

BEER[®] Bio Enhanced Energy Recovery – Development of a new Stimulation Technology

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Abstract

Energy politics changed in recent years and hydraulic stimulation restrictions arose against the use of conventional fracturing fluids. This article presents a new environmentally-friendly hydraulic fracturing fluid. Laboratory results confirmed by comparison with the desirable performance properties the principles regarding the new fluid and its applicability.

The Bio Enhanced Energy Recovery (BEER[®]) fluid is composed of four main components, namely production water as base fluid, potassium carbonate as a shale stabilizer, a corrosion inhibition and weighting agent, a biopolymer for viscosity generation, and glass beads as proppants. State-of-the-art laboratory tests to evaluate the recipe composition and the capability of the new fluid, like viscosity, hydration, shear recovery, dynamic breaking, fluid compatibility, fluid loss, proppant settling, regained permeability, and proppant crush resistance, were carried out according to the relevant standards.

K₂CO₃ successfully acts as a weighting agent, sour gas buffer, and corrosion inhibitor. K₂CO₃ increases the stability of the polymer at high temperatures, will act as a clay swelling inhibitor and as a biocide.

The standard proppants are provided with a standard grain size distribution, while the glass beads size distribution can be designed on request, which leads to a much better proppant pack permeability.

The components selected for the BEER fluid have been used successfully for years in the drilling industry in various concentrations and compositions. The novelty of this article is their application in a hydraulic stimu-

lation fluid without any harmful components to the environment and humans.

Introduction

Worldwide the performance of conventional and unconventional oil, gas, and deep geothermal reservoirs needs continuous optimization to maintain and increase productivity. Unconventional reservoirs require special measures for improving the recovery factor or even kick off economic production [7]. The pore connectivity improvement in fine-grained, low permeable, detrital rocks is achieved by stimulations through hydraulic fracturing [25]. The physics of hydraulic stimulation has not changed significantly since the early 1950s. A minimum amount of the so-called pad-fluid is pumped into the wellbore to initiate a fracture and generate enough fracture width, allowing the proppants to be pumped into it. The fracture evolves perpendicular to the least stress axis by continuing pumping and follows the maximum stress direction. Thereby the proppant concentration in the pumped fluid increases. The positioning of the proppants in the created fracture is the target of the treatment. The proppants keep the fracture open, providing a high permeability to liquid or gas flow and increasing the well's drainage area. The used fluid's properties need to be adjusted, typically by using chemicals, to maintain the carrying of proppants into the fracture, to release them at the deposition position, and to allow fluid recovery without proppants at the surface [17].

The hydraulic fracturing stimulation technique is used in many countries all around the globe. The first million wells were hydraulic-

ly stimulated between 1952 and 1992, and the first million fractures in horizontal wells were carried out between 1974 and 2013 [8]. The most stimulating jobs in history were performed in the US. In 2015, 300.000 natural gas wells were stimulated by hydraulic fracturing. In 2019, 59% or 6.44 million barrels were produced by fracked oil wells [1]. Hydraulic stimulation is a well-known and evolving technology. The global energy landscape has been redrawn since the successful application of multistage hydraulic fracturing techniques in horizontally drilled wells demonstrated economic, natural gas production from low permeability formations [49].

The extraction of natural gas from low permeable formations shows some environmental and economic benefits, but the speed of the industry expansion and public discussions on the environmental impacts have caused severe concerns [21, 24, 26, 39, 42, 54, 56]. As a result, energy politics changed in the last few years. In addition to climate change, environmental concerns caused hydraulic fracturing restrictions in several European countries. In Austria, for example, serious political discussions started in 2011 [31], and the government restricted the legislation related to unconventional fracking. The stringent requirements limit the application of reservoir stimulation. Conventional fracturing fluids use chemicals for the base fluid conditioning, typically freshwater, to achieve the required properties. Acids are used for casing and formation cleaning before fracturing fluid injection. The fracturing fluid contains components to protect the downhole equipment and the formation. Biocides are used to prevent bacteria from grow-



ing, clay stabilizers to prevent clay mobilization, resulting in fracture clogging, corrosion inhibitors to protect the downhole equipment, iron control additives to prevent the precipitation of iron compounds in the wellbore, scale inhibitors, and oxygen scavengers to prevent the premature fracture fluid breakdown by binding oxygen [50]. To fulfill its objective, the fracturing fluid contains gellants to increase fluid viscosity for improved proppant suspension during transport, surfactants, crosslinkers to increase the gel viscosity, foamers/defoamers to control the foaming tendency, friction reducers, and breakers to improve fracturing fluid recovery after proppant deposition.

Canada's Petroleum Services Association reported an average freshwater consumption between 10.000 to 50.000 m³ per stimulation job for a mid-size frac [40]. The actual amount depends on geology and several other factors. In California, the required water volume is about 440 m³ [4]. The quantity of the stimulation job's flow back fluid is about 30% and is often disposed of by injecting it into geologic formations [53]. The flowback fluid consists of frac fluid, formation water, and condensates, and produced water accounts for an average of over 9.5 million m³ per day in the United States [12]. Water-based fracturing fluids compete with the drinking water supply, and severe concern about that arose. Some operators start to reuse the produced water for frac operations [15], whereas others tested oil-based frac fluids to preserve freshwater. 100% liquefied petroleum gas was used and has demonstrated complete fracture fluid recovery and cleanup in the McCully gas field in Canada [52]. A detailed analysis of its application in China has been performed [29]. Despite these developments, strategies to optimize reservoirs in an environmentally friendly way need to be found, where reservoir stimulation with innovative carrier fluids could be one way to do so. For drilling fluids, biodegradable components like grass powder, have shown advantages compared to conventional chemicals [2].

The objective of this article is to introduce the novel eco-friendly hydraulic stimulation fluid. The Bio Enhanced Energy Recovery (BEER[®]) fluid is based on biodegradable and harmless mineral components. This novel development aims to keep the fluid biodegradable and straightforward, whilst still satisfying all the needs. Two functions are requested from the fluid. Firstly it has to transfer the stimulation pumps' energy to the formation to create fractures, and secondly, the fluid has to transport and suspend the proppants. Other necessary requirements for the fluid are formation and native fluid compatibility, easy recoverability, environmentally friendly, ease to use, and a low price. A comparison with the field requirements defined by oil companies, regarding the hydraulic stimulation fluid capabilities, demonstrates the applicability of the BEER[®] fluid.

Fluid Chemistry

Many additives are used in conventional hy-

draulic stimulation fluids. In contrast, the objective for the BEER stimulation fluid is to keep it as simple as possible. A powerful fluid needs to fulfill the following specific chemical and physical properties [22]:

- Compatibility with reservoir formation material and fluids
- Capability to develop the required fracture width, allowing proppants to pass-through
- Sufficient viscosity to transport proppants: yield viscosity rapidly and maintain viscosity at shear and temperature
- Low fluid loss into neighboring formations
- Low friction pressure losses in the pipes
- Non-damaging fluid to prevent residues in the formation
- Simple fluid preparation in the field
- Cost-effectiveness

The novel fluid is a linear gel and contains three main ingredients: water, potassium carbonate, and a gelling agent. Depending on the wellbore conditions and the operational parameters, a breaker and a fluid loss agent might be necessary. The following section presents the fluid chemistry in detail and introduces the consecutive lab test conditions.

Water

The BEER fluid can be mixed with fresh water and produced formation water depending on its composition.

Due to the diversity of produced formation water and the necessity to compare lab results, it is recommended to always mix and test the BEER[®] fluid with the actually used water to avoid possible interactions.

Potassium Carbonate

Potassium carbonate (K₂CO₃), colloquially known as potash ash, is a salt in the form of white powder that can be dissolved in water and forms a strongly alkaline solution [11]. Potassium carbonate is strongly biocompatible, well known as a shale stabilizer, and has strong corrosion-inhibiting properties. It is thermally stable and can be used for controlling the pH value of the system. The typical applications are, e.g., production of soaps and glasses, buffering agent to produce wine, or as a drying agent for organic-based compounds like ketones, alcohols, or softening hard water [9].

Gelling Agents

The biodegradable gelling agent BioPolymer I is used in the BEER[®] fluid to increase the fluid viscosity.

BioPolymer I [48] is used as a viscosifier and suspending agent in drilling and work-over operations. It has been used commercially since 1964 and is produced by a bacterium. From a chemical point of view, the polymer is a water-soluble polymer, which can be hydrated in freshwater, brackish water, or saturated salt systems, regardless of whether the water is hot or cold [22]. This polymer can carry a high proppant concentration and keeps them

in suspension due to its pseudo-plasticity. Shear-thinning properties allow an efficient separation and release of the proppants. BioPolymer I has already been used up to a temperature of 150°C [48]. While an increase in temperature changes the viscosity of standard polysaccharides, BioPolymer I stays almost unchanged at temperatures between 10 and 80°C [46]. Typically, it is used at concentrations between 1.5–4.0 kg/m³ [48], has a low friction characteristic, a high stability salt-resistance, and maintains its viscosity when salt (e.g., kali, sodium, calcium, or magnesium salt) is dissolved in the fluid. Furthermore, it is stable in pH if the pH value is between 5 and 10 [46].

Proppant

Proppants are special propping agents used to hold the fracture open after the pumping pressure is released at the surface and the fracturing fluid has leaked off. The fracture conductivity is a measurement of how a propped fracture can convey the produced fluids over the well's producing life. It is affected by the movement of formation fines in the fractures and the properties of the proppants. The ideal proppant is cheap, has an infinite strength to withstand an extreme closure pressure at zero crushing, has a spherical shape to minimize embedment and to spall, has a mono-size distribution for maximum porosity and therefore highest permeability, is chemically inert, has no HSE impact, gives the possibility to add special additives and is oil/gas and water repellent to enhance fluid flow [8]. Normal sand does not meet these requirements and is produced by mining activities. The BEER[®] fluid uses standard glass beads for proppants, which provide rigid and spherical bodies. The glass beads are made of recycled soda-lime glass that is formed into beads. The chemical composition of the glass beads is Si₂O₃ (min. 65%), CaO (min. 8%), Na₂O (min. 14%), Al₂O₃ (0.5–2.0%), Fe₂O₃ (max. 0.15%), MgO (min. 2.5%), and others (max. 2%). The main advantages of glass beads compared to other proppants are the roundness and the perfect sorting by size, which is thought to contribute to the more even packing of the proppants and better permeability (Table 1). Two types of glass beads have been investigated:

- SWARCO glass beads 400–800 µm untreated (mesh size 20/40)

Tab. 1 Glass beads specifications (Swarco, 2017)

Property	Specification
Specific Gravity	2.45 to 2.50 g/cm ³
Bulk Weight	1500 kg/m ³
Mohs Hardness	5.5
Toxicity	None
Color	Clear / Colorless
Shape	Spherical
Roundness	65 to 95%
Size Range	425 – 850 µm, 850 to 1000 µm

- SWARCO glass beads 400–800 μm treated (mesh size 20/40)

The surface-treated glass beads have a silane coating to improve adhesion. Silanes are a group of silicon-based organic-inorganic materials, which hydrolyze and form silanol groups [41]. Silane improves chemical and moisture resistance, mechanical strength, and electrical properties [14]. Research has shown that the silane coating produces an increase in adhesion strength and provides the ability to form more extensive interphases with a polymer [35].

Laboratory test results

Before a fluid can be pumped, it has to pass several laboratory tests according to the fluid’s usage. High-pressure stimulation fluids and proppants have to pass at least the below-listed tests within the given limits successfully to get the final fluid formulation and be a candidate for a field test. The baseline recipe without breaker at reservoir temperature needs to be stable at reservoir temperature for at least pumping time, by consideration of a contingency of about 50% [38].

The BEER® fluid can be characterized on the face of its four components:

- Produced formation water or tap water as the basic fluid
- a linear polymer for rheology, fluid loss control, and carrying capacity improved,
- potassium carbonate – K₂CO₃ – as weighting agent, corrosion inhibitor, clay stabilizer, sour gas buffer, friction reducer, biocide, and
- glass proppants, used for fracture opening

To show the outstanding properties of the components, especially potassium carbonate, the results of the laboratory tests will be presented. The main questions to be answered concern the temperature and pressure the fluid can resist, possible interactions of the ingredients, and effects that can arise on changing the order of mixing the ingredients.

Base water analysis

Chemicals used for stimulation fluids tend to be sensitive to the water composition. Specific water quality is required to, for example, not hinder the polymer from swelling or from getting a limited viscosity. The requested tests contain the analysis of iron, sulfate, chloride, iron, hardness, carbonate, pH, density, color, and precipitations. The standard tap water,

which was used to carry out the following laboratory tests, is within the range.

Aqueous solution density of K₂CO₃ & maximum solubility

To generate a density table of potassium carbonate, density measurements were carried out. The density of the aqueous solution with increasing concentrations of potassium carbonate were tested following API test procedures. 400 ml of water was saturated with an increasing mass concentration of potassium carbonate. An incremental increase in the mass concentration of each 40 g using 400 ml water as base fluid under atmospheric conditions at room temperature was performed. After every concentration increase, the sample was weighed ten times and the density determined.

Furthermore, the maximum solubility of potassium carbonate was determined. K₂CO₃ is soluble up to a maximum mass concentration of 1,100 kg K₂CO₃ per m³ of water. Using a higher concentration additional potassium carbonate would no longer dissolve, but sink to the bottom of the tank.

The density of the BEER® fluid itself is an important parameter as the rig sites in Europe tend to be very small and a high density could lead to a reduction in pumping capacity. The pumping capacity equals the work that has to be applied divided by the time needed. As the work is defined as weight times height and the weight includes the density, an increased density would lead to an increased pumping capacity and therefore to a higher number of pumps needed. A maximum density of 1,500 kg/m³ can be reached with the highest possible/soluble concentration of potassium carbonate. The specific gravity for a conventional aqueous fracturing fluid is 1000 kg/m³ to 1040 kg/m³. Special density borate cross-linked systems can provide densities up to 1380 kg/m³ [27].

Effect of K₂CO₃ on pH-value, temperature behavior, and volume change

To determine the pH-value, a standard pH-value measurement device – a pH-meter – was used. This device was also able to measure the temperature of the fluid. Both the pH-value and the temperature increase with an increase in the K₂CO₃ mass concentration from room temperature of about 26.5°C to 33.5°C at a mass concentration of 1,100 kg/m³. The pH-value changed from 11.5 to 12.7 for a potassium carbonate concentration of 100 kg/m³ to 1,100 kg/m³. The increase of the pH-value from 7 to 11.5 was already achieved by the mixture of tap water and 100 kg/m³ potassium carbonate. The pH-value plays an important role in the subsequent addition of the polymer and its swelling. In conventional fracturing fluids, ordinary water is used as the swelling agent (pH = 7.9 at 24°C). In this case, K₂CO₃ is added to the water before it is exposed to the polymer. This change in pH plays a role as a higher pH-value leads to a reduction in fluid viscosity.

A volume increase of about 16%, from 100 ml up to 116 ml after adding 100 g K₂CO₃ was observed. This volume increase has to be considered in the field design, regarding the available tank capacity to prevent spillover. A temperature increase with an increasing concentration of K₂CO₃ has also been seen. This temperature increase has no significant influence on the polymers later added to the solution. The polymer’s temperature limit is beyond 150°C. The pH-value increase, when using K₂CO₃, makes the fluid a sour gas buffer. The smaller the pH-value the sourer a fluid is. A fluid having a high pH-value can be used to neutralize sour environments. This effect can reduce corrosion in sour gas environments, as K₂CO₃ will remove corrosion accelerating acid constituents. Still, in the presence of K₂CO₃ corrosion of metal components can take place in the case of a high temperature and a too-long contact [59]. The inhibition effect of K₂CO₃ against corrosion is either due to the interaction between components of the inhibitor and the metal surface or due to interaction between the inhibitor and one of the ions present in the aqueous solution, which is the case in the BEER® fluid [55]. Often KCl is used in water-based fluids to prevent the clay from swelling [57], which could be problematic in chloride-sensitive environments. Potassium carbonate acts as a clay swelling inhibitor. The environmentally friendly alternative makes use of the K⁺-ions to prevent the clay from swelling.

Polymer hydration time

A hydration test aims to find the time for the polymer to develop maximum viscosity. A short hydration time of the polymer is desired in fracturing fluids. The hydration time where 80% of the hydrated viscosity is reached at 20°C and the minimum applicable surface temperature of 4°C has to be smaller than the surface time until crosslinker is added. In the laboratory, viscosity was measured after different time steps at room temperature. The measurements showed that the viscosity was fully developed after about one hour.

Viscosity Measurement

Viscosity measurements were carried out using a high-pressure, high-temperature (HPHT) rheometer. The following test conditions have been set up in the HPHT-rheometer (Tab. 2). K₂CO₃ influence on the BEER fluid’s viscosity Figure 1 shows the impact of K₂CO₃ on the BEER fluid’s viscosity. An HPHT-rheometer, using a temperature ramp to heat the fluid from 20 to 90°C, was used. An increase of K₂CO₃ leads to a slight decrease in the dynamic viscosity. There is no clear trend whether a low or a high potassium carbonate content causes a faster drop in viscosity. A bigger viscosity reduction is caused by temperature.

In the next step, the fluid was blended with 6 kg/m³ of Bio Polymer I and varying concentrations of K₂CO₃ (Fig. 2). The mass concentration has an insignificant influence on viscosity at a concentration of 150 kg/m³. Even if the datasheet of

Tab. 2 Test set-up

Pressure	2.76 MPa
Continuous shear rate	170 s-1
Test temperature	29°C, 70°C, 110°C
Geometry set	Rotor R1 / Bob B5
Annulus	0.241 cm
Bob radius	1.599 cm
Bob height	7.620 cm
Sample volume	52 ml
Zero control	Every 50 min

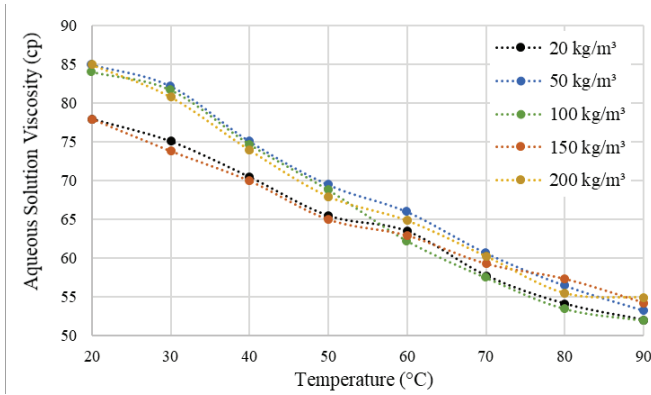


Fig. 1 Influence of mass concentration of K_2CO_3 on viscosity

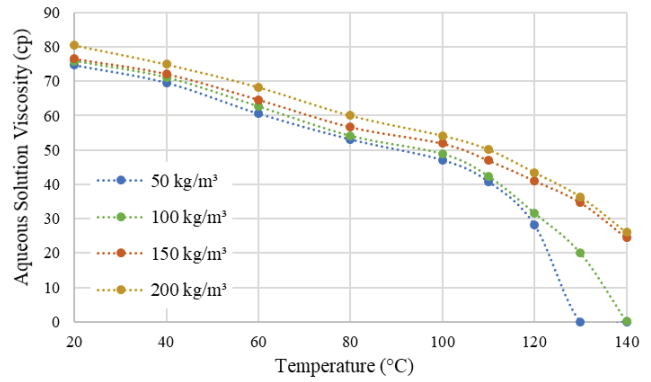


Fig. 2 Influence of K_2CO_3 on the temperature resistance of the polymer

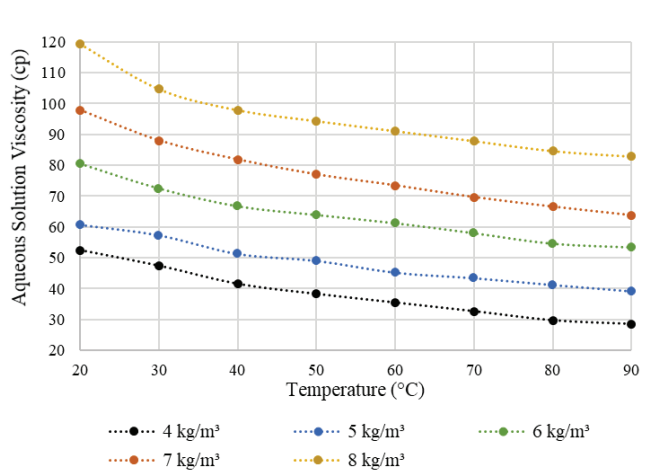


Fig. 3 Influence of the polymer concentration on viscosity

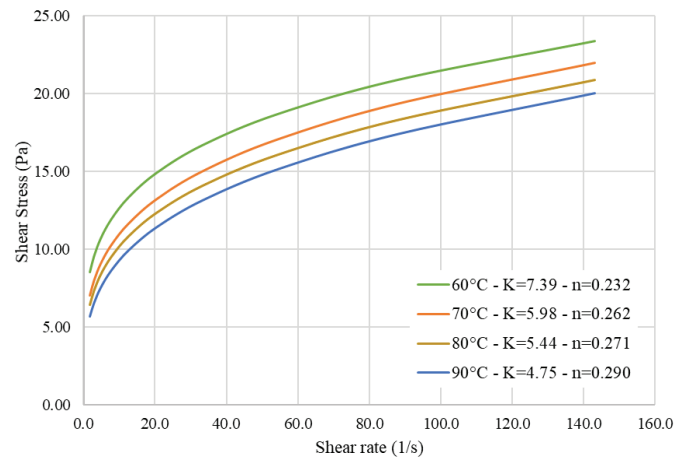


Fig. 4 Influence of K_2CO_3 on the temperature resistance of the polymer

Bio Polymer I predicts a temperature resistance of 150°C, the polymer breaks at a temperature of about 120°C, when no or just small to moderate quantities of K_2CO_3 have been added (Fig. 2). The laboratory experiments conclude that adding K_2CO_3 leads to an increase in temperature resistance. There is an insignificant difference in viscosity using 150 kg/m^3 K_2CO_3 respectively to 200 kg/m^3 K_2CO_3 . Thus, the viscosity will not change by using more K_2CO_3 . So, at this point, apart from an increase in density, it does not make any difference in the positive effects of potassium carbonate, when increasing its mass concentration.

BioPolymer I influence on the fluid viscosity

Figure 3 shows the impact of the mass concentration of BioPolymer I on viscosity. For the lab test using the HPHT-rheometer, the BEER® fluid was blended using 150 kg/m^3 K_2CO_3 with increasing mass concentrations from 4 to 8 kg/m^3 of Bio Polymer I. The polymer provider recommends a concentration between 1.5 and 4 kg/m^3 for drilling fluids. Hydraulic fracturing requires a high viscosity. As a result, the tested concentration started at 4 kg/m^3 of the polymer. To establish the limits of the polymer the mass concentration was increased up to 8 kg/m^3 . The polymer has to be added slowly to the base fluid. During this test procedure, a blending rate for the polymer of 1.5 g/min was used. Using a higher rate would

lead to small flakes that do not dissolve and the highest possible viscosity would not be reached. To not over-engineer the fluid in its viscosity, a mass concentration of 6 kg/m^3 was chosen for further tests. The results indicated the expected decrease in viscosity by temperature.

The polymer solution shows a pseudoplastic shear thinning behavior and can be described by the Power Law Fluid Model [6]. The Power Law Fluid Model is characterized by Eq. 1 and Eq. 2, which give the shear stress τ – shear rate $\dot{\gamma}$ behavior and the dynamic viscosity η - shear rate behavior. K is a temperature-dependent coefficient, and n is the exponent.

$$\tau = K(T)\dot{\gamma}^n \tag{1}$$

$$\eta = K(T)\dot{\gamma}^{n-1} \tag{2}$$

150 kg/m^3 K_2CO_3 and 6 kg/m^3 of Bio Polymer I were mixed and tested regarding rheological parameters. To get the n' and K values, tests with the HPHT-rheometer were made. Figure 4 shows the power-law fluid model for the tested fluid at various temperatures.

The K coefficient and the n exponents are shown and it can be seen that the magnitude of the K coefficient drops, whereas the magnitude of the n exponent rises with temperature. The shear stress indicates the shear thinning behavior with a decreasing trend with temper-

ature.

Proppant settling test – Proppant carrying capacity

The stimulation fluid was mixed using three different polymer concentrations and a constant K_2CO_3 concentration of 150 kg/m^3 , finally adding proppants in a concentration of 500 kg/m^3 . Two different scenarios, 50°C, and 90°C, with three different polymer concentrations each, were tested. 4, 5, and 6 kg/m^3 of Bio Polymer I concentrations were tested. As the viscosity of the polymer stays almost unchanged between temperatures of 10 and 80°C [48], 50°C was chosen as the test temperature. This temperature represents a standard reservoir temperature of a 3,000 m deep well already cooled down by circulation before the hydraulic stimulation. Furthermore, 90°C was selected to see what happens when the temperature exceeds the 80°C limit of the polymer. By putting the samples at 50°C and 90°C in the laboratory oven, the proppant settling was checked after 30 minutes and after 1 hour. Here the focus was on the time when the proppants reach the bottom of the sample glass bottles. Figure 5 shows that the carrying capacity of the polymer drops at 90°C significantly with time for all polymer concentrations, whereas the lower concentrations show a faster setting of the proppants. A temperature of 90°C leads to a visible decrease of vis-

cosity and as a consequence also to a decrease in carrying capacity. At a temperature of 50°C for a concentration of 4 kg/m³ settling can be seen. Polymer concentrations of 5 and 6 kg/m³ indicate a stable behavior.

The best results were achieved with a concentration of 6 kg/m³ and therefore it was decided that this mass concentration is a good choice to use in further laboratory tests (Table 3). During the hydraulic stimulation process, the wellbore and the surrounding rock are cooled by the circulation of the cold fracturing fluid. As the temperature decreases a polymer concentration reduction to 4 kg/m³ is possible.

Breaker Test

After leaving the proppants in the fracture, the fracturing fluid flows back through the well to the surface. As viscosity will rise with decreasing temperature in the wellbore after pumping a high amount of fluid, breakers could be necessary to avoid proppant back-flow. The test procedure is the following. Adding the lowest concentration or no breaker and checking the stability at reservoir temperature. If encapsulated breakers are used, test with 10% crushed and with 100% crushed proppants. After breaking, perform the re-heal test of the viscosity behavior. Cooling down of the fluid takes place and the viscosity shall not rise again. The viscosity must remain as low as 10 cp. The next step considers higher breaker concentrations, but the test is performed at a lower temperature. Finally, all tested fluids are cooled down in a water bath for the shut-in time of the planned operation and the re-heal tendency needs to be checked. To pump the correct breaker concentration, breaker tests are made using different temperatures and breaker concentrations. The stimulation fluid needs to drop in viscosity below 10cP to allow the proppants to settle in the created fracture. To evaluate if a breaker to reduce the BEER® fluids' viscosity is necessary, a

Tab. 3 Analysis of proppant carrying capacity of the BEER fluid using different polymer concentrations

BioPolymer I concentration	after 30 minutes	after 60 minutes	Time when glass proppant reached the bottom
6 kg/m ³	Just a few glass beads started to move to the bottom, separation of the fluid and proppants is not yet visible	Just a few glass beads moved to the bottom, separation of the fluid and proppants visible	>60 minutes
5 kg/m ³	Glass beads started to move to the bottom, separation of the fluid and proppants not clearly visible	Glass beads moving to the bottom, separation of the fluid and proppants very visible	60 minutes
4 kg/m ³	Glass beads already started to move to the bottom, separation of the fluid and proppants very visible	Glass beads already at the bottom, separation of the fluid and proppants very visible	30 min

HPHT-test has been executed. The temperature was increased first in steps to 90°C and then decreased again. As viscosity raises again with a decrease in temperature and it is much higher than 10 cP, it is necessary to use a breaker to achieve fast-breaking of the polymer. Nevertheless, the tests have shown that a shut-in period of one hour would break the polymer without the use of a breaker. In the laboratory tests, ammonium persulfate is applied as a breaker to avoid the backflow of proppants to the surface. Ammonium persulfate is an inorganic oxidizing compound, which enables the delayed release of the proppants in the fracture and the recovery of the fracturing fluid [20]. It is injected with the fracturing fluid to break the polymers in the fracturing fluid after a controlled release time [36].

At this point of research, the aim was to have a viscosity lower than 10 cp after 150 min. Breaker tests were made at different temperatures (50°C to 120°C) using different breaker concentrations. The formulation of the BEER® fluid was 6 kg/m³ Bio Polymer I and 150 kg/m³ K₂CO₃. As mentioned above an ammonium persulfate breaker was used. In general, a higher concentration of breaker is necessary with decreasing temperature. To find the optimum breaker concentrations, the trial-and-error method was used, this means different con-

centrations of the breaker were added to the BEER® fluid and tested in the HPHT-rheometer until a viscosity below 10 cP was achieved. Breaker tests were also made for other temperatures between 50°C and 120°C using different breaker concentrations until the optimum concentration – to get a viscosity lower than 10 cP – was reached. Figure 6 summarizes the breaker test results for a temperature range of 50 to 120°C. As the BEER fluid would be customized for different reservoirs, it would be necessary to repeat the breaker tests for different fluid mixtures.

Fluid Compatibility Test

Fluid compatibility tests are required to see if unwanted interaction of the stimulation fluid's ingredients occurs with other present fluids, like mixing water, wellbore fluids, or other treatment fluids. Severe reservoir damage could occur in the worst case, and the reservoir permeability could permanently be damaged [45]. Laboratory tests were all performed with tap water and produced formation water. The focus was on viscosity reductions by bacterial degradation. The stimulation fluid was mixed with a predefined concentration of water. First, bacteria tests were made, putting two samples – open and with a steel grade J55 coupon – outdoor in the sun and picturing them before and after 24 hours, including the use of a thermometer.

After 24 hours there were still no bacteria in the fluid. The comparison of the viscosity measurements before and after 24 hours indi-

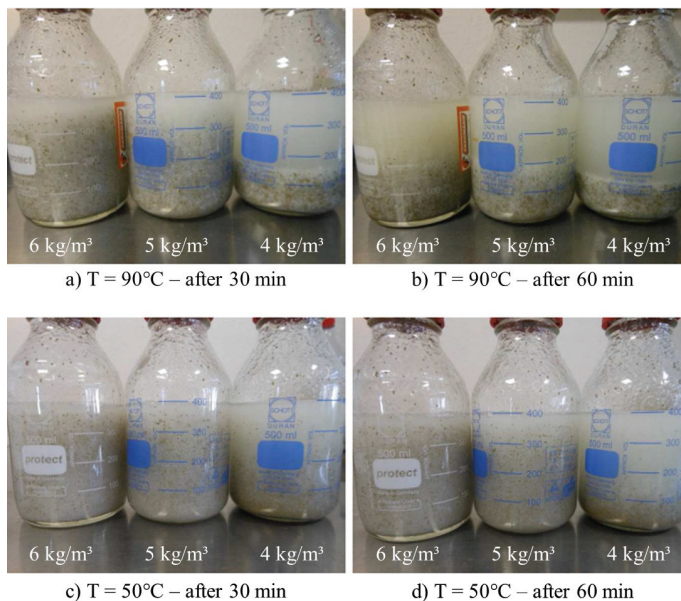


Fig. 5 Influence of the polymer concentration on viscosity

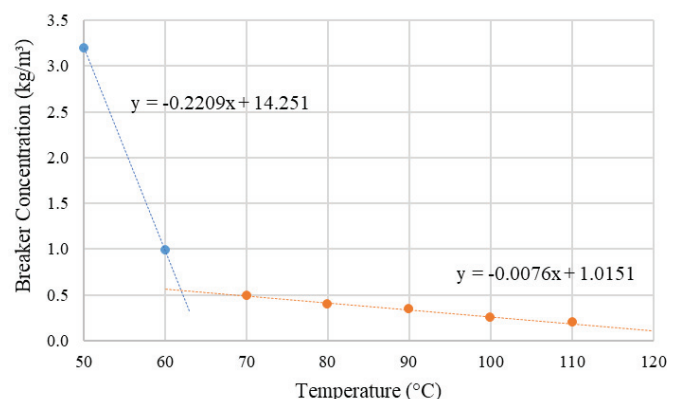


Fig. 6 Optimum breaker concentrations for different temperatures

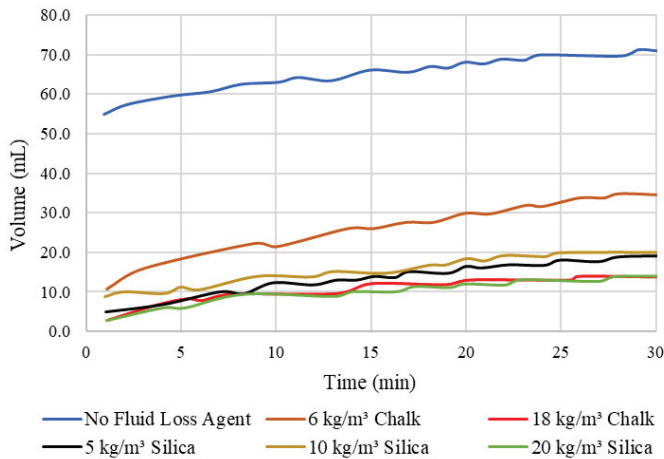


Fig. 7 Fluid loss test results

cates no significant changes. If bacteria would have been there, they would have caused the degradation of the polymer and the viscosity would have been reduced significantly. As a result, no further tests like MPN (Most Probable Number Test) or BART (Biological Activity Reaction Test) are necessary using this water sample.

Fluid Loss Test

To avoid fluid leak-off by a filter cake, the first volume of fracturing fluid, the so-called pad, is pumped without proppants. Still, there are other possibilities for fluid leak-off, like reservoir heterogeneities or fracture height migration – e.g., breaking through a thin layer that separates two permeable formations, can result in a narrow opening where fluid can escape. Both can cause slurry dehydration and a “screen out.” A screenout results in the fluid’s inability to transport the proppants, which leads to a considerable pressure increase. The lateral fracture growth is thus prevented. [18]. The fluid loss test aimed to compare the stimulation fluids’ fluid loss behavior without adding silica or chalk for fluid loss control. Chalk is a carbonate, which is insoluble in water and typically used in drilling muds as a bridging agent. It is harmless to the environment and humans and cheap [43]. Silica nanomaterials possess a significantly larger surface area, compared to micro or macro-sized particles, which causes inherent properties. Silica nanomaterials are used in drilling muds and fracturing fluids as fluid loss agents because of their high stability and functionality [44]. Using the standard API fluid loss test procedure, with a running time of 30 minutes, the volume coming out of the measuring cell was measured every minute. The API fluid loss test – low pressure, low-temperature filtration test – usually measures a mud’s filtration at room temperature and differential pressure of 100 psi [16]. The goal of the fluid loss test was to compare the BEER® fluid with and without adding silica or chalk for fluid loss control. Using the standard fluid loss test procedure, with a running time of 30 minutes, every minute the volume of fluid coming out of the

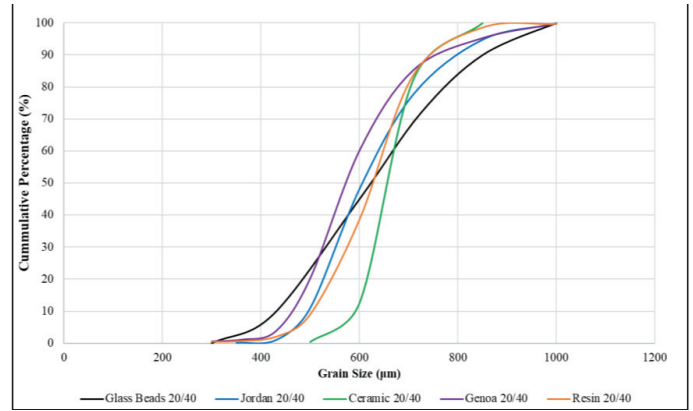


Fig. 8 Grain size distribution for glass beads - 400 to 800 µm (20/40) in comparison to standard proppants [37, 58]

measuring cell was measured (Fig. 7). The results show that a pre-pad using 18 kg/m³ chalk is necessary to prevent fluid losses effectively. The alternative silica nanoparticles (SiO₂) are harmless to the environment, but do not meet the specification of the BEER fluid due to their carcinogenic effect on humans.

Return permeability core test

Core flooding is a well-known test to determine rock permeability and to detect if permeability changes when the core comes into contact with a stimulation fluid. The high-pressure stimulation fluid is circulated at the top of the core, building up a filter cake and leaking through the plug. The permeability is afterwards determined by flowing back with the formation fluid. Using a Berea sandstone core, a return permeability test was carried out using a core flood tester. Here the pre- and also post-treatment permeability can be measured at borehole temperature and pressure. The Berea sandstone core was put into the sample holder of the testing device. Then the core was pressurized, and the temperature was set to 70°C. First, nitrogen was pumped through the core sample to set the general permeability and have a value to compare with. Finally, the stimulation fluid was pumped through the core to see if the permeability changes after the fluid contacts the core sample. A return permeability of 100% was seen.

Proppant sphericity, grain size distribution, and conductivity

All the samples visually showed perfect roundness and sphericity according to API RP 19C specifications. Under a microscope, the perfect roundness could be confirmed for about 95% of the beads, with a few of them having some inclusions of voids as can be seen in the microscopic pictures presented in Figure 9d). As shown in Figure 9a) the typical glass beads show a perfect roundness and a well sorted size distribution. In b) and c) a few imperfect glass beads could be found, like beads adhering to each other, broken beads, or beads with inclusions. Overall, most glass beads showed perfect roundness.

In the current literature some information about the chosen glass beads is provided—the sieve analysis, according to API RP-19C standard [4], was performed by Mukhamedzianova [37] and is shown in Figure 8. Seven sieves were used for the test. 120g of each sample was taken. Each sample was poured onto the top sieve, the stack of sieves was placed in the testing sieve shaker and was agitated for 10 minutes at 278 oscillations per minute. After that, the cumulative mass was weighed and confirmed within 0.5 % of the initial sample mass, as required per the standard. Figure 8 shows a comparison of the glass beads’ grain size distribution to the grain size distribution of conventional proppants of 20/40. It can be seen that the glass beads are slightly less sorted, but the producer confirmed that a better-sorted probe could be provided. In the sieve test, the proposed size was tested and can be confirmed as well within specifications.

McDaniel, R. et al. [33] evaluated the glass beads conductivity for several closure pressures. Table 4 shows that the conductivity de-

Tab. 4 Glass beads (20/40) conductivity test data [33]

Closure pressure (MPa)	Conductivity (mD . m)	Permeability (D)
13.78	951.9	150
27.58	793.4	124
41.36	634.6	100
55.15	190.5	30
68.95	76.2	12

Tab. 5 Crush resistance of SWARCO glass beads in comparison with state-of-the-art proppants

Type	% broken fines
Glass beads 400–800 µm untreated	0.61
Glass beads 400–800 µm H	0.83
Glass beads 400–800 µm H+	0.33
UniMin 20/40	9.56
Accupack 16/30	7.32
CarboLite 20/40	0.15
Borovich 20/40	0.28

creases with pressure from 951,9 md m at 13,78 MPa to 76,2 md m at 68,95 MPa.

Proppant Density Test

The proppant density was measured with a Micromeritics 1340 Helium pycnometer. This pycnometer uses the helium gas displacement method to measure the volume accurately. The glass proppants were placed in the instrument compartment with a known volume. Then helium is admitted, and the pressure before and after the expansion is measured to calculate the sample volume. The sample's mass is divided by the calculated sample volume [34]. Results show that the glass beads stay within the density range of the generally used proppant types.

Proppant Crush Resistance

The crush resistance was measured according to the procedure recommended in API RP 19C. A certain amount of dry sample is placed in a cylinder in which a flat plunger compacts the beads at a pressure of 4,000 psi (~276 bar). The particles collected by the finest sieve after applying pressure represents the percentage of broken beads. According to API a percentage of broken fines up to 10% is acceptable. Table 5 indicates the results of all tested proppants regarding crush resistance. All tested proppants are below 10%, but there is a huge difference in quality.

Result discussion

The basis of this research is to eliminate all non-relevant additives, used in conventional fracturing fluids, to replace the essential ones with environmentally friendly substances that fulfill combined needs. The small number of ingredients allows the accurate evaluation of possible interactions and dependencies like the amount of product used or the mixing order of the ingredients.

K₂CO₃

The maximum amount of potassium carbonate, which can be mixed into the fluid, is 1,100 kg/m³. A higher concentration would not dissolve, but settle down as undissolved solids and can subsequently cause pump-sticking. K₂CO₃ acts as a weighting agent. The higher the concentration of K₂CO₃ the higher the density of the fracturing fluid, which enables a reduction in the surface injection pressure, requiring fewer pumps, a smaller well site, and reduces public attention.

The lab tests have shown that the higher the concentration of K₂CO₃ the higher the pH-value will be. On the one hand, this can limit the viscosity increase of the BEER® fluid itself, but on the other hand, the fluid is a sour gas buffer, meaning that the basic fluid will act as a buffer in an acidic environment. This effect can also help regarding corrosion inhibition in sour gas environments, as K₂CO₃ will remove acid constituents, which could lead to severe corrosion. Nevertheless, a too-long contact of K₂CO₃ at high temperatures with a metal surface could lead to corrosion. This

scenario is rarely the case, as most wells get cooled down in pumping several 100 m³ of base fluid into the well during the stimulation process. K₂CO₃ increases the temperature resistance of the polymer. The lab tests indicated that a minimum amount of 100 to 150 kg/m³ potassium carbonate leads to a temperature limit of 140°C, which is quite high and will seldom be reached in a stimulation operation. A smaller concentration resulted in reduced temperature resistance, whereas a higher concentration will not lead to an even higher temperature resistance. K₂CO₃ improves the clay swelling inhibitor quality of the fluid significantly, as the K⁺-ions prevent the clay from swelling.

One more finding is that K₂CO₃ works as a biocide. Tests regarding storage capability have shown that even bacteria mixed with the fluid cannot resist K₂CO₃ due to its property of increasing the pH-value and thereby creating an environment that kills bacteria.

It is important to mention that the blending water quality is crucial. K₂CO₃ could cause scaling when mixed with the wrong water, which makes laboratory tests for every application imperative.

BioPolymer

The laboratory tests have shown that a minimum amount of 6 kg/m³ of polymer is needed to fulfill the requirements in viscosity. A good proppant carrying capacity has been seen already at a polymer concentration of 6 kg/m³. An increase in temperature reduces the carrying capacity as the viscosity of the fluid decreases, but still starting with a concentration of 6 kg/m³ the proppants stay acceptably in solution with the fluid. The HPHT-rheometer tests have shown a strong dependency of the viscosity on the amount of K₂CO₃ used. K₂CO₃ increases the pH-value of the fluid, which influences swelling of the polymer up to a mass concentration of 150 kg/m³. A higher K₂CO₃ does not show any further influence. It is recommended to first blend the water with K₂CO₃ followed by mixing the polymer, to prevent a too fast hydration of the polymer. Just in case faster hydration is required small quantities of citric acid can be mixed into the solution

An important requirement for a hydraulic stimulation fluid is good fluid loss control, as otherwise a lot of energy gets lost into the surrounding formation and improper fractures will be generated. A fluid loss test has shown that for certain formations, a fluid loss agent might be necessary. Chalk and nano-silica

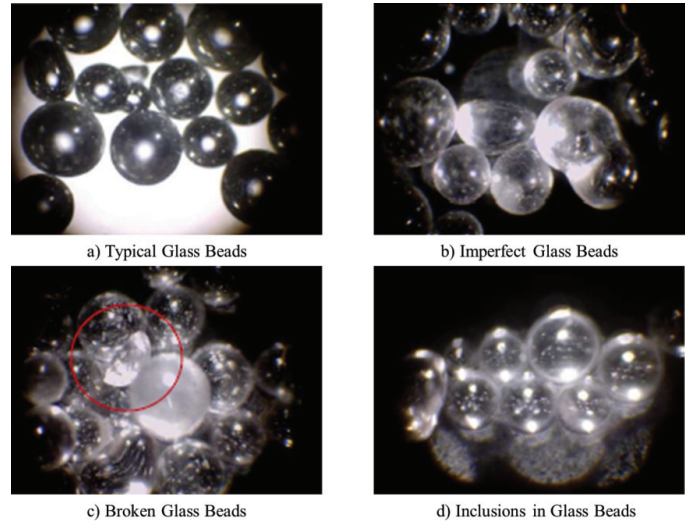


Fig. 9 Microscopic pictures of glass beads

have been tested. The use of 18 kg/m³ of chalk is recommended, as nano-silica is known to cause cancer.

After placing the proppants in the fracture, the fluid needs to break to be back-produced to the surface. Two ways have been seen during the laboratory tests. The first and environmentally friendly way is to wait for about one hour until the BEER® fluid loses its viscosity itself. The faster but less friendly option is to use a breaker. In this work breaker tests using AMPS were executed. The focus here was to reduce the viscosity to a level below 10 cp.

Proppants

Glass beads were tested for use as proppants. The advantages of glass beads in comparison to standard proppants are the grain size distribution, which can easily be designed on the needs of the formation, and the origin. Glass beads are produced from recycled glass and do not cause additional raw material consumption. Glass beads showed a slightly lower crush resistance in comparison to Carbo-Lite 20/40 (0,15% vs. 0,33%), but the glass beads will lead to a better proppant pack as the general size distribution and shape (angularity) is better.

Conclusions

To meet the Paris 2020 goal in reducing CO₂ emissions, natural gas and deep geothermal energy systems are necessary. Especially natural gas is the cleanest fossil fuel in terms of CO₂ emissions. To produce unconventional natural gas and deep geothermal energy, at some point the reservoir needs to be optimized regarding productivity by hydraulic stimulation.

The goal of this research was to create an optimized, economically friendly fluid for hydraulic stimulations. The importance of developing an economically friendly fluid evolved because of political restrictions regarding hydraulic fracturing and environmental concerns of conventional state-of-the-art fluids. These fluids include numerous additives,

where the majority are not environmentally friendly. Thereby it is impossible to identify all interaction processes and finally, not all of the designed fluid systems work efficiently. The clear focus is on keeping the fluid simple but fulfilling all the field requirements of hydraulic stimulations.

K₂CO₃ successfully acts as a weighting agent, sour gas buffer, and corrosion inhibitor. Up to several hundreds of kilograms per m³ of fluid can successfully be added to the fluid. K₂CO₃ increases the stability of the polymer at high temperatures and will act as a clay swelling inhibitor. K₂CO₃ acts furthermore as a biocide. The laboratory tests have shown that a minimum amount of 6 kg/m³ of polymer is needed to fulfill the requirements in viscosity to carry the proppants. It is recommended to mix the water with K₂CO₃ first, followed by the polymer. Depending on the formation parameters, chalk has shown excellent fluid loss capabilities in the BEER[®] fluid. Fluid breaking can take place by self-breaking within about one hour, or by using a breaker. Return permeability tests using the core flooding device have shown that a return permeability of 100% can be achieved.

The standard proppants are provided with a standard grain size distribution, while the glass beads size distribution can be designed for specific requirements, which leads to a much better proppant pack permeability.

In terms of resource and cost-saving, the BEER[®] fluid composition is customized for each application. The maximum concentrations of the additives are defined in lab tests with the focus on the smallest amount of additives necessary to work for the given water composition, temperature, and density requirements.

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