HENNA EXTRACT AS A POTENTIAL SACRIFICIAL AGENT IN REDUCING SURFACTANT ADSORPTION ONTO QUARTZ SAND AND KAOLINITE

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ABSTRACT

Surfactant flooding is one of enhanced oil recovery techniques to increase oil recovery. However, the main concern of this technique is the adsorption of surfactant onto reservoir rock which can reduce the effectiveness of the surfactant in reducing interfacial tension of oil and water. In this case, surfactant adsorption is normally reduced with the help of a sacrificial agent (SA). Studies have recently discovered the potential of plant extracts as an environmentally friendly and easily available alternative to chemical SAs. The main objective of this study is to evaluate the performance of henna extract as a SA in reducing surfactant adsorption. First, henna leaves were extracted and characterised to determine the functional groups responsible for adsorption. The ability of the henna extract to adsorb onto quartz sand and kaolinite was then analysed. Moreover, the adsorption limitation of the henna extract onto both quartz sand and kaolinite in different salinities, pH, and temperature was investigated. Finally, the performance of the surfactant adsorption onto quartz sand and kaolinite in the presence of the henna extract was evaluated. The results show that phenolic compounds are responsible for adsorption of the henna extract. Besides, twice the amount of henna extract was adsorbed onto kaolinite than quartz sand. Also, the henna extract managed to adsorb further when salinity was increased and pH was lowered. However, the adsorption of henna extract reduced with increasing temperature. The adsorption of henna extract is mainly through hydrogen bonding and electrostatic attractions while hydrophobic interactions play a minor role in the adsorption process. The henna extract reduced surfactant adsorption onto quartz sand and kaolinite by 46% and 55%, respectively, in 30,000 mg/L of NaCl. At pH 3, the surfactant adsorbed onto quartz sand and kaolinite was reduced by 32% and 39% respectively. Meanwhile, at 25^{o}C, the surfactant adsorption onto quartz sand and kaolinite was reduced by 23% and 36%, respectively. This finding proves the profound reduction in surfactant adsorption with the addition of henna extract, suggesting the possibility of utilising the extract as a sacrificial agent to reduce surfactant adsorption. In conclusion, the adsorption behaviour of henna extract in different salinity, pH, and temperature was successfully demonstrated and the henna extract was found effective as a sacrificial agent in reducing surfactant adsorption.

ABSTRAK

Pembanjiran surfaktan ialah teknik perolehan minyak tertingkat bagi meningkatkan perolehan minyak. Walau bagaimanapun, kebimbangan utama tentang teknik ini ialah penjerapan surfaktan pada batuan reservoir yang boleh menjejaskan keberkesanan surfaktan dalam mengurangkan ketegangan antara muka air dan minyak. Dalam kes ini, penjerapan surfaktan biasanya dikurangkan menerusi penggunaan agen korban (SA). Kajian terkini telah mengetengahkan potensi ekstrak tumbuhan sebagai pilihan lain kepada SA kimia yang mesra alam dan mudah diperoleh. Objektif utama kajian adalah untuk menilai prestasi ekstrak inai sebagai SA bagi mengurangkan penjerapan surfaktan. Pertama, daun inai diekstrak dan diciri bagi menentukan kumpulan berfungsi yang mengawal penjerapan. Keupayaan ekstrak inai untuk terjerap pada pasir kuartza dan kaolinit kemudiannya dianalisis. Selain itu, turut dikaji ialah had penjerapan ekstrak inai terhadap pasir kuartza dan kaolinit pada kemasinan, pH, dan suhu yang berbeza. Akhirnya, prestasi penjerapan surfaktan terhadap pasir kuartza dan kaolinit dengan kehadiran ekstrak inai dinilai. Keputusan menunjukkan bahawa sebatian fenolik dalam ekstrak inai yang menyebabkan berlakunya penjerapan ekstrak inai. Selain itu, dua kali ganda jumlah ekstrak inai terjerap pada kaolinit berbanding pasir kuartza. Ekstrak inai juga berjaya menjerap secara lebih ketara apabila meningkatnya kemasinan dan menurunnya pH. Walau bagaimanapun, penjerapan ekstrak inai berkurang berikutan meningkatnya suhu. Penjerapan utama ekstrak inai adalah menerusi ikatan hidrogen dan tarikan elektrostatik, manakala interaksi hidrofobik menghasilkan kesan yang kecil dalam proses penjerapan. Ekstrak inai mengurangkan penjerapan surfaktan pada pasir kuartza dan kaolinit masing-masing sebanyak 46% dan 55% dalam larutan NaCl berkepekatan 30,000 mg/L. Pada pH 3, surfaktan yang terjerap pada pasir kuartza dan kaolinit masing-masing berkurang sebanyak 32% dan 39%. Sementara itu, pada suhu 25^{o}C, penjerapan surfaktan pada pasir kuartza dan kaolinit berkurang masing-masing sebanyak 23% dan 36%. Penemuan ini membuktikan pengurangan yang ketara dalam penjerapan surfaktan seiring dengan penambahan ekstrak inai. Pencapaian ini mengetengahkan potensi ekstrak inai sebagai agen korban bagi mengurangkan penjerapan surfaktan. Kesimpulannya, kajian ini telah mengetengahkan tingkah laku penjerapan ekstrak inai pada kemasinan, pH, dan suhu yang berbeza, dengan ekstrak inai sesuai dijadikan agen korban bagi mengurangkan penjerapan surfaktan.

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LIST OF ABBREVIATIONS

ABTS	-	2,2'-azino-bis(3-ethylbenzothiazoline-6-sulphonic acid)
BPA	-	Bisphenol-A
BHT	-	Butylated Hydroxytoluene
CMC	-	Critical Micelle Concentration
CTAB	-	Cetyl Trimethyl Ammonium Bromide
DIW	-	Deionized Water
DMBA	-	7, 12-Dimethylbenz (a) anthracene
DPPH	-	2,2-diphenyl-1-picryl-hydrazyl-hydrate
EDL	-	Electrical Double Layer
EDX	-	Energy Dispersive X-Ray
EOR	-	Enhanced Oil Recovery
FCM	-	First Contact Miscible
FTC	-	Ferric Thiocyanate
FTIR-ATR	-	Fourier Transform Infrared-Attenuated Total Reflectance
HCl	-	Hydrochloric Acid
IFT	-	Interfacial Tension
MCM	-	Multiple Contact Miscible
NaCl	-	Sodium Chloride
NaOH	-	Sodium Hydroxide
NaCl	-	Sodium Chloride
OOIP	-	Original Oil In Place
PSA	-	Particle Size Analyzer
SA	-	Sacrificial Agent
SDS	-	Sodium Dodecyl Sulfate
SEM	-	Scanning Electron Microscope
TBA	-	Thiobarbituric Acid
TGA	-	ThermoGravimetric Analyzer
UTM	-	Universiti Teknologi Malaysia
UV-Vis	-	Ultraviolet Visible
XRD	-	X-Ray Diffraction

$Ca(OH)_2$	-	Calcium hydroxide
$MgSO_4$	-	Magnesium Sulfate
Na_2CO_3	-	Sodium Carbonate
Na_2SO_4	-	Sodium Sulfate
$NaHCO_3$	-	Sodium Bicarbonate

LIST OF SYMBOLS

q_e	-	Adsorption at equilibrium
q_m	-	Maximum adsorption capacity
C_e	-	Concentration at equilibrium
K_L	-	Langmuir constant
K_F	-	Freundlich constant
λ	-	Wavelength of the X-rays
Cu	-	Copper
K_α	-	X-ray energy emission
K_β	-	X-ray filter
n	-	Integer
d	-	Spacing between planes
θ	-	Diffraction angle

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CHAPTER 1

INTRODUCTION

1.1 Background of Study

Enhanced oil recovery (EOR) is defined as the technique used to increase the amount of oil that can be recovered from an oil reservoir. It involves the process of obtaining stranded oil that has not been recovered from a reservoir through previously implemented extraction processes. Around 30% to 60% of residual oil can be retrieved via EOR methods compared to just 20% to 40% of oil recovered via primary and secondary recovery methods (Sheng, 2013). Tertiary oil recovery processes that implement EOR methods vary according to the type of approach and the reservoir characteristics. EOR processes can be classified into four categories: miscible flooding, chemical flooding, thermal flooding, and microbial flooding.

Chemical flooding is the addition of one or more chemical compounds to an injected fluid either to increase the displacement efficiency of the injected fluid or to improve the sweep efficiency of the injected fluid. There are three general methods that falls under chemical flooding technology. First is polymer flooding, which is used to increase the displacing fluid viscosity, leading to improved sweep efficiency in the reservoir. Second and third methods, which are surfactant flooding and alkaline flooding, respectively, make use of chemicals that reduce the interfacial tension (IFT) between oil and displacing fluid.

A surfactant is a surface-active agent that has molecules containing a hydrophobic component and a hydrophilic component. Surfactants are generally divided into four groups, namely non-ionic, cationic, anionic, and zwitterionic. Ionic surfactants have a positively or negatively charged organic tail head. Cationic surfactants are positively charged, whereas anionic surfactants are negatively charged.

Non-ionic surfactants have no charge at all while zwitterionic surfactants have both positive and negative charges (Sheng, 2011).

The primary function of surfactants is to reduce the IFT between water and the reservoir oil. The surfactant travels to the interface between the oil and water phases and helps make the two phases mixable. When the interfacial tension is reduced, the capillary forces are reduced, and the oil can flow. According to Terry (2001), IFT can be reduced from 30 dynes/cm, found in typical waterflooding applications, to 10−3 dynes/cm with the addition of as little as 0.1–5.0 wt% surfactant to water-oil systems.

Surfactant flooding is a process during which surfactant is injected into the reservoir (Kamranfar & Jamialahmadi, 2014) to lower the oil/water IFT and to enhance the displacement efficiency during oil recovery (Bera *et al.*, 2013; Olajire, 2014; Zendehboudi *et al*., 2013). IFT reduction via surfactants decreases capillary forces and allows the trapped oil bank to flow (Olajire, 2014). However, the adsorption of surfactants onto the reservoir rock or the loss of the surfactant can reduce the available surfactant molecules that can adsorb onto the air/water interface; hence, reducing the effectiveness of the surfactant in reducing IFT (Apaydin & Kovscek, 2000; Daoshan *et al*., 2004; ShamsiJazeyi *et al*., 2014b; Somasundaran & Zhang, 2006).

Due to its negative impacts, efforts have been made to minimise surfactant adsorption onto reservoir rocks. It is known that surfactant adsorption is strongly dependent on the type of surfactant, the pH of the solution, the salinity of the solution, and the mineralogical properties of the reservoir rock (Paria *et al.,* 2005; Shamsijazeyi *et al.,* 2013; Weston *et al.*, 2014).

Several methods have been applied to reduce surfactant adsorption. Some studies investigated the use of surfactants and reservoir rock with the same charge and concluded that the adsorption of anionic surfactant decreased on sandstone while the adsorption of cationic surfactant decreased on carbonate due to the electrostatic repulsion between the adsorbent and the type of surfactant used (Paria & Khilar, 2004; Somasundaran & Zhang, 2006; Manev *et al.*, 2008; Ma *et al*., 2013; ShamsiJazeyi *et al.,* 2014a). However, because of the heterogeneity of the reservoir rock, especially with the presence of clay, the above statements might not hold true. Recently, several works, including Amirianshoja et al. (2013) and Yekeen et al. (2016), attempted to study the influence of the mineralogical composition of clay on the adsorption of surfactants to explain the behaviour of such compounds in the reservoir. They concluded that the adsorption of the surfactants by clay minerals depended on both the nature of the surfactant and the structure of the clay mineral.

Besides leveraging on the electrostatic repulsion between similarly charged surfactant and rock, an alkali such as sodium hydroxide or sodium carbonate is also typically used as a chemical agent to lower the adsorption of anionic surfactants. The pH of an aqueous solution is one of the essential parameters that determine the adsorption of surfactant. Theoretically, as pH increases, the adsorption of an anionic surfactant decreases. Some works on this subject include Dang et al. (2013) and Budhathoki et al. (2016), who found that when pH increased due to the addition of alkali, more hydroxyl anions (OH^-) would be present in the solution. Hence, the net negative surface charge will increase, in turn increasing the electrostatic repulsion, thereby reducing the adsorption of anionic surfactants onto sandstone. Ahmadall *et al.* (2013) mentioned that, at low pH i.e. in the presence of hydrogen cations (H^+), the surface charge of the rock became positive. These results indicate that cationic surfactants have significantly low adsorption onto carbonates. However, the use of alkali induced some problems such as severe scaling in the near wellbore and the production system (Chen *et al.*, 2013; Zhang *et al.*, 2015; Tay *et al.*, 2015) and difficulty to produce an emulsion for demulsification (Chen *et al.*, 2013).

Besides alkali, a sacrificial agent (SA) is also a promising method for reducing surfactant adsorption. Kalfoglou (1979) listed two important characteristics of satisfactory sacrificial material. First, it must be less expensive than the surfactant since it is to be sacrificed or adsorbed by the rock formation, probably never to be recovered. The second important characteristic of a sacrificial agent is that the presence of such adsorbed sacrificial material must retard or eliminate the subsequent adsorption of the surfactant onto the adsorption site of the rock formation, which refer to those parts of the surfaces of the pores of the rock formation capable of adsorbing a chemical compound from a solution on contact. Weston et al. (2014) found that surfactant molecules were able to form admicelles on a solid surface, which is the primary reason for adsorption to occur. The SA method prevents the formation of these admicelles, and thus lowers adsorption, making it a very desirable method.

The application of SA in EOR has been documented in previous studies (Feng *et al.*, 2012; Shamsijazeyi *et al.*, 2013; Weston *et al.*, 2014; Wang *et al.*, 2015; Budhathoki *et al.*, 2016). These works concluded that, the presence of a SA enhanced surfactant performance, reduced surfactant adsorption onto the rock formation, and resulted in a good correlation between EOR and reduced surfactant adsorption. Lignosulfonate and polyelectrolyte are the most common SAs that have been tested. Daud et al. (2009) reported the reduced adsorption of an anionic surfactant after lignosulfonate was used as a pre-flush method before injecting the surfactant. ShamsiJazeyi et al. (2014) reported the ability of polyelectrolyte in reducing the adsorption of anionic surfactant onto carbonates and clay minerals. Moreover, Budhathoki et al. (2016) demonstrated that the addition of polyelectrolyte successfully reduced surfactant adsorption.

However, the materials used in reducing surfactant adsorption included chemicals that may be hazardous and not environmentally friendly. Thus, current research is looking into suitable materials that could serve as a SA or inhibitor that are also environmentally friendly, readily available, and derived from natural products.

In this research, a natural plant-based SA was investigated, namely *Lawsonia inermis* L., known as henna. This material is generally considered native to Africa and Asia. It is a tree with numerous branches and thickly surrounded by small green leaves (Musa & Gasmelseed, 2012; Patel *et al.,* 2013). Henna has attracted the attention of researchers worldwide for its cosmetics and medicinal properties (El-Babili *et al.*, 2013; Jain *et al*., 2010; Singh & Narke, 2015). In West Asia and Africa, henna is used as a hair dye and in the shampoo industry due to its proven dermatological effects (Rehan, 2003). Besides that, henna has been applied as a corrosion inhibitor in past studies (Chaudhari & Vashi, 2016; Hamdy & El-gendy, 2013; Rajendran *et al*., 2009). For example, Moslemizadeh et al. (2015) revealed that when exposed to sodium bentonite, the henna extract managed to reduce the swelling of sodium bentonite more so than polyamine and potassium chloride, owing to henna's inhibitive capability.

1.2 Problem Statement

Some studies proved henna extract to be a functional inhibitor that prevented the corrosion of metal (Vashi & Prajapati, 2017; Zulkifli *et al.*, 2017). Furthermore, another study tested henna as an inhibitor, showing that it successfully reduced the swelling of sodium bentonite clay in aqueous solution and improved wellbore instability (Moslemizadeh *et al.*, 2015). Both studies concluded that henna extract showed remarkable inhibition properties. Since more than 60% of the world oil reserves are found in sandstone reservoirs (Dimri *et al.*, 2012), nevertheless, only a few studies have investigated the adsorption of henna extract onto quartz sand and kaolinite, since the major mineral in sandstone reservoirs is quartz sand and kaolinite is the most common clay mineral in the sandstone reservoirs. Hence, this study was done to shed light on the adsorption ability of henna extract onto quartz sand and kaolinite.

Several researchers have conducted comprehensive studies on the influence of different parameters of surfactant adsorption, namely the impact of added salts, and reported that surfactant adsorption increased with increasing NaCl concentrations (Budhathoki *et al.*, 2016; Safarzadeh *et al.*, 2016). Besides that, the extent of surfactant adsorption onto reservoir rock and clay minerals depended mostly on the concentrations of the electrolyte and the mineralogical composition of the adsorbent. Therefore, in order to prevent surfactant adsorption in environments with high salinity and different mineralogical content, there is a need to investigate the limitations of henna extract in a saline environment.

It is a fact that pH plays a vital role in surfactant adsorption, as it is directly related to the charge of the solid surface. High pH will lead to high OH^- ions in the solution, creating a negatively charged solid whereas low pH will lead to high H^+ in the solutions, creating a positively charged solid. In the case of quartz sand, which is negatively charged, and coupled with an anionic surfactant in a high pH environment, surfactant adsorption could be reduced due to electrostatic repulsion (Li & Ishiguro, 2016). Nevertheless, in a low pH environment, which has more positive ions (H^+), severe surfactant adsorption will occur, owing to the electrostatic attraction between the anionic surfactant and the positively charged quartz sand surface (Julius *et al.*, 2015). Hence, this study aims to investigate the adsorption of henna extract onto quartz sand and kaolinite under the effect of varying pH to determine the limitations of henna extract in different pH environments.

Another important factor to consider is the influence of temperature. Temperature significantly influences the adsorption of surfactant onto a reservoir rock surface (Barati *et al.,* 2016). Nevertheless, the influence of temperature on the ability of henna extract to adsorb onto quartz sand and kaolinite is still unclear. Hence, there is a need to study the limitations of henna extract adsorption onto quartz sand and kaolinite under different temperatures.

Furthermore, there is an absence of detailed knowledge on the performance of henna extract as a SA in reducing surfactant adsorption, taking into account the effect of salinity, pH, and temperature. Thus, the efficiency of the adsorption of henna extract onto quartz sand and kaolinite as a SA in reducing surfactant in varying saline conditions, pH, and temperatures was identified.

1.3 Objectives

This research aims to evaluate the performance of henna extract as a sacrificial agent (SA) in reducing surfactant adsorption. To achieve this aim, several key objectives have been identified, as follows:

1. To characterize the henna extract from fresh henna leaves.

2. To analyze the adsorption ability and the adsorption limitations of henna extract onto quartz sand and kaolinite in different salinities, pH and temperature.

3. To evaluate the performance of henna extract in reducing surfactant adsorption onto quartz sand and kaolinite in different salinities, pH and temperature.

1.4 Scope of Study

In this study, laboratory work was done to evaluate the performance of henna extract as a SA in reducing surfactant adsorption. The scope of this work was limited to the static adsorption test. Two different minerals were used, namely quartz and kaolinite to represent sandstone reservoir. Sodium Dodecyl Sulfate (SDS) was used as the anionic surfactant. Henna extract at different concentrations (3000 mg/L to 8000 mg/L) and SDS at different concentrations (500 mg/L to 8000 mg/L) were used accordingly in this study. The influence of salinity (10000 mg/L, 20000 mg/L, 30000 mg/L, and 50000 mg/L) was assessed using NaCl salt. The influence of pH (pH 3, pH 4, pH 9, and pH 11) was assessed using hydrochloric acid (HCl) and sodium hydroxide (NaOH), which were used to adjust the pH accordingly. The influence of temperature (25^{o}C, 45^{o}C, 65^{o}C, and 75^{o}C due to the limitations of the equipment) on the henna extract adsorption was also tested.

1.5 Significance of Study

The adsorption of surfactant from the solution to the solid surface is of technological, environmental, and biological importance. However, in many cases, especially in the petroleum industry, and particularly in EOR, the adsorption of surfactant can be a detrimental factor to the process of oil recovery as well as the economy. Hence, this study was carried out to address this issue. There is a risk of losing the surfactant solution to its adsorption onto the rock formation in the reservoir; therefore it is vital to minimise the adsorption of surfactant in the oil and gas industry.

Hence, this research investigates the ability of henna extract, which is environment-friendly and readily available, as a sacrificial agent in reducing the adsorption of surfactant.

1.6 Thesis Outline

This thesis is divided into five chapters. The problems regarding surfactant flooding and the methods to prevent this issue are presented in the current chapter. Next, the problem statement, as well as the objectives of the research, are reported, followed by the scope and significance of the study. In Chapter 2, the background information about surfactants, reservoir rock, and adsorption is explained. In Chapter 3, the methodology of the research is clarified. Chapter 4 discusses the result and discussion of the current study and finally, Chapter 5 outlines the conclusion for this research as well as recommendations for further studies.

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