Petroleum 7 (2021) 152-159

Contents lists available at ScienceDirect

Petroleum



journal homepage: www.keaipublishing.com/en/journals/petIm

Experimental investigation of the application of Eucalyptus bark to prevent lost circulation in pay zones with acid dissolution capability



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ARTICLE INFO

Article history: Received 28 October 2019 Received in revised form 26 January 2020 Accepted 14 April 2020

KeAi

Keywords: Eucalyptus bark Lost circulation Acid solubility Drilling mud Fluid loss

ABSTRACT

Loss of drilling fluid is a common problem during the drilling of wells and it restricts the appropriate functionality of muds. Drilling fluid loss significantly increases drilling costs and non-productive time as well as the drilling operation risks. Various investigations have been carried out in order to find appropriate mud additives that either block fractures and pores or reduce fluid loss by improving the fluid rheology. Cheap, environmentally friendly and effective additives are still required by the drilling industry. Hence, the application of available materials in each region, to produce appropriate additives, is a challenge for the oil industry.

In this study, Eucalyptus Camaldulensis (EUC) bark powder has been chosen as a new, fibrous, cheap, environmentally friendly and available material to control fluid loss, particularly in southern Iran. Different characterization tests, such as acid dissolution and fluid loss control, were carried out to study the performance of the new proposed additive. Removal by hydrochloric acid and sulfuric acid were studied at various acid concentrations and temperatures. Dynamic fluid loss was also measured at different EUC concentrations. Our study showed that EUC powder can reduce the final fluid loss by 88 -97%, the initial fluid loss by 45–66%, and the total loss by 87–94%, which is a satisfactory level.

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1. Introduction

Loss / Loss of drilling fluid circulation is a common problem in almost all drilling wells, especially in highly permeable and fractured formations. The invasion of fluids to the formations' pores and fractures weakens the performance of drilling mud and increases non-productive drilling time, project costs, surge pressure and cementing problems, as well as creating excessive drag. It can result in a stuck pipe, reduced productivity and, most importantly, a drop in the drilling fluid column in the well, which may increase potential for blow out.

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The amount of mud loss varies for different wells and various downhole conditions. Mud loss is usually more severe in the lower parts of the formation. Generally, the fluid loss intensity is classified into three categories, as described below [1-3]:

- (1) Seepage loss (fluid loss rate up to 10 bbl/h);
- (2) Partial loss (fluid loss rate between 10 and 500 bbl/h);
- (3) Complete loss (fluid loss rate more than 500 bbl/h).

An attractive option for controlling fluid loss is the application of natural materials as an additive to drilling mud. Table 1 shows a list of proposed materials to control lost circulation and filtration.

In 1987, the performance of commonly used lost circulation materials (LCM) (such as mica, cottonseed hulls, and ground walnut shells) in controlling fluid loss in fractured formations was experimentally compared to a new material composed of thermoset rubber, for different reservoir drilling fluid types (water-based, oilbased, mineral-oil-based and invert-emulsion oil-based). In general, the results indicated that thermoset rubber was superior to the other mentioned LCMs in controlling fluid loss, due to the optimum particle size distribution (PSD), high particle strength,

https://doi.org/10.1016/i.petlm.2020.04.005

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Table 1

Natural drilling fluid additives suggested by different researchers [4,5].

Inventor	Natural Material	Function
Green (1984) [6]	Ground cocoa bean shells	Lost circulation material
Burts (1997) [7]	Rice fractions (rice hulls, rice tips, rice straw and rice bran)	Lost circulation material
Cremeans et al. (2003) [8]	Cotton seed hull	Lost circulation material
Macquoid and Skodack (2004) [9]	Coconut coir	Lost circulation material
Ghassemzadeh (2013) [10]	Fibers	Lost circulation material
Okon et al (2014) [11]	Bice husk particles	Filtration controlling
Dagde and Nmegbu (2014) [12]	Groundnut Husk	Filtration controlling
Amanullah et al. (2016) [13]	Date seed powder	Filtration controlling

deformability, mud compatibility, and thermal stability (up to 400 C). It was observed that at high pressure differentials (600–1000 psi) and when the solid concentration in the mud was low, the granular LCMs (such as individual, coarse walnut shells or medium thermoset rubber), exhibited channeling effects and channels were formed within the LCM deposit, which intensify the fluid loss. Also, it was illustrated that, in some drilling fluids, high concentrations of LCMs would lead to a poorer performance compared with lower LCM concentrations, due to alterations in mud rheology [3].

In 2002, the influence of five ranges of commercially available calcium carbonate particle sizes in water-based drilling fluid samples (containing base brine, viscosifier, starch and bridging agents) on fluid loss control capability was investigated utilizing the Permeability Plugging Test (PPT) [14].

In 2007, the effects of walnut shells and polymer XT presence on the rheological properties and fluid loss control capabilities of water-based drilling fluid samples were studied and it was concluded that walnut shells may be a good option for plugging the pores and fractures in lost zones [15].

The performance of cheap fluid loss control agents such as graphite, calcium carbonate, nut shells, and cellulosic fibers was investigated in samples of simple water/bentonite drilling fluids. LCMs with deformable and irregular particle shapes, such as nut shells, have the capability of plugging large fractures when used either individually or in combination with other LCMs. It was also found that, although increasing the LCM concentration generally leads to a better fluid loss control capability, there are some cases in which the concentration of LCM should not exceed a critical maximum percentage [16].

Bagasse, peanut hulls and sawdust were used to plug lost zones and control the fluid loss in samples of water-based drilling fluid comprising water and 22.5 lb/bbl bentonite [17]. The results indicated that three major parameters control the amount of fluid loss, namely the LCM material type, concentration, and particle size distribution. Peanut hulls (especially fine particle sizes with better filling properties) perform better when compared to other materials. LCMs with finer sized particles have better filling characteristics, higher pressure resistance, and better fluid loss control capabilities due to their higher surface area [17].

Hossain and Wajheeuddin (2016) analyzed pulverized grass as a cost effective and environmentally friendly loss control agent additive for water/bentonite drilling fluid samples [5]. The fluid loss and rheology of water-based drilling fluid samples containing XC polymer and modified starch with different concentrations of calcium carbonate (10, 20 and 30 lb/bbl) were analyzed utilizing a PPT (Permeability Plug Test) apparatus at a temperature of 75°C and pressure of 300 psi [2]. When the particle size of the LCM is larger than the mean pore size diameter, then increasing the LCM concentration increases the filtration performance.

Onuh (2017) studied the fluid loss of water/bentonite drilling fluid samples containing different concentrations of corn cob and coconut shell (up to 10 lb/bbl) as cost effective, non-toxic, biodegradable and environment friendly fluid loss control additives [18]. Also in 2017, a smart expandable material created from shape memory polymers was proposed as a cheap, light weight, non-toxic and biodegradable lost circulation control agent. The results proved a fluid loss control capability along with pressure resistance up to 5000 psi [19].

The effectiveness of two new ecofriendly and biodegradable LCMs (namely, RIPI-LQ (a cellulosic material made from a special type of grass) and X1-Seal (a material made from a flowering plant)), was studied at different concentrations to observe how they controlled the fluid loss of a heavy-weight mud and an oil-based mud [1]. Davoodi et al. (2018) substituted pistachio shell as a cheap, degradable and environment friendly additive in the form of two distinct particle sizes at three concentrations (up to 9 lb/bbl) in water based mud samples. The results showed that Pistachio powder reduced fluid loss more than PAC. Moreover, they measured pistachio powder solubility in three concentrations of hydrochloric acid (i.e. 15%, 28% and 37%) which was measured as 22.5%, 53.6% and 82.0% after 72 h, respectively [4].

Ramasamy and Amanullah (2018) developed a novel fibrous lost circulation material (Arc Fiber SF) using date tree waste available locally as the raw material. HPHT filtration control experiments at 212°F and 500 psi proved the ability of fibers as a cheap LCM in controlling the filtrate. Novel fibrous and particulate LCM products were also developed by physio-mechanical treatment and processing of various waste components of date palm trees [20]. Experimental studies by PPT apparatus at 500 and 1500 psi pressure values and 250°F temperature using 2 mm slotted disc demonstrated the capability of these components to block pores. Engineered fiber blends produced from date palm waste showed similar or better performance than the commercial LCM blends [21].

Based on the above research, there are different natural materials that can be used to control fluid loss. Hence, it is critical to find appropriate cheap candidates to be used as an additive to drilling mud. The lost circulation materials currently being used still have disadvantages, such as being hazardous to the environment and personnel, damaging pay-zones, having low acid solubility, and failing to seal large fractures or plugging drilling tools. As a result, work on finding new additives with more advantageous characteristics is continuing [19].

Clearly, an effective LCM agent should minimize fluid loss as much as possible and have good solubility in acid in order to be removed after drilling. As most Iranian reservoirs are fractured and have a huge amount of fluid loss, it is important to find the most appropriate material with acceptable solubility and loss control performance.

In this study, Eucalyptus camaldulensis (EUC) bark powder - a lignocellulosic resource – was selected as being an available, cheap, safe, and environment friendly material that could be used as a fluid loss control additive. Different tests were carried out to study the performance of EUC in controlling loss in pay-zone sections during drilling and to analyze their solubility in sulfuric acid and

hydrochloric acid. Different characterization measurements, such as Scanning Electron Microscope (SEM), rheology tests, and pH analyses were carried out to study the effect of acid on EUC powder and, also, the effect of EUC on mud properties. The dynamic fluid loss of a water-based drilling mud with EUC powder as an additive was studied in this work. Moreover, the mud invasion depth and extent of formation damage due to mud circulation were investigated by a CT scan imaging approach.

2. Methodology

2.1. EUC powder preparation

EUC bark was gathered from E. Camaldulensis (EUC red gum) trees in the Petroleum University of Technology yard. This was washed and soaked in tap water in order to remove any dust or other unwanted material associated with bark surfaces and dried in the fresh air for one day. The bark pieces were then put into an oven for 1 day at 105°c to remove any residual adsorbed water. Dry bark pieces were then ground, using a 8300-grinder, and packed in ziplock plastic bags for additional processing. Finally, the ground and packed EUC powder was sieved using a 800-µm sieve, to remove any out of range and coarse fragments and stowed for future experiments.

2.2. Dissolution in acid

In order to analyze the dissolution of the powder in acid, 40 ml of different acid solutions with different acid concentrations were added to 2 g of the prepared powder, to investigate the effects of time, acid concentration, and temperature on the solubility. Sulfuric acid and hydrochloric acid were used at concentrations of 15%, 28% and 37%, and at 25 °C (room temperature) and 40 °C (the reservoir environment). Solubility was measured after 1 day and 2 days of acid/powder contact, to study the effects of time. After each acid dissolution experiment, a centrifuge (model 5082W MSE) was used for 5 min at 4500 RPM to separate the residual powder from the solution for further drying and weighting steps.

To prepare different acid solutions, water was added to sulfuric acid at 96% mass percent and hydrochloric acid at 37% mass percent. For each test the volume of distilled water added to the initial acid was calculated by:

$$V_{dw} = V_{A,f} \left(1 - \frac{m_f \rho_{a,f}}{m_i \rho_{a,i}}\right) \tag{1}$$

where $V_{A,f}$ is the final acid volume, *m* is acid concentration (gr/gr), and ρ_a is the acid density (gr/ml). The density of acids at different concentrations and at different temperatures were measured by Ref. [6].

After each separation process, the solution phase was discharged and water was again added to the residual solids to repeat the separation process. This step was repeated five times to remove

 Table 2

 Mud additives used in this experiment plus additive concentrations and functions.

Additive	Amount	Function
Distilled Water	350 cc	Continuous Phase
Soda Ash (Na ₂ CO ₃)	1 g	Hardness Removal
Caustic Soda (NaOH)	1.5 g	Alkalinity Control
Salt	80 g	Weighting Agent
Starch LV	12 g	Fluid loss Control
Xanthan Gum	1 g	Viscosifier
Eucalyptus Powder	10-30 g	Lost Control Agent

all acidic traces from the mixture. After separation was completed, falcon tubes were put into an oven at 105 °C for 1 day to ensure complete drying of the powder. The tubes were then weighted in order to calculate the solubility of the powder. Samples of EUC powder were scanned before and after acid dissolution, using SEM apparatus to visualize the effects of acid on the size and shape of the EUC powder.

2.3. Fluid loss control analysis

2.3.1. Preparation of base mud and measurement of basic parameters

Based on the mud recipe typically used in the drilling of wells in the study area, different additives were selected and mixed with the base water. Table 2 shows the mud additives used in this study.

All additives were mixed in order (from the top to the bottom of Table 2) using an IKA RW16 basic mixer at its highest applicable RPM (1200). Before pouring any new additive, the solution was stirred for 5 min to ensure complete dissolution or mixing.

Mud samples were prepared using a JB-12KD mixer at 6000 RPM. Rheological properties were measured by a Fann-Visometer (model 35SA). Mud weight was measured by a Fann-Mud balance system. The Alkalinity of the prepared samples was measured using a calibrated AZ pH meter.

2.3.2. Dynamic fluid loss measurement setup

Fig. 1 shows the set-up to analyze lost control capability of EUC powder. The system consists of a dosing pump (to inject at constant rates), a PVC cell (for cross flow filtration), an aluminum standing board (to change the direction of flow), two aluminum tanks (for the preparation of mud and spacer fluid), a mixer (to prepare a homogeneous mud), a back pressure valve (to provide the differential pressure between the mud and reservoir in an overbalance drilling condition), an air pump, and a tank (for the simulation of static fluid losses).

Using this setup, it is possible to mimic real drilling conditions. A synthetic core was prepared using glass beads and epoxy resin to mimic the porous sandstone media; this better represented the reservoir condition than filter paper. A special PVC core holder cell was designed for this system, as shown in Fig. 2. Hence, this cell can be used during CT scan imaging procedure.

The three cracks at the bottom of the cuboid serve as the outlet routes and allow linear flow through the porous medium.



Fig. 1. Schematic of Set-up to analyze lost control capability of EUC powder.



(a)



(b)



(c)



(d)

Fig. 2. PVC core holder cell. (Outlet routes-top left, Replaceable funnel-top right, Junction and the connected cell-bottom right & left).

Furthermore, there is a replaceable funnel connected to the bottom of the cuboid which concentrates the flow being expelled from the cracks, and which then pours into the container below the cell. The junction and the connected cell are also shown in this Figure. The cell and the junction were connected to each other using two fasteners. Moreover, there are two Siliconic Gaskets, one between the funnel and the cuboid and the other between the cuboid and the junction, to prevent any possible fluid leakage during the test.

In order to build a synthetic core, two different size ranges of glass beads (500–700 μ m and 700–900 μ m), purchased from Rahnama Glass Beads Industries were used. Fig. 3 shows samples of the glass beads used in our experiments. At the top of the synthetic core, glass beads were mixed with epoxy resin to avoid any possible movement of the glass beads into the flow. At the base of the core, epoxy was used to prevent downfall of the glass beads. The complete arrangement was slowly stirred and poured into the PVC cell. For a faster solidification of the mixture, the cell is put into the oven for 1 day at 30 °C.

The synthetic core built in the PVC cell is shown in Fig. 4. The prepared cell was put in its specific position as shown in Fig. 5. Stirring of the mixture was performed slowly to avoid any possible bubble production in the mixture.

Fig. 6 depicts all of the components that comprise the dynamic filtration set up and the PVC cell. According to Fig. 6, drilling fluid circulates in to the PVC cell after preparation in the mud tank. The cross flow of the drilling fluid circulates above the synthetic core and, consequently, filtrates pass through it because of the pressure



Fig. 3. Glass beads.



Fig. 4. Synthetic core in the PVC core holder.



Fig. 5. PVC cell at the test position.



Fig. 6. Dynamic Circulation of Filtration Set up.

differential exerted by the static tank. The drilling fluid returns back to the mud tank after exiting from the PVC cell.

The experiment was planned to simulate an over-balanced horizontal well condition with a 100 psi differential pressure. After the system was plugged in, the dosing pump was put at its maximum injection rate (60 l/h). Different mud samples were injected through the cell for the same time period and the amount of fluid loss versus time was measured. After each test, the cell was put into an oven for 1 day at 80 °C and then the hospital CT scanner was used to visualize the alteration in the pores due to the mud invasion.

3. Results and discussions

3.1. Analysis of the EUC powder/acid interaction

After the drying process in the oven, the residual powder was weighed and the solubility achieved by each experiment was measured. The final results are shown in Fig. 8. Removal of LCM particles after the drilling phase and before the production phase is critical. Hence the solubility of LCM in acids should be high. As shown in Fig. 7, the EUC solubility in HCl is at an acceptable range in high temperature conditions. The solubility would be higher at higher temperatures in reservoirs.

Solubility of EUC in HCl is higher than H_2SO_4 and this makes HCl a better option for acidizing. Also, the results showed that raising the temperature from 25 °C to 40 °C had a lot more influence on EUC solubility than raising the soaking/aging period from 1 day to 2 days.

In order to analyze the effect of acid on the shape and size of the particles, SEM images were taken before and after the dissolution experiments. Fig. 8 shows the SEM image of the EUC particles before the dissolution experiments. The particles are mostly fibrous and do not have any regular shape. The size range of the particles is wide and includes small fines as well as long particles up to 1400 μ m.

Figs. 9 and 10 show the SEM images after interaction with 28% H_2SO_4 and 28% HCl, at 40 °C. The particle sizes in the powder were reduced to a range of less than 150 μ m up to 700 μ m, after reacting with H_2SO_4 . After interaction with 28% HCl solution, the particles experienced considerable size change and the size range of the particles shrunk to less than 65 μ m up to 245 μ m (for the largest particles). The reduction in the shape and size of the fibrous particles, observed by SEM images, shows the lower chance of induced formation damage using EUC.

Therefore, HCl is more effective in dissolving EUC additive which leads to smaller particle size, better evacuation of the residual particles from the near wellbore area during the back flow and lower formation damage. By analyzing the above tests and field operational limitations, we recommend the application of 28% HCl and an aging time of 2–3 days for better dissolution process.

3.1.1. The EUC powder effects on mud rheology and pH

The effects of adding EUC to the drilling mud on the rheological behavior and pH is shown in Fig. 11 and Table 4. The addition of 1.5 lb/bbl caustic soda caused the base mud pH to rise considerably, reaching 12.69. EUC powder has a considerable effect on drilling pH and 30 lb/bbl EUC powder reduced the pH to 9.54, which is a common mud pH in field applications. Therefore, EUC powder noticeably affects mud pH. Thus, EUC powder can only be considered as a LCM agent when the original mud is highly alkaline and the reduction of pH neither changes the mud characteristics nor increases the risk of corrosion problems. Also, it is possible to add NaOH powder to the mud, along with the EUC powder, to compensate for the acidic nature of the EUC.



Fig. 7. EUC Solubility in HCl and H₂SO₄ at 15%, 28% and 37% acid concentrations, at 25 and 40 °C after 1 day and 2 days.



Fig. 8. SEM image of the EUC powder before any treatment.



Fig. 9. SEM image of the EUC powder after 2 days in 28% H₂SO₄.

Adding 30 lb/bbl EUC powder improves the rheological properties of the mud significantly. Higher values of rheological properties increase the pressure in the base of the hole, which affects the drilling operations and pump performance; this should be considered in the well planning process. According to the considerable effects on mud rheology, it is suggested that a suitable dispersant additive should be combined with EUC powder.



Fig. 10. SEM image of the EUC powder after 2 days in 28% HCl.



Fig. 11. Rheological behavior of the samples prepared in this experiment.

3.1.2. Fluid loss control capability of the EUC powder

As the pump started injecting, data gathering commenced using a video camera to record the fluid loss flow in order to calculate the fluid loss rate. The results are shown in Figs. 12 and 13. The loss rate and total loss volume decreased dramatically after application of the EUC powder as an additive to the base mud. The total volume loss of the base mud decreased from 8800 ml to 1140 ml (an 87% reduction) and 536 ml (a 94% reduction) for samples containing 10 lb/bbl and 30 lb/bbl EUC powder, respectively. Moreover, the initial loss rate of the mud decreased from 15.00 to 8.27 and 5.07 ml/s for the mud samples containing 10 lb/bbl and 30 lb/bbl EUC powder, respectively. The final loss rate of the base mud reduced from 0.557 ml/s to 0.068 ml/s and 0.015 ml/s for samples containing 10

M. Sedaghatzadeh, K. Shahbazi, P. Pourafshary et al.

Petroleum 7 (2021) 152-159

Table 4

The EUC powder effects on mud sample rheology and pH.

Specifications	Base Mud	Base Mud +10 lb/bbl EUC	Base Mud +30 lb/bbl EUC
Yield Point	13	16	23
Plastic viscosity	14	17	22
Gel Strength (10 s)	6.0	8.5	12.0
Gel Strength (10 min)	7.5	10.0	14.5
pH	12.69	11.44	9.54
Mud Weight (ppg)	9.55	9.60	9.70



Fig. 12. Fluid loss rate of each sample during the injection process.



Fig. 13. Total volume loss of each sample during the injection process.

lb/bbl and 30 lb/bbl EUC powder, respectively. During the injection of the base mud, no mud cake was formed due to the small particle size of the solid particles in the base mud compared to the pore sizes of the core. As a result, no considerable bridging occurred during the base mud injection process. On the other hand, when we used EUC, mud cake was formed. As can be seen in Fig. 11, the base mud loss did not reach a stable mode over the duration of the experiment but when we used EUC powder loss became stable after 9 and 13 min, for 10 and 30 lb/bbl EUC powder, respectively.

The addition of EUC powder decreased the fluid loss rate and total loss volume considerably, due to the noticeable size difference between the EUC particles and other particles in the base mud, which affected the base mud rheology. Our experiments showed the capability of EUC to control fluid loss in a slightly over-balanced drilling condition in a non-fractured system.

3.1.3. Effect of EUC on formation damage during drilling

Residual mud in porous media causes severe formation damage which is a critical point to be considered. In this work, we compared CT scan images of the porous medium before and after injection of the base mud containing 30 lb/bbl EUC powder in order to investigate the possible damage. A sample of these images is shown in Fig. 14. As shown in this figure, EUC powder did not invade the core



Fig. 14. CT scan images of the core cell (a) After base mud injection, (b) After base mud + 30 lb/bbl EUC injection.

deeply but blocked the core entrance. This blockage controls the fluid loss into the porous media. Based on Fig. 14-b, the invasion length when using 30 lb/bbl EUC in drilling fluid is shallower than the base mud injection. The glass beads in the porous media were not affected, which means that there is no deep damage or porosity alteration in the formation.

The development of cake by EUC improves fluid loss control and reduces formation damage which makes it an effective additive to drilling muds for application in fractured Iranian fields. Therefore, we recommend the application of EUC due its capabilities, low price and ease of preparation.

4. Conclusions

A new LCM additive was studied and analyzed in this paper. Different important parameters, such as fluid loss control properties, formation damage control, and solubility in acid were studied to confirm the capabilities of the new proposed material. The following conclusions were reached.

- (1) Acceptable acid solubility of EUC was observed. We recommend the use of 28% HCl solution due to the higher solubility of EUC and considering that higher acid concentrations are not difficult to achieve in on-site applications.
- (2) EUC powder had a great effect on the rheology and pH of the base mud, which must be considered in any well planning process before drilling operations. 30 lb/bbl EUC powder could reduce the base mud pH by approximately 3 units, thus, they are applicable in highly alkaline muds or when they are added into the mud with additional caustic soda to compensate for the acidic nature of the powder.
- (3) EUC powder improved the rheological properties of the base mud. Hence, the addition of EUC powder to the base mud

reduced the loss rate and the total loss volume considerably at a differential pressure of 100 psi. The percentage reduction in initial, final and total fluid loss volume by using EUC powder was found to be 66%, 97%, and 94%, respectively. This can be considered to be a satisfying result.

- (4) The blockage of the core by the EUC particles occurred in the entrance of the cell and so, consequently, only a small amount of damage would be imparted to the reservoir in an on-site application.
- (5) In general, the results of the experiments indicated that EUC powder has several impressive beneficial features, including the fact that it is environmentally friendly, has low cost, and has good fluid loss control capability.

Declaration of competing interests

The authors declare that they have no conflict of interests.

Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.petlm.2020.04.005.

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