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Buffering Capability of *ß*-chloropropionic Acid–Sodium *ß*-chloropropionate System in Acidic Geothermal Brine

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The Philippines, being part of the Pacific "Ring of Fire," has abundant geothermal energy. Since a substantial number of drilled wells produce acidic geofluid with high enthalpy and high wellhead pressure, it becomes imperative to develop these wells for power generation. A potential pH-buffering method for commercializing high-enthalpy acidic geothermal wells was explored in this study to raise the pH of the geofluids to ≥3.5, the level considered by geothermal reservoir engineers to be noncorrosive to low-carbon steel. The noncondensible gases of acidic geothermal wells are relatively high in H_2S and CO_2 while the brine is high in sulphates. These substances, aside from being potential hazards, account for the acidity of geofluid.

Design-Expert® 6 was used in modeling pH buffering using five design factors (pH geofluid, pH BCPH–NaBCP, volume BCPH–NaBCP, concentration BCPH–NaBCP, and temperature of solution) and one response factor (pH of resulting solution). Two experimental designs were used for the study. Experimental laboratory results showed that BCPH–NaBCP could raise the pH to >3.5 and that the initial pH of the geofluid as well as the pH, concentration, and volume of the BCPH–NaBCP solution significantly influenced the stable pH of the final solution. The best combination of factors was then used in a bench scale setup to investigate the possibility of applying pH buffering at high velocity. Results showed that BCPH–NaBCP was useful for fast buffering and could be used to commercialize acidic wells.

Keywords: Acidic geothermal wells, *ß*-chloropropionic acid–sodium *ß*-chloropropionate (BCPH–NaBCP) buffer, corrosives, geofluid, *and* pH buffering.

INTRODUCTION

Geothermal energy is virtually inexhaustible since it draws heat from the earth and its emission of greenhouse gases is minimal compared to fossil fuels. The removal of hydrogen sulfide from high-temperature steam and the injection of spent geothermal fluids into the ground make potential negative environmental effects negligible.

The Philippines, being a part of the Pacific "Ring of Fire," is abundant in geothermal energy. The country ranks second only to the United States in installed capacity and third behind Mexico in power generation (Quijano 1993). In spite of this huge available geothermal resource, many of the geothermal wells that have been drilled produce acidic geofluid. Although these wells have high energy content, they cannot be commercialized because their geofluid is corrosive to the casing and pipelines of geothermal plants. From interviews with the reservoir engineers of the Philippine National Oil Company (PNOC) and the Philippine Geothermal, Inc. (PGI), it was gathered that of over 600 wells drilled, about 27 wells are of high enthalpy but are corrosive (Brondial and Puertollano 2000).

For many years, the geothermal industry has tried to develop technologies that can commercialize high enthalpy but low pH (acidic) wells in order to avoid the wasteful plugging or abandoning of wells. An abandoned site entails a financial loss that could amount to more than U.S.$3M depending on the well's size and depth.

Probable sources or origin of acidic geothermal fluids

Matsuda et al. (2000) classified acid geothermal fluids into two types, based on the main acid compound that controls their acidity, namely: (a) the *hydrochloric acid* or *(Cl)⁻ type*, which includes Larderello, The Geysers, Kakkonda, and Onikobe; and (b) the *sulfuric acid* or $(SO_4)^{-2}$ *type*, which includes Palinpinon, BacMan, Mt. Apo, and Sumikawa. The sulfuric acid type can be formed either by oxidation of hydrogen sulfide (H_2S), hydrolysis of sulfur dioxide (SO_2), or hydrolysis of native sulfur (S).

The oxidation of H_2S is a typical mechanism that causes acid sulfate hot spring water. This mechanism, however, is rarely the cause of acidity of deep geothermal fluids due to the (a) highly reduced conditions in the geothermal reservoir and (b) impermeable layer preventing shallower fluids from infiltrating the reservoir.

On the one hand, the hydrolysis of SO_2 is represented by

$$4\,SO_2+4\,H_2O\rightarrow 3\,H_2SO_4+H_2S \qquad (1)$$

This reaction, as well as the dissolution of HCl, is related to the source of acidity of many SO_4–Cl type hot spring waters in Japan. Likewise, this is also the formation mechanism of the acidic geothermal waters in Mt. Apo (Salonga 1996) and Sumikawa (Ueda et al. 1991). Acid fluids of this type tend to exist in great depth near the heat source.

On the other hand, the hydrolysis of native sulfur is represented by the following equation:

$$4S+4\,H_2O\rightarrow H_2SO_4+\,3H_2S \qquad (2)$$

Ellis and Giggenbach (1971) stated that sulfur hydrolysis is common in active volcanic areas. This hydrolysis of sulfur occurs at a temperature lower than 224°C. Some Cl-rich acid waters at depth have been explained by this mechanism (Ellis and Mahon 1977, NEDO 1993). The Cl-rich acid water is believed to be formed when high-temperature Cl-type water comes in contact at depth with sulfur-containing rocks.

A study done by the PNOC at the BacMan (Bacon-Manito, Albay) and Palinpinon (Negros Oriental) geothermal fields reported that acidic fluids may be generated by buried sulfur. The presence of native sulfur at depth is perhaps the result of the partial oxidation of H_2S. Specifically abundant in solfatara areas, sulfur may have been carried into the reservoir by downward-moving acid sulphate fluids. Also, the burial of relic solfataras by flush volcanics may have resulted in buried sulfur deposits. Likewise, according to the study, volcanic gas emissions associated with the formation of the caldera may result in sulfur formation within the fractures. The following reactions may occur:

$$SO_2\,(g)+H_2\,O(l)\rightarrow H_2SO_3(l) \qquad (3)$$

$$3H_2SO_3(l)\rightarrow 2\,H_2SO_4(l)+S(s)+H_2O(g) \qquad (4)$$

In effect, according to the PNOC report, this is the proposed general mechanism that describe the process of acid fluid production. Geothermal fluids rich in dissolved H_2S gas is under extreme pressure at great depths. As the fluid rises to the surface, and pressure decreases, steam which carries dissolved H_2S gas and other gases exsolve and separate from the parent fluid. Between the water table and ground surface, the rising hydrogen sulfide gas reacts with downward diffusing atmospheric oxygen to form H_2SO_4. The strong acid is then diluted by downward percolating groundwater as well as by condensing steam. The acidic fluid may trickle down from the surface at considerable depths probably through the same channels bringing gas to the surface. Anhydrite deposition may occur upon mixing with neutral chloride water and this may seal the acid fluids to a confined zone. However, acid fluids may spill over and remix with neutral fluids due to tectonic movements that open new permeability or through wells.

Present mitigation processes for acidic geothermal wells

Although the corrosive properties of these acidic fluids limit their use for power generation, recent successes in new methods of treating them by raising pH levels to a noncorrosive level may bring them back to power lines.

At the Onikobe Geothermal Power Plant in Japan, the countermeasures were as follows: (a) for underground facilities, the internal surface of the acidic wells were coated with PbS; (b) the production casing from the well mouth to a depth of 200 m was made of CR–25, 40LB stainless steel containing 25% chromium; (c) ceramic plasma was sprayed on the coupling section to provide insulation between different metals to prevent galvanic corrosion; and (d) a third stage casing was inserted for depths of 350–600 m such that if in case of damage to the production casing, the double- or triple-casing would still be present to extend the well life (Abe 1993).

At the Coldwater Creek Steam Field and the Aidlin Geothermal Project of The Geysers in California, which produced acidic chloride condensate in the well casings and steam lines of certain fields, a complete treatment was installed utilizing caustic/water injection and steam washing as part of an overall corrosion mitigation system (Meeker et al. 1990).

At the Miravalles Geothermal Field in Costa Rica, 50% by weight sodium hydroxide was injected at 950-m depth to neutralize the acid fluid. The developed neutralization system raised the surface pH values from 2.5 to 6.5 and maintained the pH range stable over the test period of 28 days (Sanchez et al. 2000).

In the Philippines, PNOC had performed a mitigation process for the Mahanagdong Acidic Well at Tongonan, Leyte, in 1998. The mitigation process consisted of injecting NaOH solution into the wellbore to neutralize the acidity of the well (Villa et al. 2000). At the Tiwi Geothermal Field in Albay, NaOH injection was performed at its Bariis 8 well. The well was connected to the Tiwi steam gathering system for three months and then stopped because the well flow decreased slightly. Efforts to pull the injection assembly out of the well have been unsuccessful to date and the commercial mitigation operation had to be stopped for safety considerations pending further attempts to pull the injection assembly out of the well (Gardner et al. 2001).

pH buffering and buffer solution

This study focuses on the application of pH buffering technique on low pH geofluid to pH values equal to or above 3.5 at high velocity. The idea of buffering is to adjust the pH of the solution to a noncorrosive level such as pH >3.5. At this pH level, the construction material of the geothermal well casing and the steam gathering system will not corrode due to acidity.

A buffer composed of a weak acid and its strong salt with a strong base, or a weak base and its strong salt with a weak acid, has the ability to maintain the pH of a solution between narrow limits (Kenner and Busch 1979). Therefore, a solution that is buffered will have no appreciable change in pH upon dilution or addition of slight to moderate amounts of a strong acid or base. This ability of a buffer to prevent a significant change in pH is directly related to the total concentration of the buffering substances and to their concentration ratio in terms of moles acid (or base)/moles salt.

The new buffer used in the present study is β-chloropropionic acid–sodium β-chloropropionate (BCPH–NaBCP). It is a mixture of *β-chloropropionic acid* (BCPH), a monocarboxylic acid, and its sodium salt (Na–β-chloropropionate). BCPH has a $pKa=4.11$ (Harris 1998). The chlorine atom increases the molecular weight of BCPH, thus increasing its boiling point. The chlorine atom is also responsible for the acid's high ionization. BCPH has a high boiling temperature of 205°C (Dean 1999), which ensures its stability at high temperature and pressure. Thermal stability is very important because this buffer is intended for use in high-temperature geothermal reservoirs. Likewise, BCPH has a high decomposition point in water (>200) via hydrolysis (Merck Index 1989). The stable buffering pH-range of BCPH–NaBCP is 4.11±1 (Harris 1998). This means that the buffer is effective from pH 3.11 up to 5.11.

Limitations of the study

This study evaluated the buffering effectiveness of BCPH–NaBCP solutions in an acidic geofluid. The bench-scale laboratory setup for the simulation of a geothermal well was heuristically designed and fabricated within a given budget. A boiler provided the steam to heat the geofluid to a maximum pressure of 125 psig while the maximum temperature of the geofluid remained below 100°C compared to the high-temperature and -pressure conditions in a geothermal well.

EXPERIMENTAL PROCEDURES

Laboratory tests

Five factors were included in each of the two experimental designs (see Table 1). The factors were held at specified levels, classified as *controllable,* while the temperature of the resulting solution was allowed to vary in Design I but not in Design II. The response variable employed was the resulting pH of the solution. After entering all the factors to be considered and the responses to be obtained by experimentation, a tabular form with a total of 50 experiments at different combinations of factors were generated for each design. The response (pH of solution) was experimentally determined for each combination of factors.

This study, however, did not make use of actual acidic geofluid which is impossible to obtain. This is so because in the Philippines, all the acidic wells are plugged or cemented and abandoned. Thus, to maintain the necessary chemical composition, instead of preparing a synthetic geothermal fluid the experiment used geofluid obtained from a geothermal well located at Mak-Ban Geothermal Field, the most accessible geothermal field outside of Manila from De La Salle University. The geofluid had an average pH of 6.2. A few milliliters of HCl were added to a large volume of this geofluid to simulate the acidic geofluid to be used in the laboratory experiments and for the DLSU bench-scale setup simulations.

For the laboratory experiments done at room temperature, 750 ml of geofluid was placed in a 1L Erlenmeyer flask into which were added the pH, concentration, and volume of the BCPH–NaBCP buffer required by the experimental design. The pH of the resulting solution was obtained using an Orion 230A pH meter.

However, for the experiments conducted at temperatures greater than room temperature, the 750-ml geofluid was placed in a three-necked flask in a reflux setup into which was added the required combination of pH, concentration, and volume of the BCPH–NaBCP buffer. In the reflux setup, the first neck was inserted with a thermometer, the second neck with an ordinary condenser, and the third neck was where the sampling was done. Since the pH meter has a temperature probe, the actual reading in the pH meter was recorded.

Table 1. Factors Used in the Two Experimental Designs

Factors	I	II
pH geofluid	2.0, 2.5, *and* 3.0	1.5, 2.25, *and* 3.0
pH BCPH–NaBCP	3.5, 4.0, *and* 4.5	4.0, 4.5, *and* 5.0
vol BCPH–NaBCP	5–45 ml	5–45 ml
Concentration BCPH–NaBCP	0.01, 0.025, 0.075, 0.1, 0.25, *and* 0.5 M	0.20, 0.35, *and* 0.50 M
Temperature of the solution	25–100°C	30°C, 45°C, *and* 60°C

The tests for the thermal stability of the buffering strengths of BCPH–NaBCP solutions were done by heating the solutions for one hour in sealed glass bulblets at 100°C, 120°C, 140°C, 160°C, 80°C, and 200°C. The hot solutions were allowed to cool and their corresponding pH measured. This test was done to find out if the BCPH–NaBCP buffer will maintain almost the same pH when subjected to different temperatures.

In this study, the buffer capacity of the BCPH-NaBCP solution to an added H_2SO_4 was determined by using BCPH–NaBCP solutions of pH 4 and 4.5 and of different concentrations (0.05M, 0.075M, 0.10M, and 0.25M). The actual amount of H_2SO_4 needed to lower by one unit the pH of the solution was obtained. Likewise, the beta value or factor of the BCPH–NaBCP solution was determined by using BCPH–NaBCP Solution of pH 4 and 4.5 of different concentrations (0.05M, 0.075M, 0.10M, and 0.25M), and the amount of NaOH needed to increase the pH by one unit was obtained.

Experiments using the bench-scale setup

The buffering effectiveness of the BCPH–NaBCP solutions was evaluated using the bench-scale setup shown in Figure 1.

Geothermal brine (*geofluid*) from the brine tank was pumped into the mixing tank where it was heated with the steam coming from the boiler. Then, the heated geothermal brine was passed through the bottom of the 7.62-m long, 3.81-cm diameter column and mixed with the BCPH–NaBCP solution pumped from the solution tank. After two minutes, samples of the mixed geothermal brine and BCPH-NaBCP solutions are collected at the five ports starting at the bottom of the column and at 5-ft intervals up to the top of the column. The same procedure was done using this time the 25-ft long, 2.0" diameter column.

RESULTS AND DISCUSSION

Laboratory results

In this research, the statistical analysis software package Design-Expert® 6 was used to process the data collected from the experiments conducted based on an experimental design generated by the same software. Using the Response Surface Method with emphasis on Central Composite Design, each experimental design generated a total of 50 runs for the different factor combinations. The data were analyzed through graphs and model adequacy testing and, then, confidence interval estimation procedures were carried out. Residual analysis was also done.

Table 2. Experimental Design 1 Analysis of Variance

Source		F Value	Prob > F
Model		52.67	< 0.0001
A – pH Geo		246.82	< 0.0001
B – pH YODIN		63.63	< 0.0001
C – vol YODIN		18.45	0.0001
D – conc YODIN		58.43	< 0.0001
A2		29.95	< 0.0001
AC		6.35	0.0158
BC		6.80	0.0127
BD		39.12	< 0.0001
CD		4.50	0.0402
Std. Dev.	0.18	R-Squared	0.9222
Mean	2.96	Adj. R-Squared	0.9047
C.V.	6.03	Pred. R-Squared	0.8717
PRESS	2.10	Adeq. Precision	28.213

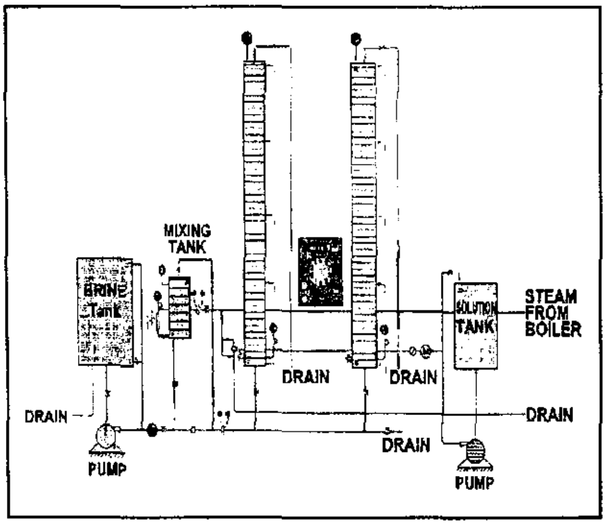

Figure 1. Bench-scale Setup for the Simulation of a Geothermal Well

Results showed that based on a completely randomized blocking experiments, the equation for the pH of solution as obtained from the software used for the experimental design I is:

$$\begin{aligned}\text{pH Solution} = &-6.94715+7.08047*\text{pH Geo}\\ &-0.12811*\text{pH BCPH-NaBCP}\\ &-0.00993464*\text{vol BCPH-NaBCP}\\ &-5.82739*\text{conc BCPH-NaBCP}-1.18407*\text{pH Geo}^2\\ &-7.950000\text{E}-003*\text{pH Geo}*\text{volBCPH-NaBCP}\\ &+0.008225*\text{pH BCPH-NaBCP}*\text{vol BCPH-NaBCP}\\ &+1.61020*\text{pH BCPH-NaBCP}*\text{conc BCPH-NaBCP}\\ &+0.013648*\text{vol BCPH-NaBCP}*\text{conc BCPH-NaBCP}\end{aligned} \tag{5}$$

Table 3. Experimental Design II Analysis of Variance

Source		F Value	Prob > F
Model		91.52464758	< 0.0001
A		313.9628066	< 0.0001
B		31.96289312	< 0.0001
D		79.16955519	< 0.0001
A2		88.43014647	< 0.0001
AD		31.04989775	< 0.0001
BD		4.572586313	0.0382
Std. Dev.	0.271403	R-Squared	0.927383
Mean	3.3371	Adj R-Squared	0.91725
C.V.	8.132905	Pred R-Squared	0.894557
PRESS	4.599132	Adeq Precision	29.58213

$$\begin{aligned}\text{pH Solution} = &-9.38831+7.73522*\text{pH Geo}\\ &+0.53962*\text{pH BCPH-NaBCP}\\ &-0.026396*\text{vol BCPH-NaBCP}-1.37556*\text{pH Geo}^2\\ &-0.017283*\text{pH Geo}*\text{vol BCPH-NaBCP}\\ &+0.020519*\text{pH BCPH-NaBCP}*\text{vol BCPH-NaBCP}\end{aligned} \tag{6}$$

The normal probability plots as shown in figures 2 and 3 have scatter points which indicate that the residuals follow a normal distribution. Both figures reveal no apparent problem since the straight line concentrates on the central portion of the data.

Predicting the best pH for the solution using different combinations of parameters through the statistical analysis software package was done to find out the best combination of factors to use in the bench-scale setup. Due to the high cost of chemicals needed, this procedure was considered necessary so that a lesser number of trials could be performed and still come up with the best results. Thus, for the bench-scale setup, the following were used: pH geofluid = 3.0, pH buffer = 4.0, and concentration buffer = 0.25.

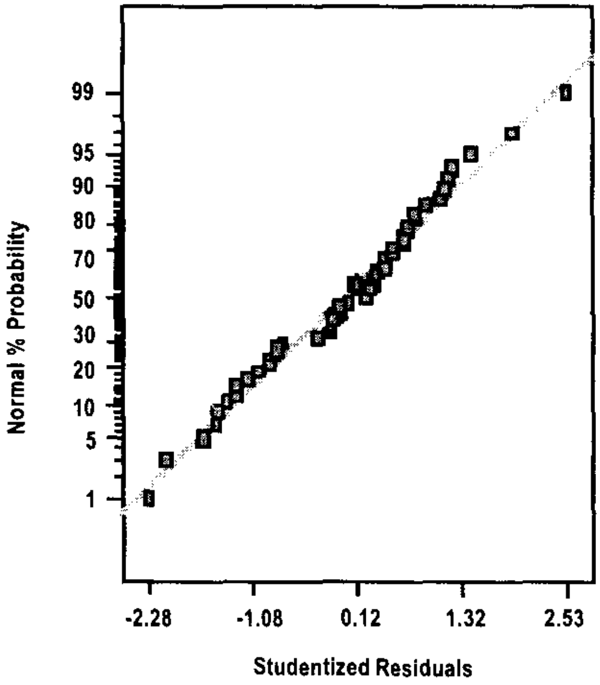

Figure 2. Normal Plot of Residuals of pH Solution for Experimental Design I

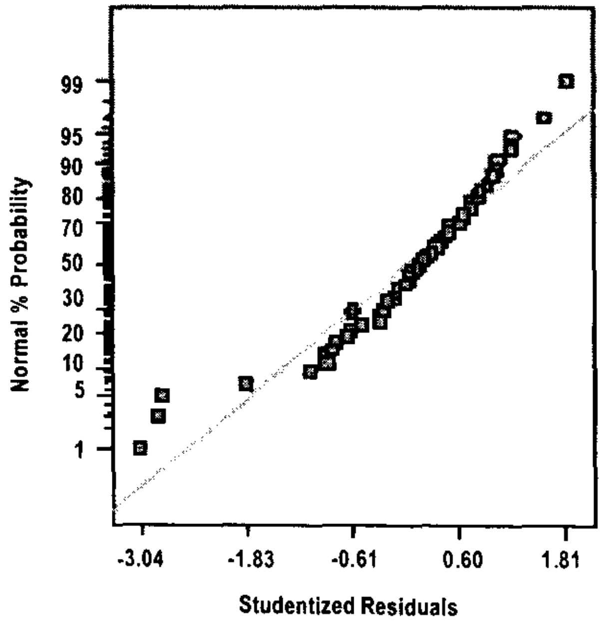

Figure 3. Normal Plot of Residuals of pH Solution for Experimental Design II

Results of the bench-scale setup experiments

Each of the pH of solution at the different ports—from the bottom to the top of the column which are 5 feet from each other—has a value greater than 3.5. Hence, the results showed that the buffer system can effectively buffer the geofluid.

Likewise, the bench-scale setup experiments using the 2-inch diameter column showed the results of the pH of solutions at the different ports to be greater than 3.5. These results were obtained using the following values: pH geofluid = 3.088, pH buffer = 4.0, and concentration buffer = 0.25M. Since the results for both 1.5- and 2-inch columns gave the desired pH of solution, then the pH buffering of the acidic geothermal fluid is independent of the volume of geofluid going out of the 1.5-inch diameter column and the 2-inch diameter column.

Capacity and beta value of the BCPH–NaBCP buffer system

Results (see Figure 4) show that the capacity of BCPH–NaBCP increases with concentration for each pH. If a greater capacity is required of the buffer system, a more concentrated buffer has to be used. Likewise, the results in Figure 5 reveal that the beta value or beta factor increases with the concentration of the buffer system for each pH. Hence, a more concentrated buffer system is to be used if a stronger base is to be added to the buffer system to make the buffer more effective.

The capacity of a buffer towards an added acid or base depends both on the (a) concentration ratio of acid and salt and (b) their total concentration in the buffer. The buffer's maximum capacity to either acid or base for any given total concentration occurs when the concentrations of the acid and the salt are the same.

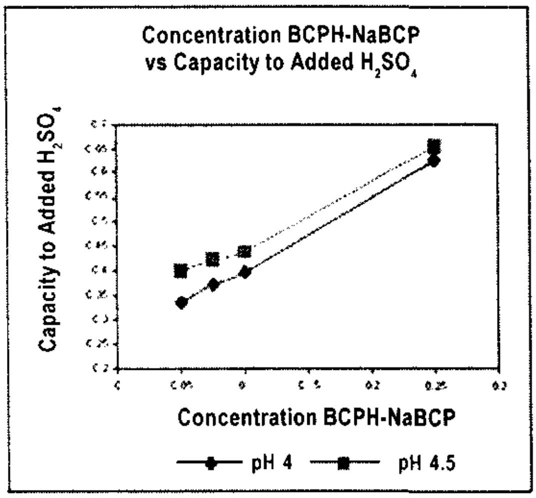

Figure 4. Capacity of BCPH–NaBCP

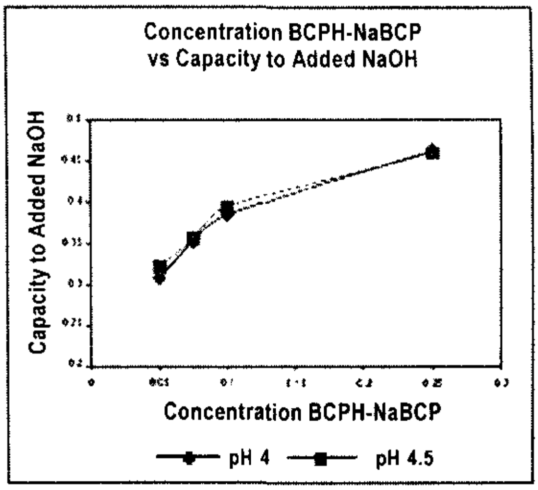

Figure 5. Beta Value of BCPH–NaBCP

Table 4. Bench-Scale Setup Data for the 1.5-inch Diameter Column [pH Geofluid = 3.0, pH BCPH-NaBCP = 4.0, and Conc. BCPH-NaBCP = 0.25M]

Height *(ft)*	1	2	3	4	5	6
20	4.3240	4.3400	3.630	4.020	3.480	3.559
15	3.8420	3.5726	3.650	3.858	3.595	3.538
10	4.1860	3.8860	3.719	3.648	3.470	3.530
5	3.7516	3.6750	3.650	3.699	3.691	3.527
0	4.2330	4.0140	3.915	3.699	3.440	3.476

Table 5. Bench-Scale Setup Data for the 2.0-inch Diameter Column [pH Geofluid = 3.088, pH BCPH-NaBCP = 4.0, and Conc. BCPH-NaBCP = 0.25M]

Height *(ft)*	1	2	3	4	5	6
20	3.883	3.830	3.830	3.980	3.840	3.890
15	3.870	3.890	3.783	4.004	3.840	3.740
10	3.900	3.860	3.800	3.920	3.720	3.850
5	3.962	3.880	3.890	3.896	3.950	3.910
0	3.940	3.913	4.055	4.024	3.960	3.950

Thus, the solutions that contain more acid than salt will have a higher capacity to added base than to added acid; whereas solutions containing more salt than acid will have a higher capacity to added acid than to added base.

CONCLUSIONS

The results of the laboratory tests showed that the pH-buffering technique that use BCPH–NaBCP solutions raised the geofluid pH to as high as 4.086 for the first experimental design and 4.369 for the second design. A pH level that is ≥3.5 has been considered noncorrosive to low-carbon steel (Abe 1993).

The laboratory tests showed that the initial pH of geofluid as well as the pH, concentration, and volume of BCPH–NaBCP solutions significantly influenced the stable pH of the final solution.

Experiments on the capacity of BCPH–NaBCP to changes in pH from 4.0 to 3.0 and from 4.5 to 3.5 by the addition of a strong acid showed that the higher the concentration of BCPH–NaBCP, the higher would be the capacity of the buffer solution. Likewise, experiments on the beta factor or beta value of BCPH–NaBCP for changes in pH from 4.0 to 5.0 and from 4.5 to 5.5 showed that the beta factor increased with the concentration of the buffer system for the same pH.

Bench-scale setup

To find out the best combination of factors to be used in the bench-scale setup, the best pH of solution using different combinations of parameters was predicted using Design-Expert® 6. Using a set of fixed values, namely: pH Geo = 3.0, pH buffer = 4.0, and concentration buffer = 0.25, the experiments were done on the bench-scale setup.

The results of the 1.5-inch diameter column (refer to Table 3) and the 2-inch diameter column (refer to Table 4) showed that BCPH-NaBCP buffer system raised the geofluid pH to a value greater than 3.5 in all the five ports in each column with an interval of 5 feet. Based on the pH of the resulting solution, the diameter of the column is immaterial in the buffering of the acidic geothermal brine because the range of values for the 1.5-inch column and the 2-inch column are almost the same which is equal or greater than a pH of 3.5.

RECOMMENDATIONS

Since this research has not been tested in an actual acidic geothermal well, it is recommended that the most advisable strategy is to introduce the BCPH–NaBCP buffer solution at high concentration in a reservoir and then to shut-in the well for a day or two to allow the well to heat up and to build up pressure. Afterwards, the BCPH–NaBCP solution can be introduced using an acid string set at the production well. The acid string will be inserted through the hole at the top of the geothermal well assembly to reach the level where acidic waters enter the well. Then, let the geofluid flow out of the well while injecting a regulated amount of relatively high concentration of BCPH–NaBCP.

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