Study on *Hibiscus Rosa-Sinensis* Extract as a Bio-Surfactant in Enhanced Oil Recovery

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***Abstract*: Enhanced oil recovery using chemicals is quite successful in producing a good amount of hydrocarbon accumulated within the reservoir. The chemicals which can be injected include surfactant mixed in solutions or long chain polymer flooding. The surfactants function by altering the rock wettability. It also affects the water oil interfacial tension. But the application of chemical surfactants has detrimental impact on the environment as these are mostly non-biodegradable. The chemicals when disposed in the aquatic bodies cause serious threat to the aquatic plants and animals. The paper revolves around the study carried out to analyze the impact of bio-surfactant derived from *Hibiscus rosa-sinensis* extract on changing the surface tension in between the water-oil system and environment. The results demonstrated that the surfactant reduced the interfacial tension significantly from 37.1 mN/m to 13.2 mN/m at a critical micelle concentration of 2.2 wt%. As a result, upon comparison to industrial surfactants the bio-surfactant had excellent activity on the interfacial surface. The exposure of the surfactant solution didn't affect the soil quality and pH.**

Keywords— Hibiscus leaves, bio-surfactant, Enhanced Oil Recovery, wettability alteration, IFT

INTRODUCTION

Oil production from reservoir has become successful due to the different techniques of oil recovery. These techniques are primary, secondary and tertiary techniques of oil recovery. The primary techniques depend on the natural drive mechanisms which are prevalent within the reservoir. The secondary mechanism of oil recovery depends on water flooding, and gas flooding to impact the natural drives present within the reservoir. As the oil is produced there is a decline in the reservoir pressure causing entrapment of oil within the small pore spaces. There is a reduction of oil mobility within the reservoir. Hence, after implementation of these two techniques, about 60-70 percent of the initial oil remains within the pore spaces. Rapid industrialization and growing human needs require extraction of more and more barrels of oil from the reservoir. So, to recovery a significant amount of the remaining oil in place, the industry professionals implement usage of enhanced oil recovery methods. The enhanced oil recovery methods that are usually common include thermal, chemical, microbial, and gas injection. These methods increase the oil production by affecting the oil mobility, rock-oil wettability alteration, oil density alteration and changes in capillary forces. The thermal injection employs application of heat to alter the viscosity of oil. Due to application of heat, a part of the hydrocarbon is vaporized. It affects the mobility. In reservoirs, where heavy crude oil is present, application of thermal injection is very successful. The surface tension is reduced. This technique recovers about 40 percent of the remaining oil in place. The industry sees usage of cyclic steam injection in which the steam gets evaporated in the steam zone. Then the vapor is expanded in the hot water zone. This system is quite relevant technique in US.

Carbonate reservoirs are quite prevalent in middle east region. In this region application of chemical enhanced oil recovery is a common trend. The chemical enhanced oil recovery functions by altering the rock-oil wettability system. It also impacts the interfacial tension between the oil and water system. The interfacial tension is reduced thus increasing the overall oil production. There are number of chemicals which are used in the chemical enhanced oil recovery. These chemicals can be long chain polymers or surfactants. Polymer flooding involves forming dilute solutions of long chain polymers and then injecting it within the reservoir. The polymer flooding increases the water viscosity present within the reservoir and thus improving the mobility. But the limitation of the process is its high cost and environment impact.[1],[5],[10]

Surfactant flooding in chemical enhanced oil recovery uses chemicals which can affect the interfacial tension between oil water system. The surfactant also alters the rock-oil wettability. These two factors increase the overall oil production. The surfactants which are injected can be artificially synthesized or naturally derived. Application of artificial surfactants are costly due to the high price of the chemicals but the system to produce the natural surfactant is quite economical. While the initial efforts of using the surfactants were unsuccessful in 1900s, the failure originated the idea of using in-situ synthesized chemical based surfactants. Later in 1970s the artificial surfactants produced from petroleum sulfonates came into usage. In 1980s the first alkaline surfactant polymer was synthesized which could show the benefits of both alkaline and surfactant flooding. From then, a number of surfactants were synthesized based on the reservoir needs. For instance, carbonate reservoirs required the usage of cationic surfactants. In order to increase the solubility of oil and water in micro-emulsions, the surfactants with branched hydrocarbon chains were used that also helped in decreasing the hydrophilic propylene oxide group. The variety of surfactants that are used in the field include petroleum sulfonates, ethyl sulfate, ethoxyalkylphenol sulfonate, alkane sulfonate, fatty-acid alkanolamide double polyethenoxy ether, alkyl aromatic sulfonate, ether sulfate, ammonium lignosulfonate (Petrolig ERA-27), nonioninc PLURONIC L64 (BASF), nonionic POA, CAC, Petrostep surfactants, Butyl Cellosolve (Dow), SO3NH4, SPS (CS-2000), Lignin II, TRS 10-410 Sulfonate, Witco's TRS 10-410, TRS-40, TRS-18, Peronate TRS 10-80, Texaco LN60-COS, Conoco Alfonic 610-50, S13D, S2.[2],[3]

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The synthesis of these chemicals is quite complex thus increasing the overall cost of the operation. Also, the chemical surfactants pose a negative impact on the environment. Upon dumping in soil, the pH is hindered. The nutrient quality of soil is disturbed. The surfactant solution also causes pollution of water table. Global concern about the reduction in levels of drinking water, global warming, and environment degradation led to framing of stringent laws. Government in different countries have restricted the implementation of chemicals in the petroleum operation. So, majority of the oil and gas companies are trying to shift on eco-friendly methods of petroleum operations. Operators of oil and gas companies are finding replacements of surfactants that are naturally based. Bio-surfactants synthesized from plants are under experimentation. Extracts from plants like mulberry, spistan, prosopsis, ziziphus, olive, eucalyptus, and henna have been used for making the surfactants. While efficiency of altering the rock oil wettability is maximum in mulberry extract, eucalyptus plant-based extract reduced the IFT from 35.2 to 10.2 mN/m. The naturally derived surfactants are rich in dry matter, crude proteins and organic matter. Some other compositions of the bio-surfactants are crude fiber, crude proteins, ether extract, acid and organic detergent fibers, and acid detergent cellulose. The biological and chemical content of the leaves and fruits that are used for bio-surfactant synthesis affect the chemical activity. The Fourier Transformation Infra-red Spectrometry analysis of these plant extracts have revealed to contain functional groups like phosphate, carbonate, silicate, sulphate functional groups. These functional groups help affect the micelles forming capacity. The surfactants are rich in carbohydrates, lignocellulose, fats and ash.[4][6][7]

In this paper we study the impact of utilizing extract synthesized from *Hibiscus rosa-sinensis* plant on enhanced oil recovery by means of measuring the interfacial tension across the water-oil system and critical micelle concentration. We would also find out the impact of the surfactant solution on the pH of soil upon disposal. Further, we would also try to study its impact on the plant growth rate.

MATERIAL USED:

The materials required for the experimentation include deionized water, hibiscus leaves, kerosene oil. In order to study the interfacial tension between the oil and water system, it is mostly preferred to use clean oil. So, we decided to use kerosene oil for ease of our experiment. The density of kerosene oil at the time of experimentation is 0.7769 grams per cubic meter. Deionized water was used for making the surfactant solution because the surfactants would precipitate out in presence of brine solution. The plant under our experimentation belong to the same clade as that of the ziziphus. Since, extract of ziziphus has proven to be a good replacement of the chemical surfactants, so investigation can be carried out on the activity of rock-oil wettability alteration using another plant belonging to same clade.

The apparatus required for the experimentation are Soxhlet apparatus, conductivity meter, weighing balance, stirrer, beaker, test tubes, and pendant drop apparatus. I would also need a sapling planted in a tree pot to identify impact of the surfactant solution on soil and plant health.

METHODOLGY:

The experimentational investigation starts with characterization of *Hibiscus rosa-sinensis* leaves followed by surfactant production to test the IFT and CMC. Finally, the pH of soil will also be measured to check the environmental impact of the prepared surfactant.

1. Characterization of Plant: The characterization of *Hibiscus rosa-sinensis* plant has been done using Fourier Transformation Infrared (FTIR) Spectrometry. In order to carry out FTIR analysis, the plant leaves were dried at high temperature. The dried leaves are then crushed manually into powder. A little amount of KBr is taken in a mortar to which a very less amount of crushed leaf powder was added. The mix was then pressurized to form a pellet. The pellet was put in a slide and finally analyzed under infrared light.

2. Surfactant Production: For obtaining the extract from the leaves of hibiscus plant, we use the Soxhlet apparatus. The solvent used is deionized water for forming the solution with extract. The fresh, clean leaves of hibiscus plants are put in the apparatus and we start the process of extraction and leave it for 24 hours. After 24 hours, the solution of extract and deionized water is taken and heated at a temperature greater than the boiling point of water to remove the solvent. After the solvent is vaporized completely, we get the pure extract. The pure extract precipitate is then left for cooling for 3 hours. The solid extract has soapy touch in feeling, confirming its ability to act as a surfactant.

3. CMC measurement: Conductivity experiment is carried out to measure the critical micelle concentration of surfactant. The apparatus and the electrodes are washed using distilled water to remove any impurities. The apparatus is calibrated using a standard solution and then the conductivity experiment is carried out. The critical micelle concentration is obtained by plotting the graph of surfactant concentration vs the reading of conductivity meter.

4. IFT measurement: Pendant drop analysis is used for measuring the interfacial tension between the oil and surfactant solution. This method requires the density of surfactant solution that is prepared using different concentration of surfactant and oil. During the experiment, the pump of the apparatus to ensure a low flow rate of the injected surfactant solution. The oil drop which is released from the apparatus is due to difference in the densities of the solution. The change in shape of drops is utilized for measuring the IFT across the two layers.

The interfacial tension formula

$$\sigma = \frac{\Delta \rho g R^2}{\beta}$$

where,
σ is the interfacial tension,
$\Delta\rho$ is the density difference between two phases,
g is the acceleration due to gravity.

β and R are determined as follows:

$$\beta = 0.12836 - 0.7577S + 1.7713S^2 - 0.54326S^3$$

$$\left(\frac{d}{2R}\right) = 0.9987 - 0.1971\beta - 0.0734\beta^2 + o.34798\beta^2$$

where,
S = d/D, termed as Drop Shape Factor,

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D is the maximum diameter of the drop
D is the horizontal perimeter of drop

5. Soil pH analysis: A plant is taken and the initial pH of soil present in the pot is measured using the digital pH meter. This reading is labelled as initial pH. The unused surfactant solution is then discarded in the plant pot. Digital pH meter is then put in the soil in which we disposed the surfactant solution to find out the impact of surfactant solution on soil pH. The reading on digital pH meter is noted. The measurement of soil pH is repeated for different concentration of the surfactant solution.

RESULT AND ANALYSIS:

The FTIR analysis of hibiscus leaves are presented in Fig 1. The analysis shows that the leaves have presence of phosphate, carbonate, silicate functional groups. Presence of these functional groups indicate that the plant can be used for synthesis of surfactants.

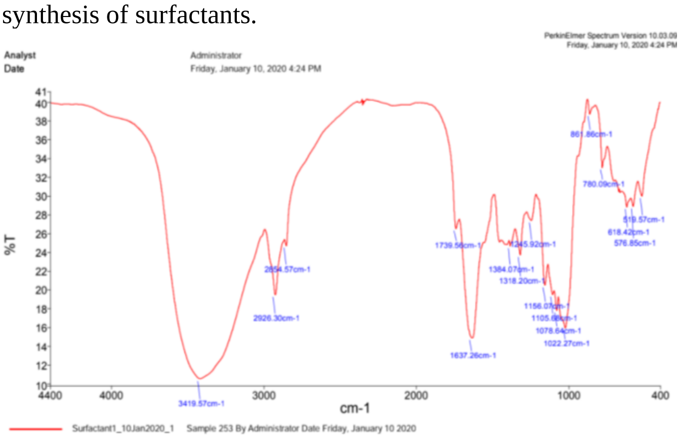

Fig 1: FTIR Analysis of Hibiscus Leaves

The conductivity experiment carried out on the hibiscus leaf-based surfactant showed that the critical micelle concentration is around 2.2wt%. The plot of conductivity vs surfactant concentration is shown in Fig 2. The point at which there will be a turning point (2.2 wt%) in the conductivity plot highlights the CMC. Below the CMC, formation of micelles is impossible.

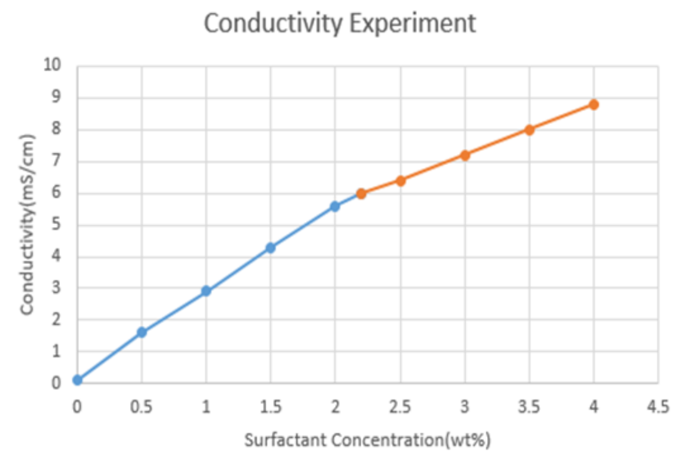

Fig 2: Conductivity of Solution(mS/cm) Vs Surfactant Concentration(wt%)

The conductivity experiment is followed by the measurement of interfacial tension (IFT). The results of IFT experiment are put in Fig 3. From the graph it is quite prominent that at a concentration of 2.2 wt% surfactant in solution, the IFT is reduced. Beyond CMC, the interfacial tension is reduced but too much accumulation reduces the oil upliftment due to its absorption by oil molecules.

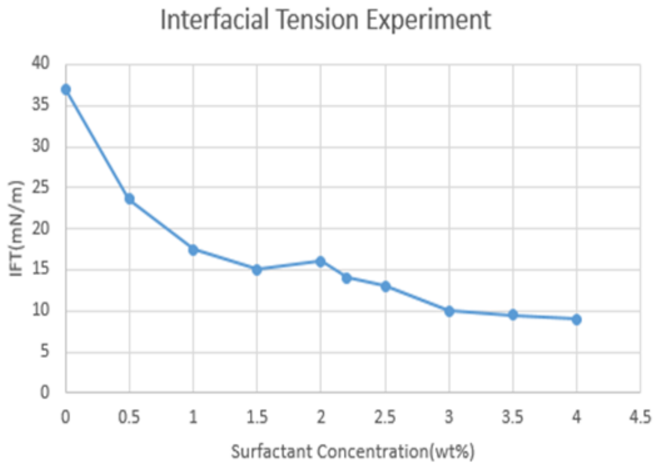

Fig 3: Interfacial Tension (mN/m) Vs Surfactant Concentration(wt%)

The pH analysis of soil reveals that the surfactant concentration in solution doesn't have effect on the pH. Even though the concentration was increased, the pH of the soil was constant at 6.7 which is ideal for growth of the plants. So, the proposed surfactant solution doesn't impact the environment and cause soil pollution.
Comparison of the chemical activity of experimented surfactant is also made with the other conventional and bio-surfactants through literature survey. It has been found that extract from *Glycyrrhiza Glarba* could reduce the interfacial tension from 3.5 mN/m to 7.9 mN/m. Also, there is reduction of IFT from 32 mN/m to 8.9 mN/m using the surfactant solution of *Sedila rosmarinos*. The reduction of interfacial tension using different natural surfactant is so close to that of the chemical surfactant that we can think of replacing the chemical surfactants by bio-surfactants.[8],[9]

CONCLUSION

The experimental study tries to find out the impact of bio-surfactant synthesized from *Hibiscus rosa-sinensis* in enhanced oil recovery as well as environment. The surfactant shows to reduce the interfacial tension between oil and water solution to 13.2 mN/m from 37.1 mN/m at a critical micelle concentration of 2.2 wt%. The environmental impact of these surfactant solution is less. It doesn't impact the soil pH much. The pH meter indicates the soil pH to be 6.7 even after dumping the surfactant solution. Thus, this can be a good replacement to the chemicals which cause environmental degradation by means of lowering pH of the soil as well as ground water.

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