution of CAN was prepared for 100ml solution by dissolving CAN powder with 1M Nitric Acid to get 0.7M. The third stock solution was prepared by dissolving Chitosan (CH) flakes with 10ml of hydrochloric acid (HCl) and stirred to get 20,000ppm. CH concentrated, then, diluted to 1,000ppm concentration.

The samples was prepared under ambient atmosphere and temperature in 10 ml glass tube. In typical reaction, 5ml GG solution was measured. Then, 1ml CAN solution ware added at 0.7M concentration. Later, AN were added at 4v%. Later, 4v% of AA and CH added last with 20v%. Vigorous shaken blends then placed into oven at temperature ranging of 60◦C to 110◦C. Reaction growth is recorded and categorized into gelation time and rigid gel time. Table 1 gives the list of different polymer carrier solvent.

Gelation time test

Gelation time test was conducted by observing the formation of gelation from the mixed solution using coding system as shown in Table 2. The observation starts from the beginning with every 15 minutes interval until the polymer exceeds Code 1. The observation was done by tilting the sample bottle method to see polymer transition from liquid to solid. The point at which polymer appeared in gel form was known as Gelation Time (GT) and the time which polymer were hard n stayed upon tilting known as Rigid Gel Time (RGT). Two important code to be noticed which were the time of polymer gel started to develop (Code 1) and time when polymer gel was rigid and attached to bottles' wall (Code 3). The code is inspired by Syndansk's code which has been altered to suit the present polymer characteristic. The codes are as below:

Table 1: Concentration of salts and pH level

Sample	Solvent	Concentration	Sample	Solution	pH
1	DI water	-	8	NaOH	8
2	KCl	2v%	9	NaOH	10
3	KCl	6v%	10	NaOH	12
4	KCl	10v%	11	HCl	2
5	NaCl	2v%	12	HCl	4
6	NaCl	6v%	13	HCl	6
7	NaCl	10v%	14	Synthetic Seawater	-

Determination of Polymer Strength

Polymer strength (PS) is an important factor in developing the polymer gel. To understand the valid composition of polymer and its strength, the polymer was tested using the set-up as shown in Figure 2. This experimental set-up was meant to determine the performance of polymer against suction pressure. First polymer tear was recorded as polymer strength. This simple observation was also done by Dai *et al.*, (2014).

Table 2: Bulk gelation time coding system

Code	Bulk studies	Notation
0	Blank	Good injectivity
1	Cloudy, low viscosity	Filtration, high pressure
2	Cloudy, high viscosity	Filtration, high pressure
3	Rigid gel	Complete blocking

Polymer growth confirmation by conductivity test

Growth of composite polymer was studied to by recording the time of polymer transition from liquid solution to solid gel. This confirmation test was carried by using conductivity test by using HI9829-03042 Multi-parameter from Hanna Instruments. The idea of this test is to measure free flow charges usually in water as medium and also other dissolved chemicals components. The conductivity of polymer was measured from liquid solution into harden polymer and were observed for 24 hours.

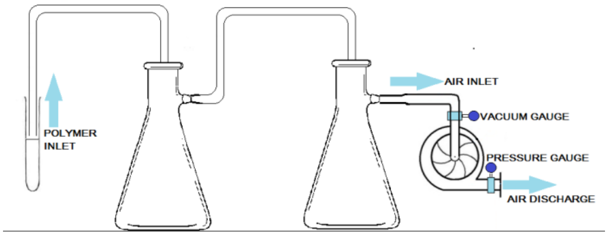

Figure 2: Diagram of polymer strength evaluation test (Dai et. al., 2014)

Viscosity test

Polymer viscosity was tested by using Brookfield Engineering Programmable LVDV II+ Viscometer. This polymer is tested to confirm gelation time by inspired Syndansk's tilting method. The polymer solution was placed in the water jacket at the temperature of 60◦C and viscosity of the polymer was recorded for every 10 minutes time interval. The recorded data continued until polymer reaching Code 1. Further then Code 1, viscosity is not applicable due to water separation from the polymer at the time of polymerization progress.

Rheology test

The rheological behaviour of polymer has been analysed by using HR-2 Discovery Hybrid Rheometer. Sinusoidal shear stress was measured to monitor the performance of cured polymer and gel effect without affecting the 3D structure of the gel. The cured polymer was tested to determine the characteristic of synthesized polymer at 60◦C.

Polymer expansion evaluation study

The cured polymer then will undergo expansion test to evaluate the effect of shrinkage or expanding polymer by time being under given condition such as salts contact, temperature and acidic or alkalic contact. These cured polymers were set in its present condition and observed until 45 days. Data was collected by measuring the length of polymer either loss or gain. These data are presented in percentage.

Permeability Study

Permeability reduction is the aimed results for confirming the reliability of water shut off fluid. Therefore, the polymer was designed for water permeability reduction. Figure 3 illustrates the diagram of core flooding rig for permeability study. The procedure started with water flooding through Berea sandstone core with assigned pressure condition until water discharge was stable. The polymer solution was then injected by same pressure condition with 10 pore volume (PV). Later on, the polymer saturated core was placed in the oven at 60◦C temperature for 72 hours for the purpose of polymer curing. Then, cured polymer core was then being flowed with water again from opposite core direction at same pressure condition. Pressure depletion was recorded by 24 hours gap until there was a noticeable pressure drop.

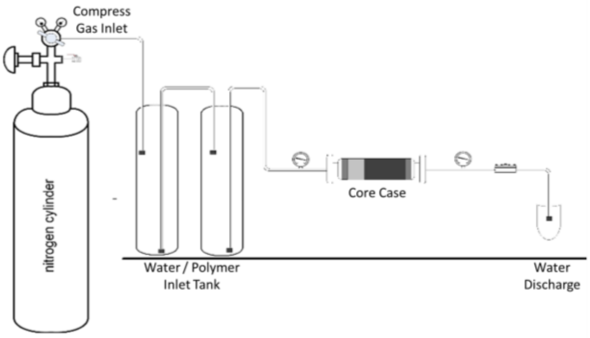

Figure 3: Core flooding rig

RESULT AND DISCUSSION

Polymer synthesized with the present of different salts

The effects of synthesized polymers in different solvent were recorded on notation of gelation time (GT), rigid get time (RGT) and polymer strength (PS). Table 3 shows the results of polymer gelation time, rigid gel time and polymer strength in different solvent at 60◦C.

Table 3: Polymer Gelation and Polymer Strength

Sample	Solvent Composition	Gelation Time (m)	Rigid Gel Time (m)	Polymer Strength (bar)
1	Distilled water	120	720	0.68
2	2% KCl solution	90	3660	0.52
3	6% KCl solution	150	3690	0.50
4	10% KCl solution	600	3760	0.50
5	2% NaCl solution	90	600	0.68
6	6% NaCl solution	105	630	0.68
7	10% NaCl solution	120	700	0.68
8	pH 8, NaOH solution	120	600	0.68
9	pH 10, NaOH solution	150	600	0.65
10	pH 12, NaOH solution	3760	4000	0.06
11	pH 2, HCl solution	180	600	0.68
12	pH 4, HCl solution	210	700	0.68
13	pH 6, HCl solution	230	1880	0.48
14	Synthetic Seawater	120	1880	0.58

Different behaviour of polymer in the presence of other chemical compounds during blending was observed. Sample 1 was taken as blank sample for the rest of solvent composition. The desired GT, RGT and PS for this experiment were 120 minutes, 720 minutes and 0.68 bar respectively which similar to Sample 1. From Table 3, it was observed that, the presence of NaCl compound, low alkalic solution and high acidic solution did not weaken the composite polymer strength. However, the period of achieving the desired GT and RGT were shifted to be faster or slower depending on the ionic interruption during polymerization. In the presence of KCl compound, high alkalic solution and low acidic solution, the period taken and polymer strength were interrupted. From these results, the present of other chemical compounds had to be understood in order to adjust the period and pre-treatment approaches during polymer injection into water streaks wellbore.

Polymer growth confirmation by conductivity test

Synthesized guar gum polymer was tested to understand the performance and period taken for polymerization. The polymerization time is a crucial factor in developing polymeric water shut off fluid. In this work, conductivity test had been performed to investigate the growth of synthesized polymer. The decrease in charges indicated polymer solution turned into solid by time. Figure 4 shows the effect of polymer conductivity with time. The results can generally be divided into four section. Section 1 shows increasing charges of conductivity which may indicate the initiation of polymerization sites by the presence of oxidant. Line 1 was the gelation time indicated as Code 1 (Table 4), where first gel formation appeared.

Section 2 was indicated by the fluctuation of conductivity reading. The fluctuation might cause from the formation of gel polymer and its reaction. Line 2 is the end point of polymer fluctuation.

Section 3 is illustrated by stable decrement in conductivity values. This indicated polymerization process was final and water absorption process took place. At this time water absorption may cause water charges to be lesser and trapped inside polymer structure. Line 3 indicates the RGT from observation which polymer hardened.

Section 4 showed the conductivity value uniformly drops and was continued for 24 hours. These free charges expected to increase and fluctuate in a time range then decrease due to initiation, propagation and termination step in polymerization, and absorption of water charges by polymer charges which turn into the solid gel.

Viscosity of composite polymer

Figure 5 showed viscosity progression during polymerization of composite polymer. Composite polymer showed polymer growth was insignificant in term of viscosity increment at Code 1 until 120 minutes to 150 minutes. Then, after 150 minutes, polymer growth were noticeable due to the increase in viscosity. It was noticed that harden polymer was measured at 120 cP. The notation for polymer were tabulated in Table 4.

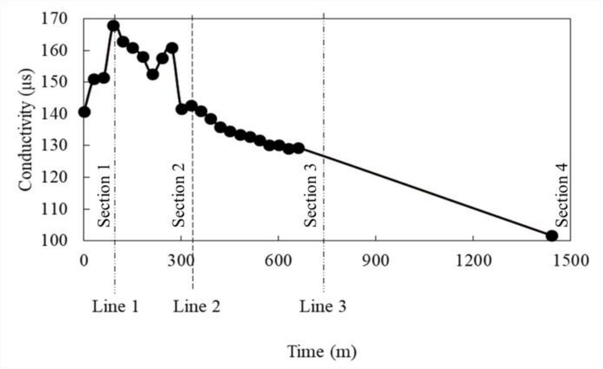

Figure 4: Effect of polymer conductivity with time

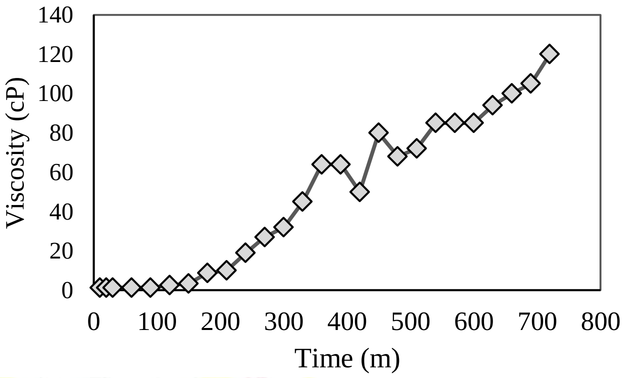

Figure 5: Viscosity pattern of grafted guar gum by time

Rheology characteristic of the composite polymer

Rheology study of the polymer is an important factor to engineered the designed polymer. This is to ensure polymer is strong enough to hold its network and its shape to withstand robust flow (high stress) condition inside the wellbore. Storage and loss modulus are the stress response for a visco-elastic fluid in oscillatory shear. Usually, storage modulus (G') is indicating sturdiness of polymer to overcome torque and maintaining its original shape, and loss modulus (G”) is the viscous respond which indicates the flowing behaviour of polymer. Figure 6 shows the G’ and G” of synthesized polymer at 60◦C. The result reveals that the G’ of composite polymer is higher compare to G”. This indicated that the synthesized polymer gave elastic behaviour rather than viscous behaviour. By increasing angular frequency, polymer elasticity increased. This findings is good since the composite polymer able to retain its structure at higher shear force.

These visco-elastic property were determined by calculating applied strain, □, of $\tan^{-1}$ (loss modulus/elastic modulus). The range of visco-elastic properties was between 0◦ until 90◦, where 0◦ was an ideal elastic solid and 90◦ is an ideal Newtonian fluid. The reading of CHS/GG-g-PAN/AA was around 0.3. the stability of composite polymer were aligned with findings from few researchers such, the acrylic acid (AA) and Chitosan (CH) which act as filler (Jamaludin and Hashim, 2011) and time delay agent (Zheng *et al.*, 2016), which proven aid of these two polymers may strengthen the composite polymer (Kundu *et al.*, 2015) with time targeting polymerization process.

Table 4: Bulk gelation, notation and the correlation with viscosity

Code	Bulk studies	Notation	Viscosity Build up (cP)
0	Blank	Good injectivity	1
1	Cloudy , low viscosity	Filtration, low pressure	2.56
2	Cloudy, high viscosity	Filtration, high pressure	64
3	Rigid gel	Complete blocking	120

These visco-elastic property were determined by calculating applied strain, □, of $\tan^{-1}$ (loss modulus/elastic modulus). The range of visco-elastic properties was between 0◦ until 90◦, where 0◦ was an ideal elastic solid and 90◦ is an ideal Newtonian fluid. The reading of CHS/GG-g-PAN/AA was around 0.3. the stability of composite polymer were aligned with findings from few researchers such, the acrylic acid (AA) and Chitosan (CH) which act as filler (Jamaludin and Hashim, 2011) and time delay agent (Zheng *et al.*, 2016), which proven aid of these two polymers may strengthen the composite polymer (Kundu *et al.*, 2015) with time targeting polymerization process.

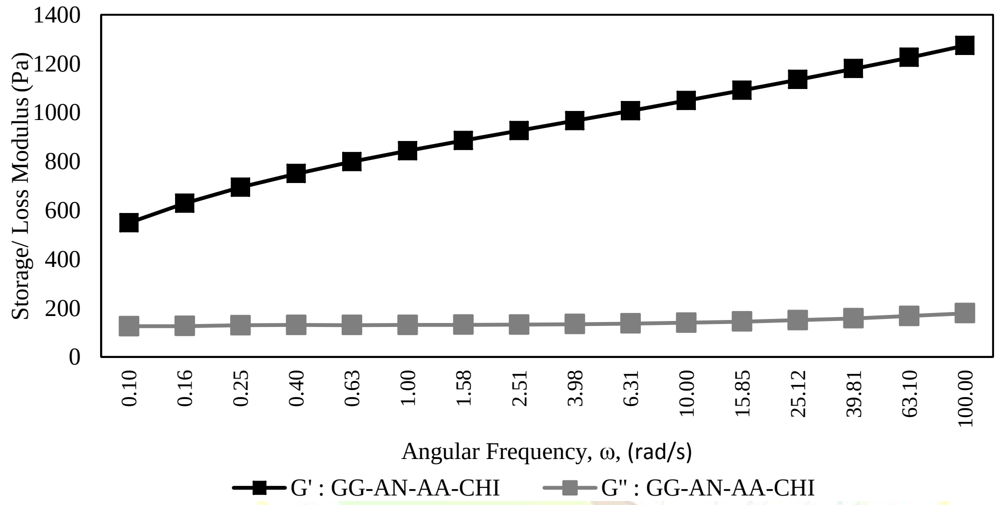

Figure 6: Storage modulus and Loss modulus of GG-CH-g-PAN/AA

Expansion test of polymer in contact with different salts and pH

Expansion evaluation is a test to study the ability of a polymer to withstand its structure with different salts contact and pH imbalance once it cured. This test is aimed to prove that GG-CH-g-PAN/AA can be a good polymeric water shut off fluid due to its stability toward external factors. Effect of salts was investigated to understand the reaction of polymer towards the presence of salt solution in a carrier medium. Two different salts solutions were set in contact with the cured polymer for 180 days. The negative value in percentage indicating shrinkages of the polymer by time and the positive value indicating expansion of polymer by time. It was known that the ideal efficient polymer, was the one that inert to external factors. However, this was very hard to achieve. Due to the molarity of water inside polymer, there will be some changes that are preferable to avoid.

Figure 7 shows the results of expansion test for composite polymer in contact with different KCl solution concentrations at different temperature for 180 days. From the results, GG-CH-g-PAN/AA has experienced shrinkages at 60◦C much obvious for all KCl concentration compared to higher temperature. For 85◦C, there were slight decrease in shrinkage percentage and noticeable expansion at higher KCl concentrations. However, at 110◦C, the polymer were experienced imbibition which polymer tend to expand from initial length for all condition.

Imbibition factor does give some pros and cons for polymeric water shut off fluid. It can give good impact by sealing polymer pores much deeper thus hinder water movement affecting areal sweep. However, in this current experiment, viscous respond were noticed at high temperature. This can cause the polymer to loosen its visco-elastic characteristic due to its excessive expansion thus loosen its 3D structure which later, water may sweep the polymer bond by continuous water flooding. Thus, this can cause thermal polymer degradation and polymeric water shut off are ineffective anymore.

Figure 8 shows the expansion effects when in contact with different NaCl solution concentrations at different temperature. It was noticed that when in contact with NaCl, composite polymer gave less polymer interruption compared to it when in contact with KCl. At 60◦C and 85◦C, composite polymer experienced only shrinkage rather than imbibe. However, at 110◦C, when in contact with water and 2% NaCl the composite polymer showed imbibition effect. The presence of NaCl solution was noticed to curb excessive imbibition of composite polymer.

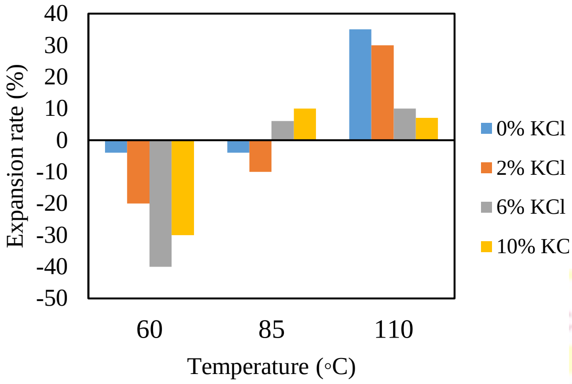

Figure 7: Expansion test of composite polymer in contact with different KCl solution concentrations at different temperature for 180 days.

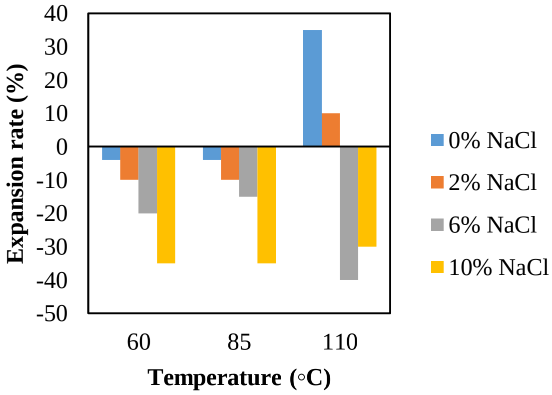

Figure 8: Expansion test of composite polymer in contact with different NaCl content at different temperature for 180 days.

The effect of pH was investigated to understand the polymer reaction toward different pH level when in contact with the composite polymer. Figure 9 shows the results of expansion test for GG-CH-g-PAN/AA at different pH and temperature. Shrinkages were unnoticeable at low temperature, 60◦C and medium temperature, 85◦C for all pH. However, at high temperature, 110◦C, composite polymer experienced excessive expansion at all pH. The imbibition process was obvious which can be considered polymer lost its shape. Water sweeping may cause the polymer to degrade further by the presence of different pH. At high temperature, 110◦C, this composite polymer was found stable in the acidic condition which was around pH 2 compare to the one in contact with water. It was noticed that, high acidity solution can curb polymer to excess imbibe by the hindrance of H^+ ions from water ions attach to polymer sites. This was also lead to temperature effects on polymer were curb due to strong ionic hindrance.

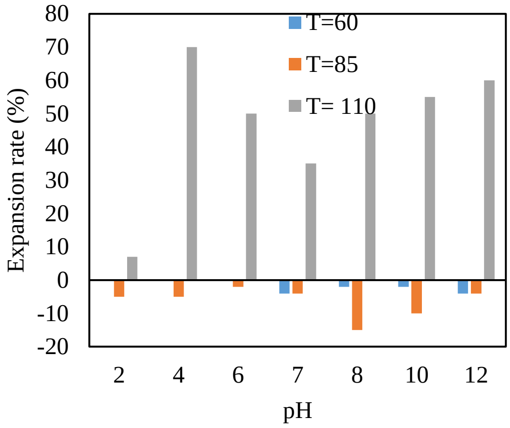

Figure 9: Expansion test for GG-CH-g-PAN/AA at different pH and temperature.

Permeability Study

Permeability study of GG-CH-g-PAN as water shut off fluid was investigated using the same amount of GG, AN, AA, CHS and CAN as the previous tests. The experimental investigation was presented in Table 5. The initial permeability was 9.3107 Darcy. After flooding with 2% KCl brine for 4 hours at constant pressure, 20 psi until brine discharge was stable, composite polymer solution was injected with 10PV for 2hours with the contact pressure of 20 psi to ensure fully saturated core with the polymer solution. Next, the saturated polymer was cured for 3 days to ensure full polymerization were completed. After in situ gelation, brine was flooded backward at 20 psi with 10PV as well. The initial pressure is set at 20 psi, and further noticeable pressure drop showed.

Table 5: Effect of permeability on Berea sandstone core

Porosity	28%
Initial permeability	9.3107 Darcy
Post gelation permeability	0.0019 Darcy
Permeability reduction	99.98
Resistance factor	29.5
Residual resistance factor	0.0075

It was observed that permeability of polymer saturated core was reduced to 0.0091 Darcy, with the percentage of permeability reduction of 99.98% and the resistance factor was about 29.5 was reduced to 0.0075. There were huge different in permeability by the present of polymer, indicated that GG-CH-g-PAN/AA composite was an excellent candidate to be as polymeric water shut off fluid. The ability and stability of polymer as water shut off fluid were proven to be able to seal water streaks and pores which can be applied in oilfield industries (Singh and Mahto, 2016).

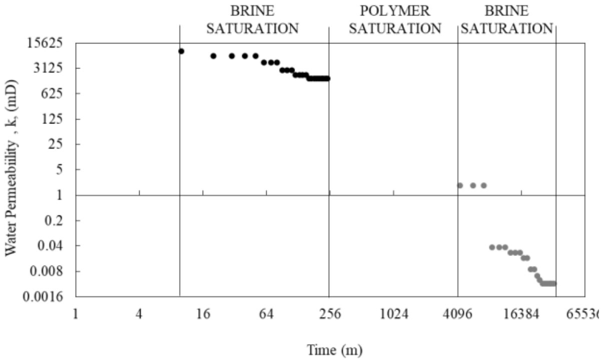

Figure 10: Permeability reduction pattern of GG-CH-g-PAN/AA by time

The experimental works performed for GG-CH-g-PAN/AA composite gave a positive result and proved the designed composite is suitable to be water shut off fluid at the temperature of 60°C. The polymer strength of effectively well-developed composite was at 0.68 bar for GG-CH-g-PAN/AA along with the optimal gelation time (GT) at 120 minutes and the rigid gel time (RGT) was at 720 minutes. For the syneresis effect of the composite polymer, synthesized GG-CH-g-PAN/AA did not experience much syneresis or further imbibition effect when in contact with different salts solutions at different concentration. However, at high temperature, polymer composite gave less syneresis impact compared to the lower temperature. Furthermore, synthesized polymer composite gave effects due to the imbalance of pH value. Expansion of polymer were noticed at high temperature due to further imbibition while, at low temperature the composite polymer experienced shrinkage due to syneresis. Effect of polymer expansion was more obvious due to pH imbalance compared to salts solution concentration at high temperature. The composite polymer showed almost 100% permeability reduction. Thus, this GG-CH based composite polymer could be a good candidate as polymeric water shut off fluid.

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