THE EVALUATION OF POLYMERS PERFORMANCE AS FLUID DIVERSION GELLING AGENTS IN MATRIX ACIDIZING

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Abstract. This paper discusses the use of diversion mechanism in matrix acidizing to increase the flow resistance in highly permeable regions due to the presence of polymer temporary plugs. In this study, three polymers, namely hydroxyethyl cellulose (HEC), hydroxypropyl guar (HPG), and xanthan gum were evaluated in the laboratory to determine the best diverting agent to enhance the performance of matrix acidizing. An acidizing system was set-up using coreflooding techniques where Berea sandstone cores saturated with Sarapar were damaged by oil-based mud. Treatment fluids used were mud acid (3% HF – 12% HCl), HCl, polymer gels, and ethylene glycol monobutyl ether (EGMBE) as the mutual solvent. Polymer solutions were prepared at 40 lb/Mgal (4793 ppm) as per field formulation. The experimental results revealed that at low injection pressure, mud acid without diverting agents furnished the best performance where the improvement ratio achieved was 4.88 at 50 psi (344.7 kPa), compared with improvement ratio of 2.42 for HEC, 2.66 for HPG, and 1.35 for xanthan gum. However, at higher injection pressure, mud acid with diverting agents gave better results, where at injection pressure of 100 psi (689.5 kPa), mud acid with HEC diverting agent produced the best result with improvement ratio of 4.88 compared to 1.80 for mud acid without diverting agents. Generally, at low injection pressure, mud acid is found to stay longer in the core and has sufficient time to dissolve mud particles. Nevertheless, low injection pressure produces insufficient force to push acid deeper into the formation and fails to dissolve the viscous diverting agents completely. At high injection pressure, there is sufficient force for the acid to dissolve mud particles and viscous diverting agents, hence the diverting agents served its purpose by diverting the mud acid into low permeability zone.

Keywords: Diverting agents, fluid diversion, formation damage, improvement ratio, matrix acidizing

Abstrak. Kertas kerja ini membincangkan tentang penggunaan mekanisme pelencongan dalam pengasidan matriks bagi meningkatkan rintangan terhadap aliran di kawasan berketer talentedan tinggi, berpuncu daripada kehadiran palam sementara polimer. Dalam kajian ini, tiga jenis polimer iaitu selulosa hidroksitil (HEC), guar hidrokspropil (HPG), dan gam xanthan dinilai keupayaan masing-masing di maksimal untuk menentukan agen lencong terbaik dalam meningkatkan prestasi pengasidan matriks. Sistem pengasidan dibentuk menggunakan teknik banjir teras berdasarkan teras barat pasir Berea ditupuk dengan Sarapar sebelum dirosakkan menggunakan lumpur dasar minyak. Bendalir perawat yang digunakan ialah asid lumpur (3% HF – 12% HCl), HCl, gel polimer, dan etilena glikol monobutil eter (EGMBE) sebagai pelarut saling. Larutan polimer disediakan pada kadar 40 lb/Mgal (4793 ppm) berdasarkan rumusan median. Keputusan uji kami membuktikan bahawa pada tekanan suntikan yang rendah, lumpur asid tanpa agen lencong menghasilkan prestasi

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1.0 INTRODUCTION

In petroleum industry, one of the most unpopular phrases among the petroleum engineers is formation damage. Generally, formation damage can restrict or prevent effective depletion of formation fluids from the reservoir. Contact with a foreign fluid is the main cause of formation damage. This foreign fluid may be drilling mud, completion or workover fluid, stimulation or well treating fluid, or even reservoir fluid itself if the original characteristics are altered. Laboratory research done by Krueger [1] found that most of the field operations, namely well drilling, well completion, production, and well stimulation are potential sources for formation damage.

There are various methods used to combat formation damage. One of them is acidizing. The oil and gas industry has been acidizing oil and/or gas bearing formations since the 1890s [2]. Acids may be used to reduce damage near the wellbore in all types of formations. Inorganic, organic, and combinations of these acids are used in variety of well stimulation treatments.

There are two types of acid job, namely matrix acidizing and acid fracturing [3]. In this study, the acidizing method chosen was matrix acidizing. Matrix acidizing involves the use of acid injected below fracture pressure. It is normally used for the removal of skin damage associated with workover, well killing or injection fluids, and precipitation of scale deposits in tubular, the wellbore, or within the formation.

Removal of near wellbore damage can result in significant stimulation, in some cases may reach three folds [1]. Treatment normally involves injecting mud acid (3% HF – 12% HCl) followed by a sufficient afterflush or water or hydrocarbon to clear all acids from well tubular. A corrosion inhibitor is added to the acid to protect tubular during exposure to acids. Other additives, such as anti-sludge agents, iron chelating agents, de-emulsifiers, and mutual solvents are added as required for a specific function.
According to Arcasolve Technical Document [3], matrix acidizing may also be used to increase formation permeability in undamaged wells. The aim of this treatment is to achieve a radial acid penetration deep into the formation to increase the formation permeability around the wellbore. Deep penetration can only be achieved with retarded acid system.

In undamaged formation, even significant permeability increases over three to six meter radius around the wellbore will result in less dramatic stimulation than achieved when removing damage. There is a practical limit of about 50% increase in injectivity or productivity of undamaged oil or water wells that can be achieved by matrix stimulation.

Again, according to Arcasolve Technical Document [3], successful matrix acidizing of zones longer than 10 ft (30.48 m) must include a mean of diverting the acid so that it may enter all permeable zones. Since solids from the drilling mud have invaded the most permeable zone, it is likely to be the most difficult zone to treat with acid. If a diverting system is not used, the most productive zone may remain partially plugged with solids.

The type of the gelled acid chosen has to be compatible with the formation that is to be treated. Incompatible gelled acid would cause secondary formation damage. The use of highly viscous solution would cause plugging of formation that may worsen the existing damaged conditions and affect injectivity. Besides, acid slug size must also be taken into consideration. If the slug size is too big, it can affect the permeability of the formation. If it is too small, particles tend to flow through the porous zone and diversion would not occur. Other parameters such as formation pressure, temperature, and salinity also affect the performance of gelled acid.

In this study, the performance of three water-soluble polymers, namely hydroxyethyl cellulose (HEC), hydroxypropyl guar (HPG), and xanthan gum as diverting agents was evaluated in the Drilling Engineering Laboratory of Universiti Teknologi Malaysia. These polymers would form a viscous based fluid to control the mobility of the flowing acid and divert it to the low permeability zones. Careful observation and measurements in the laboratory were done before and after acidizing process to identify changes in permeability of the Berea sandstone core samples. The effects of injection pressure and exposure time were also studied based on the permeability ratio obtained.

2.0 MATERIALS AND EXPERIMENTAL PROCEDURES

Acids used in this study were hydrochloric acid and hydrofluoric acid. The HEC, HPG, and xanthan gum polymers and the mutual solvent, ethylene glycol monobutyl ether (EGMBe) were supplied by Halliburton Energy Services. Polymer gels were formulated to a concentration of 40 lb/Mgal (4793 ppm). Sarapar, the synthetic colourless base oil was used as the base fluid.
The cores used in this study were Berea sandstone cores with dimensions of 1 inch (2.54 cm) in diameter and 1.3 inch (3.30 cm) in length. The cores had a bulk volume of 1.02 inch (16.73 cm³) and the average porosity was 20%.

2.1 Experimental System

Figure 1 shows the schematic diagram of the experimental system. The system comprised a nitrogen gas line, a mud cell, 3 mm stainless steel tubing, a core holder, valves, and pressure gauges.

Based on the experimental set-up, the flow system was operated in a counter-current flow method. The injected fluid was introduced on shell side and the permeating stream was collected at the bore side of the core holder. The direction of Sarapar injection was considered as the production direction, and the direction of treatment fluids injection such as hydrochloric acid, mud acid, polymer gels, and mutual solvent was considered as the injection direction. Oil-based mud used to damage the cores was also injected in injection direction.
2.2 The Preparation of Oil-based Mud

In this study, the Berea sandstone core samples were damaged using oil-based mud. The base oil (Sarapar 147) and mud additives were supplied by Shell MDS (Malaysia) and Kota Minerals & Chemicals (KMC Sdn. Bhd.), respectively. Mud was formulated as per field formulation. The compositions of drilling mud used to damage the core samples based on 1 lab barrel (1 lab bbl = 350 cc) are given in Table 1.

<table>
<thead>
<tr>
<th>Material</th>
<th>Quantity</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sarapar 147</td>
<td>242.0 cc</td>
</tr>
<tr>
<td>Versamul</td>
<td>5.0 ppb</td>
</tr>
<tr>
<td>Versacoat</td>
<td>2.0 ppb</td>
</tr>
<tr>
<td>Lime</td>
<td>5.0 ppb</td>
</tr>
<tr>
<td>Drill water</td>
<td>60.6 cc</td>
</tr>
<tr>
<td>Calcium chloride</td>
<td>15.0 ppb</td>
</tr>
<tr>
<td>Visplus</td>
<td>6.0 ppb</td>
</tr>
<tr>
<td>Versatrol</td>
<td>5.0 ppb</td>
</tr>
<tr>
<td>Barite</td>
<td>170.0 ppb</td>
</tr>
</tbody>
</table>

Note: ppb means pound per barrel, and cc means cubic centimeter

2.3 The Preparation of Polymer Solutions as Diverting Agents

Polymer solutions were prepared strictly as per specifications given by Halliburton [4]. This is to ensure that the polymer solutions achieved the field concentration and properties. The procedures are as follows:

(i) The concentration for each polymer solution was fixed at 40 lb/Mgal (4793 ppm). To achieve this, 1.82 grams of each polymers was diluted into 455 cc of distilled water.

(ii) The polymer powders were added slowly to avoid the formation of fish eyes [5].

(iii) For each of the polymers to form gel, it has to achieve its hydration pH. The hydration pH of HEC, HPG, and xanthan gum are 9, 5, and 8, respectively.

(iv) For HEC and xanthan gum, sodium hydroxide (NaOH) was added until the hydration pH was achieved. For HPG, however, hydrochloric acid was added until the hydration pH was achieved.

(v) Once the hydration pH was achieved, the polymer solution gelled up. The rotor of the mixer should be maintained at normal speed throughout the preparation of the polymer solution to avoid the formation of fish eyes.
The rheological properties of the polymer solutions were determined using a Fann Rheometer at 300 rpm.

2.4 Acidizing the Core Sample

After damaging, permeability of the core was determined prior to injecting gelled acid into it. In this study, the injection pressure was varied to determine the effectiveness of matrix acidizing using gelling agents. The direction of acid injection was in the same direction of mud injection. The flowrate of acid was determined by taking the ratio of volume to the time taken to achieve the volume. Total volumes of acid used were 10 pore volumes or 33 cc [6, 7]. The acidizing procedures are as follows:

(i) The Berea sandstone core sample in the mud cell was filled with preflush, which was 5% HCl.
(ii) After all the valves were closed, confining pressure was applied, then followed by injection pressure. Injection pressure was varied from 25 (172.4 kPa) to 125 psi (861.8 kPa). The confining pressure was twice the injection pressure.
(iii) Preflush of 15% HCl was flowed into the core. For the first stage of acidizing, 5% of total volume or 2 cc was used.
(iv) The flowrate of the preflush was recorded.
(v) Steps (i) to (iv) were repeated with (a) mud acid (3% HF – 12% HCl), (b) polymer gel, (c) mud acid at a volume of 15% of total volume or 5 cc, and (d) afterflush (15% HCl – 10% v/v EGMBE).
(vi) For the second stage of acidizing, steps (i) to (v) were repeated with (a) a volume of 30% of total volume or 10 cc, and (b) a volume of 50% of total volume or 17 cc.

2.5 Determination of Improved Permeability and Improvement Ratio

After the cores were treated with acid, permeability of the cores was re-measured (known as improved permeability, $k_{imp}$). The base fluid used in measuring the permeability was Sarapar 147. To determine improved permeability of the cores, similar procedures of determining the initial permeability ($k_i$) and damaged permeability ($k_d$) were used. All the permeability values were calculated using Darcy’s equation (Equation (1)). Once the improved permeability has been determined, the improvement ratio can be calculated using Equation (2).

$$k = \frac{qL}{A\Delta p}$$

(1)
The improvement ratio equation is given by:

$$\text{Improvement ratio} = \frac{k_{\text{imp}}}{k_d}$$  \hspace{1cm} (2)

From the improvement ratio equation, if the ratio is greater than unity, there is an improvement in the permeability after acidizing. However, if the improvement ratio is less than unity, it shows that the acidizing process fails to remove the damage.

### 3.0 RESULTS AND DISCUSSION

The experimental results discussed comprise the effect of concentration on gel viscosity, the effect of pH towards viscosity, the effect of injection pressure on improvement ratio, and mud acid with diverters.

#### 3.1 The Effect of Concentration on Gel Viscosity

The most important property of a polymer solution for oil field use is its viscosity. Measurement of viscosity under conditions of shear rate, temperature, and polymer concentration appropriate to actual field use is important. Likewise, the viscosity comparisons of polymer solutions must be done under similar conditions.

Figures 2, 3, and 4 clearly reveal that the viscosity of gel increases with concentration of polymers. Increasing concentration of polymer means more polymer powder is added into water. Gel is a long chain polymer that hydrates in water at proper pH values. The polymer itself is a long chain of monomers attached with hydrogen bonds. By forming gels, the water molecules can no longer move freely but move relative to other polymer clusters. Increasing polymer concentration or the number of polymer clusters will increase the resistance to flow, hence, resulting in increase of viscosity [8].

![Figure 2](image.png) Viscosity versus concentration for HEC gels
Generally, different polymers give different viscosity values. HEC shows the highest viscosity values at 36 cp (0.036 Pa.s), followed by HPG at 24 cp (0.024 Pa.s) and xanthan gum at 32 cp (0.032 Pa.s) at a concentration of 40 lb/Mgal (4793 ppm). According to Fried [8] and Rahmat [9], the viscosity of polymer gels increases with the molecular weight of the polymer. The increase in molecular weight is caused by the increase in the number of atoms in the chain. Larger chain means longer structure. Thus it becomes harder to flow, hence, increase in viscosity.
3.2 The Effect of pH towards Viscosity

The polymer solution is pH sensitive. It only hydrates up at its hydration pH where it starts to gel up. Figure 5 shows the polymer solution gels up or hydrates at certain pH. For HEC, hydration pH is at 9, HPG’s hydration pH is at 5 and xanthan gum’s hydration pH is at 8. The experimental data was found to be in good agreement with the field values.

Polymer gels have specific pH values to hydrate due to its structure. Polymer powder diluted in distilled water will just form strands of polymer in water solution. However at the hydration pH, polar hydrogen ions will bridge the strands of polymer together forming stable polymer structures, hence increasing the resistance to flow. Generally, acidic hydration pH shows that a high concentration of hydrogen ions is needed to bridge, whereas alkaline hydration pH shows the opposite. Once stable polymer structures are formed, water molecules would move relatively against them thus it increases resistance, hence viscosity of the gel.

![Figure 5](image)

**Figure 5** Viscosity versus pH for polymer gels

3.3 The Effect of Injection Pressure on Improvement Ratio

The injection pressure was varied to determine its effect towards improving permeability of the damaged core. Since the volume of the acids and the diverting agents were the same, the difference was actually the exposure time, or the time of the fluids remained in the core.

In the acidizing experiment, only mud acid was used to improve the permeability of the damaged core. In fact, it was the reference experiment used to evaluate the effect of the polymer diverters.

Figure 6 clearly shows the improvement ratio was 3.63 at 25 psi (172.4 kPa) and increased to 4.88 at 50 psi (344.7 kPa), then steadily decreased to 1.61 at 125 psi.
This trend shows that at lower injection pressure (less than 75 psi (517.1 kPa)), improvement ratio was high and reached the optimum at 50 psi (344.7 kPa). At low injection pressure, the exposure time for acid in the core was longer, thus it gave more time for the mud acid to dissolve mud particles in the pore space. Improvement ratio increased from 25 (172.4 kPa) to 50 psi (344.7 kPa) because even though at 25 psi (172.4 kPa), the exposure time was longer than 50 psi (344.7 kPa), but there was insufficient force to push out the dissolved mud particles. At higher injection pressures (75 psi (517.1 kPa) and more), the exposure time was relatively short which gave insufficient time for the acid to react with mud particles.

3.4 Mud Acid with Diversers

The polymer diverters discussed comprise HEC, HPG, and xanthan gum.

3.4.1 HEC Diverter

HEC is a non-ionic polymer that is compatible with salts of monovalent metals (i.e. NaCl, KCl, etc.), and also with many divalent metals (i.e. CaCl\textsubscript{2}, MgCl\textsubscript{2}, etc.). HEC finds wide application in completion and workover fluids. Most fluid loss additives used in cementing is based on HEC. HEC can be split at the acetal link by acid [8]. The resulting depolymerized solution has very little residue to cause formation damage. HEC is also susceptible to enzyme degradation but not to the extent of guar gum.

In this experiment, HEC diverter was added after mud acid then acidized again. It was intended to divert the acid into the low permeable zone by reducing the mobility of the flowing acid. The HEC solution used was 4793 ppm with a viscosity of 35 cp (0.035 Pa.s). Figure 6 depicts the trend of improvement ratio using HEC diverter to enhance matrix acidizing.

Figure 6 shows that an odd curve was developed compared to the curve for mud acid only. HEC is a polymer that produces no residue after acid breakthrough. By
right, it should produce a curve similar to mud acid. The sudden decrease in improvement ratio at 50 psi was believed due to the presence of fish eyes or micro gels – a phenomenon which is difficult to avoid in the laboratory work even though the polymer was added slowly to overcome this problem. Generally, HEC has a strong tendency to form fish eyes – clumps of unhydrated polymer visible in the fluid, i.e. larger than 250 microns in size (1/100") – and also micro gels that can be described as smaller fish eyes (typically between 10 and 100 microns). Micro gels are microscopic globules of unhydrated polymer primarily formed in the manufacturing process and during storage of dry powder. Fish eyes or micro gels happen during the mixing process on the rig or in the laboratory. Caution must be exercised to avoid the formation of fish eyes or micro gels during preparation of HEC gels in the laboratory, as highlighted by references [5, 11 - 13]. In the field, the fish eyes or micro gels would disappear as fluid is circulated into the well, where temperature and pressure increase linearly with depth and coupled with the effect of turbulence.

Fish eyes form when a large clump of dry powder hydrates rapidly. Hydration of the polymer on the outside of the clump forms a shell that slows down or even prevent further penetration of water inside the aggregate to complete the hydration. In the core, this shell would prevent acid from breaking it, hence the gel remains viscous, thus it reduces the permeability ratio.

Figure 6 also shows the improvement ratio steadily increased until it achieved maximum value of 4.20 at 100 psi (689.5 kPa). This is due to sufficient force and exposure time to break the mud particles and also the viscous gel hence proper acid treatment over the core sample area. Improvement ratio decreased to 1.69 at 125 psi (861.8 kPa) because at high injection pressure, there was very limited exposure time for the acid to be effectively diverted to dissolve mud particles and viscous gel, but instead it bulldozed its way through the core sample, thus it produces lower improvement ratio.

3.4.2 HPG Diverter

Hydroxypropyl guar (HPG) is prepared by reacting highly purified guar with propylene oxide [10]. This process consists of putting guar seed endosperm splits through a series of acid and water soaks to remove most of the embryo and hull before grinding of splits. Broken HPG contains no more than 2% insoluble materials [3].

In the experimental work of using HPG diverter to enhance the performance of matrix acidizing, HPG was prepared at 4793 ppm with gel viscosity of 32 cp (0.032 Pa.s). Figure 6 depicts the improvement ratio when using HPG diverter with mud acid.

It is clear that improvement ratio was 3.70 at 25 psi (172.4 kPa), then steadily decreased to 1.94 at 125 psi (861.8 kPa). The curve shows a trend of decreasing
improvement ratio. In fact, the HPG diverter failed to produce a curve similar to mud acid without diverter or mud acid with HEC diverter. The reason is insoluble residue was left behind. This insoluble residue was formed when HPG diverter had been broken by acid. The residue left behind by HPG diverter could reach up to 2% [4]. Since HPG was less viscous than HEC, it could effectively divert acid and be dissolved at lower injection pressure. That is why at lower injection pressure, the improvement ratio is high because the exposure time for the acid was long enough for the acid to dissolve the mud particles and the viscous gel. Even though it is more effective at lower injection pressure, the optimum injection pressure was still higher when compared with mud acid without diverter. At high injection pressure, there was insufficient time for the acid to dissolve mud particles and viscous gel because the acid would just bulldoze its way through the core.

3.4.3 Xanthan Diverter

Often called biopolymer or XC polymer, xanthan gum is exuded by the microorganisms Xanthomonas Campestris [8]. Below 1000 ppm, xanthan gum solutions lose viscosity significantly in the presence of low salt concentration. Xanthan gum is quite stable in acid or in the presence of enzymes.

In this study, xanthan gum was prepared at 4793 ppm with viscosity of 22 cp (0.022 Pa.s) as a diverting agent to enhance matrix acidizing. Figure 6 depicts the improvement ratio after acidizing core samples with xanthan gum as the diverting agent.

The structure of xanthan gum is such that the break site is protected, and therefore, is not readily susceptible to acid, enzyme, or oxidation degradation. This property limits its use in fracture fluids or gravel packing fluids. This explains why the improvement ratio for xanthan gum is lower compared to other acidizing systems conducted in this study.

Figure 6 shows a similar curve as the HPG diverter. In this case, the improvement ratio was higher at lower injection pressure because the exposure time was sufficient for the acid to dissolve mud particles and viscous gel, whereas at higher injection pressure, the exposure time was insufficient for the acid to properly dissolve mud particles and the viscous gel.

 Likewise HPG diverter, xanthan gum produced an insoluble residue when broken by acid. It was found to be more severe where up to 8% insoluble residue could be formed [4]. Hence this explains why the improvement ratio is lower compared to the other two diveters and even mud acid without diveters. This statement was supported by field experience (as per Halliburton personnel) where he was strongly against the use of xanthan gum as diverter agent even though it is cheaper [13].

Generally, mud acid with polymer gels capable of diverting mud acid into the low permeability zone, thus they can produce higher improvement ratios at moderate injection pressure and also ensure an evenly treated area. In the low permeability
zone, the diverted mud acid dissolves mud particles only, whereas at the high permeability zone, the mud acid must remove polymer gel prior to dissolving mud particles. This improvement is clearly shown in Figure 6 where the mud acid with HEC at injection pressure of 100 psi (689.5 kPa) gave better results as compared to mud acid only and mud acid with HPG and xanthan gum. In fact, the improvement ratio could exceed 4.88, as achieved by mud acid only at an injection pressure of 50 psi (344.7 kPa), if fish-eyes and micro gels were not formed in the mud acid with HEC system.

4.0 CONCLUSIONS

From the matrix acidizing study performed on the Berea sandstone core samples using polymers as diverting agents, several conclusions were derived as follows:

(i) Formation damage caused by drilling mud can be treated with matrix acidizing. Nevertheless, improvement ratio can be increased through the utilization of a suitable diverter as compared to the standard mud acid system.

(ii) HEC gel is found to be the best diverter followed by HPG gels and xanthan gels, when injection pressure exceeds 75 psi (517.1 kPa).

(iii) Moderate injection pressures should be used to ensure sufficient exposure time for improvement, and at the same time high enough to push the acid deeper into the formation.

(iv) Mud acid with diverter produces higher improvement ratios at moderate injection pressure because the viscous gel diverts mud acid into the low permeability zone to ensure an evenly treated area.

ACKNOWLEDGEMENTS

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NOMENCLATURE

\[ k \] – Permeability (mD)
\[ k_d \] – Damaged permeability (mD)
\[ k_{imp} \] – Improved permeability (mD)
\[ q \] – Flowrate (cc/s)
\[ \mu \] – Fluid viscosity (cp)
\[ L \] – Core length (cm)
\[ A \] – Core area (cm²)
\[ \Delta P \] – Differential pressure across the core (atm)
rpm – Revolutions per minute
EGMGE – Ethylene glycol monobutyl ether
HF – Hydrofluoric acid
HCl – Hydrochloric acid
HEC – Hydroxethyl cellulose
HPG – Hydroxypropyl guar

REFERENCES