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Estimation of Retained Crude Oil Associated with Crushed Salt and Salt Cores in the Presence of Near-Saturated Brine

Timothy J. O'Hern, Thomas E. Hinkebein, and Thomas W. Grasser

Prepared by
Sandia National Laboratories
Albuquerque, New Mexico 87185 and Livermore, California 94550

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ESTIMATION OF RETAINED CRUDE OIL ASSOCIATED WITH CRUSHED SALT AND SALT CORES IN THE PRESENCE OF NEAR-SATURATED BRINE

Timothy J. O'Hern
Energetic and Multiphase Processes Department

Thomas E. Hinkebein
Underground Storage Technology Department

Thomas W. Grasser
Energetic and Multiphase Processes Department

Sandia National Laboratories
P. O. Box 5800
Albuquerque, NM 87185-0706

ABSTRACT

This paper describes three experiments whose purpose is to determine the amount of retained oil on massive salt surfaces and in crushed salt in the presence of water and brine. These experiments have application to the decommissioning process for the Weeks Island mine. In the first experiment, oil-coated salt cores were immersed in either fresh water or in 85% brine. In the case of both fluids, the oil was completely removed from the cores within several hours. In the second experiment, oil-coated salt pieces were suspended in air and the oil was allowed to drain. The weight of retained oil clinging to the salt was determined. This experiment was used to estimate the total amount of oil clinging to the roofs of the mine. The total amount of oil clinging to the roofs of the mine is estimated to be between 240 and 400 m^3 (1500 and 2500 BBL). In the third experiment, a pan of oil-soaked crushed salt was immersed in 85% brine, and oil removal from the salt was monitored as a function of time. At the start of the experiment, prior to immersion, 16% of the bulk volume of the crushed salt was determined to be interstitial oil. After the pan of crushed salt was immersed in 85% brine, 80% of the oil, which had been in the interstitial spaces of the crushed salt, immediately floated to the surface of the brine. This oil was not bound and was immediately released. During the next 380 hours, oil continued to separate from the salt and the rate of transfer was governed by a mass-transfer rate limitation. The final estimate of the fraction of permanently bound oil retained in the crushed salt is 10% ±5% of the initial interstitial oil.

CONTENTS

FIGURES

1.0 INTRODUCTION

During the planning period for the decommissioning of the Weeks Island Mine, it was necessary to provide a rough estimate of the amount of oil which would be available for skimming and which would be retained in the mine. At that time, the primary concern was schedule development and equipment definition. Because of time constraints and restricted access to the Weeks Island mine, crushed salt and salt cores from the mine were not available for this study. Hence, surrogate salt materials were selected to evaluate the mechanisms of interaction between Weeks Island oil and salt. In the selection of these materials, careful attention was given to the important representative properties of the substitute materials. In particular, the minimum particle size of the crushed salt was selected to approximately match the minimum particle size reported for salt piles in the mine. Also, the solid salt core samples used in these studies had fractured faces that qualitatively matched the wall and roof surfaces of those in a drill and blast mine.

The specific areas of interest in these experiments are the remaining oil trapped in the crushed salt layer on the mine floor, and the oil clinging to the walls and ceiling of the mine after oil withdrawal and brine flooding. For this reason three different types of experiments were conceived to model the oil withdrawal and brine flooding processes. In the first set of experiments various salt cores were soaked in crude oil and then were immersed in either fresh water or in 85% brine. The oil removal process was observed visually using the shadowgraph technique.

In the second set of experiments, various blocks of broken salt core were soaked in crude oil, then suspended above a collection pan in both horizontal or vertical orientations, and their weight and the weight of the oil dripping from them were monitored over a period of several days.

In the third experiment, a crushed salt material simulates the residual crushed salt layer on the floor of the mine. The object of this experiment was to determine how quickly oil rises from the oil-saturated crushed salt on the mine floor and how much oil remains trapped in the salt floor layer upon prolonged exposure to 85% saturated brine (density of 1.17 g/mL). There was no imposed flow of brine over the crushed salt layer, and the experiment was allowed to run for 380 hours.

These experiments were performed and interpreted to allow a determination of important phenomena and to provide a qualitative estimation of the amount of oil which is likely to end up floating on oil (hence potentially available for skimming) and the amount of oil which is permanently trapped in the mine.

Other investigators (Bear, 1975) have considered the permanent entrapment of oil in porous media. Naturally occurring particles will retain oil by capillary forces. Also, small amounts of oil become trapped in the sediments and do not move. It is only when the critical oil saturation is exceeded that any oil movement will occur. This is the primary limiting phenomenon in the production of oil from oil sands. Because of this effect, recovery of oil by water flooding techniques seldom reduces oil to less than 20% of the total pore volume.

2.0 COATED SALT CORE EXPERIMENTS

2.1 Coated salt core experimental setup and procedure

The experiments involved preparing segments of solid salt core, soaking them in Weeks Island mine crude oil for at least 24 hours, submerging them in a tank containing either fresh water or approximately 85% saturated brine (density approximately 1.17 g/mL), then observing the resulting flow and oil removal. Salt cores are 102 mm (4 inches) in diameter and 102 to 127 mm (4 to 5 inches) long. Salt cores were Big Hill 103 bottom hole cores that were available from the Sandia core library. These cores were very clean, having approximately 3% non-salt solids, typical of both Big Hill and Weeks Island. Figure 1 shows an oil-soaked salt core ready to be submerged in the water tank. The soaked cores appear to be fairly uniformly coated with oil prior to immersion. Figure 2 shows the salt core immersed in the fresh water tank. The tank cross-sectional dimensions are 570 mm by 575 mm, and it was filled to a depth of 254 mm, for a liquid volume of 83.2 L. The core is placed on a 102 mm (4 inch) diameter Lucite base to provide better positioning for observation. During the experiment, oil droplets are released from the surface of the salt core, and rise to the water's surface, while saturated brine dissolving from the salt core settles to the bottom of the tank.

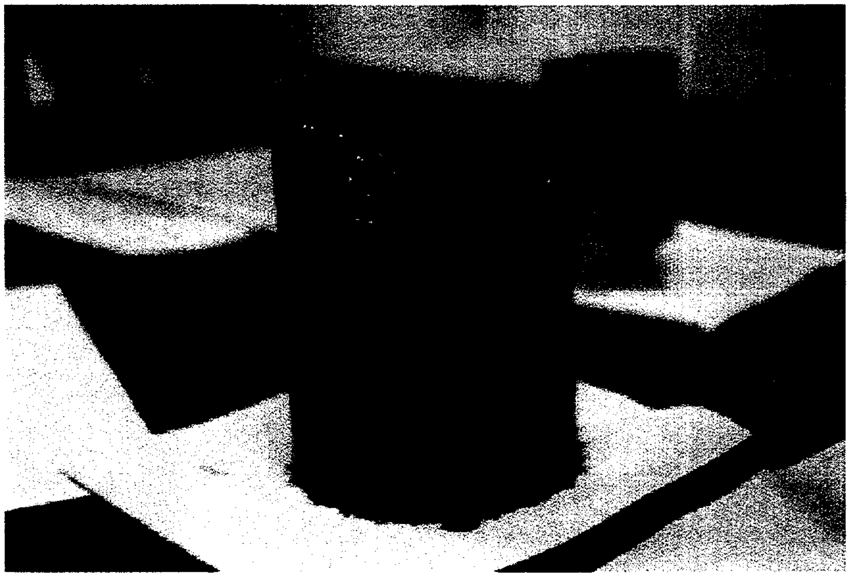

Figure 1. Oil-coated salt core prior to immersion. Each core was submerged in Weeks Island crude oil for 24 to 72 hours.

Figure 2. Oil-coated salt core during fresh water immersion experiment. View is down through free surface to salt core.

The primary observation technique applied was shadowgraphy, which records variations in density through its effect on the refractive index n (specifically, the shadowgraph makes use of the second spatial derivative of n, e.g., $\partial^2 n/\partial x^2$). Figure 3 shows the shadowgraph system set up for observation in the tank. Figure 4 is a still photo demonstrating the type of shadowgraph seen in the videos. The saturated brine boundary layer flows down the wall of the core (especially evident in the fresh water case).

Figure 3. Setup for salt-core/crude oil shadowgraphy.

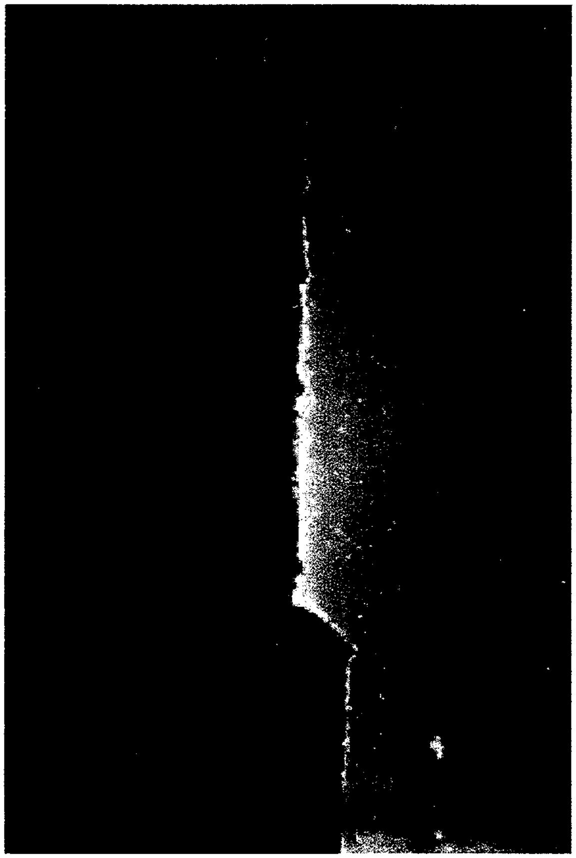

Figure 4. Shadowgraph of salt core oil release in fresh water.

Two basic experiments were performed. These involved soaking a salt core (102 mm diameter, roughly 100 mm long) in crude oil for at least 24 hours, then:

1. Immersing in fresh tap water (density of 1 g/mL) and using shadowgraphs and visual observations to monitor how long it takes for oil to leave the salt walls, or
2. Repeating in approximately 85% saturated brine (density of 1.174 g/mL). The brine solution was prepared by dissolving a known mass of Diamond Crystal "Solar Salt" in a measured volume of tap water. The mixture was stirred until the salt was completely dissolved, then a sample was withdrawn to check the brine density by weighing a known volume.

The salt cores were then left submerged until they reached approximately 50% of their original volume, and this time was noted.

2.3 Coated salt core results and discussion

Regardless of the immersion fluid, the oil is eventually removed from the core walls, although the time scale is roughly 6 times longer in the brine case than in the fresh water case (approximately 6 hrs vs. 1 hr, respectively). Note that there is no imposed flow of water or brine over the salt-soaked cores; all flow is due only to natural convection of the higher density saturated salt boundary layer flowing down the core walls. When exposed to fresh water, the sides of the oil-soaked salt cores are free of oil in about 20 minutes, and the top is clean in about 1 hr. The sides appear to be scrubbed clean by a turbulent, downward oriented, saturated brine boundary layer, as shown in Figure 4.

For 85% saturated brine, both the sides and top are oil-free in about 6 hours. The fact that the sides and top act essentially the same in the brine case seems to indicate a dissolution mechanism, as opposed to the hydrodynamic scrubbing mechanism in the fresh water case. Supporting this idea are the shadowgraphs, which show a very thin, slow boundary layer for cores immersed in brine, unlike the strong turbulent boundary layer flowing down the core walls when immersed in fresh water. In brine, some of the surface oil droplets extend through the thin boundary layer.

As expected, the salt dissolution rate was greatly reduced when the cores were exposed to 85% saturated brine as opposed to fresh water. In fresh water, the originally 102 mm (4 inch) diameter core had lost about 50% of its volume after 4 hours (final diameter 70 mm (2.75 in)). In 85% saturated brine, the originally 102 mm (4 inch) diameter core lost only about 6% of its volume after 8 hours (diameter 98.4 mm (3.875 in)), and only after 48 hours did it lose 50% of its volume (final diameter 70 mm (2.75 in)).

Time-lapse videos of the surface oil, while not providing a good view of the saturated brine boundary layer, are very useful for visualizing the salt removal process. The salt cores soaked in crude oil for 24 to 72 hours are uniformly coated. After a few minutes of immersion, the oil film on the core surface "congeals" to a distribution of droplets, such that the core appears "cleaner" even before oil droplets start rising to the water surface. The oil droplets along the sides of the salt cores are then released over the remaining time (approximately 20 minutes for fresh water, and 6 hours for brine). Figure 5 is a photo taken in the middle of the brine test. After tests in both fresh water and 85% saturated brine, the post-test cores always contain some small amount of oil that looks like a stain on the core. This is caused by oil permeating into core fractures. Because the oil soaking process was not done under pressure, the amount of oil penetration in the mine may be greater than what was observed here.

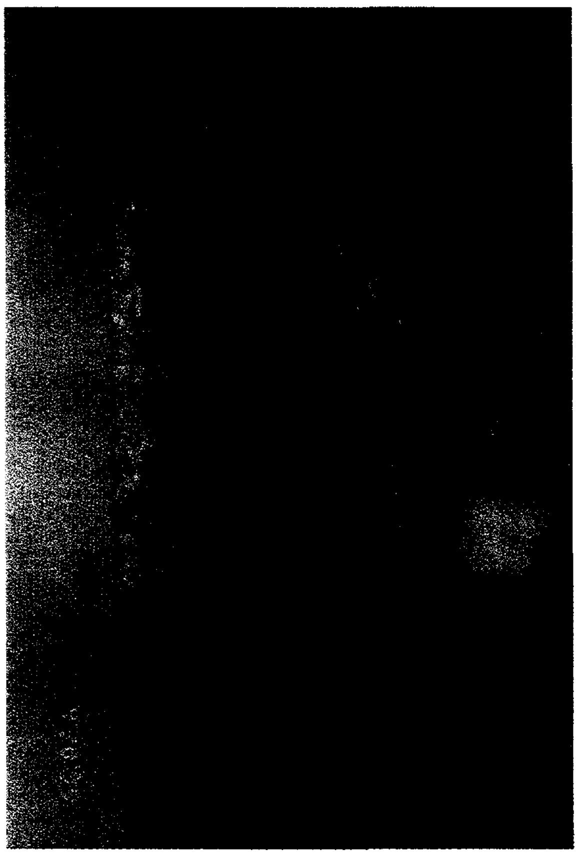

Figure 5. Salt-core oil release in 85% saturated brine.

In addition to visual observation of the oil release process, both videos and still photos were taken to provide a data record. The video sequences have been saved on S-VHS tapes, and an 8 minute summary video prepared. In the summary video, highlights of the shadowgraph tests are shown, demonstrating the key points discussed above. The final 2 minutes of the summary video is a "time-lapse" showing the surface oil on the core submerged in 85% saturated brine, and how it evolves over 7.5 hours.

2.4 Conclusions from results of coated salt core experiments

Although oil removal from salt cores occurs more readily when exposed to fresh water, the process still works even with 85% saturated brine. The time scale for oil removal is much longer in the 85% saturated brine case, on the order of 6 hours as compared to one hour or less in fresh water. In addition, shadowgraphs indicate that the primary oil removal mechanism changes from a hydrodynamic scrubbing in the fresh water case to salt dissolution in the brine case. However, both of these time periods are very short relative to the brine flooding times, and it is expected that complete oil removal from the walls of the mine will occur.

3.0 SALT DRAINAGE EXPERIMENTS

3.1 Draining salt (walls and ceiling) experimental setup and procedure

For the wall and ceiling experiments, Weeks Island mine salt cores were sectioned, leaving flat exposed faces. The oil-wetted face area was carefully measured. These faces were then soaked in crude oil for several hours. The oil-coated samples were suspended above an oil collection pan in either horizontal or vertical orientations to simulate walls and ceilings, respectively. The entire assembly was enclosed in an oil-vapor saturated environment to minimize evaporation, and the weight of collected oil was monitored over a period of several days. The weights of the cores, and of the oil dripping from them, were monitored over a period of several days to determine oil remaining on the salt. At the conclusion of the experiment the oil wet core was reweighed to determine the amount of oil clinging to the salt. All equipment was placed under a plastic tent to minimize evaporation, and the reported results were adjusted to account for oil evaporation.

3.2 Draining salt (walls and ceiling) results

The results of these experiments are summarized in the following table.

Sample Number	Orientation	Retained Oil (g)	Projected Area (m^2)	Weight/Area (g/m^2)
1	Horizontal	4.12	$8.11*10^{-3}$	508
2	Horizontal	3.60	$1.12*10^{-2}$	322
3	Vertical	1.88	$1.23*10^{-2}$	153
4	Vertical	3.39	$1.16*10^{-2}$	292

Oil drainage from salt cores was observed to occur over a six hour time period. After six hours no additional oil was found to drip from the salt. Further, these experiments indicate that only a small amount of oil remains after this relatively short time period. Additionally, it was observed that the salt orientation did not have any appreciable effect on the results. The biggest factor in the difference between one sample and the next was the amount of penetration into cracks in the salt. The sample with the highest amount of oil clinging to the salt had a significant amount of oil that had penetrated far into the cracks of the salt core.

3.3 Discussion of results for salt drainage experiments

The oil drainage experiments are used to estimate the total amount of oil clinging to the roofs of the mine. In this case the experimental range of values for oil clinging to the roof of the mine is between $153 g/m^2$ and $508 g/m^2$. The roof area estimates for the upper and lower levels of the mine are 227,000 and 473,000 m^2 (2,439,000 and 5,086,000 ft^2.) Hence, the total amount of oil clinging to the roof of the lower level is estimated to be between 160 and 270 m^3 (1000 and 1700 BBL). The total amount of oil clinging to the roof of the upper level is estimated to be between 80 and 130 m^3 (500 and 800 BBL). This gives an estimate of the total amount of clinging surface oil as 240 and 400 m^3 (1500 and 2500 BBL).

4.0 CRUSHED SALT EXPERIMENTS

4.1 Crushed salt experimental setup and procedure

A crushed salt experiment simulates the crushed salt on the floor of mine. The object of this experiment was to determine how quickly oil rises from the oil-saturated crushed salt on the mine floor and how much oil remains trapped in the crushed salt upon prolonged exposure to approximately 85% saturated brine.

A pan of oil-saturated crushed salt was prepared by filling a Plexiglas dish to a depth of 25.4 mm with rock salt (Diamond Crystal "Solar Salt," Akzonobel Corporation). The pan had horizontal dimensions of 275 mm by 273 mm. This salt has an average grain size of approximately 4 mm with a minimum size of 1.8 mm and a maximum size of 8 mm. Based on measurements reported by Acres (1977), the crushed salt in the Weeks Island Mine had a particle size range of 2 mm to 20 mm. The salt filled a volume of 1905 mL. The salt was then soaked in one liter of Weeks Island mine crude oil for approximately 24 hours, then 700 mL of residual free oil was poured off, leaving an oil-saturated crushed salt layer. Hence, the oil filling the salt porosity was 300 mL or 16% of the total salt volume. The brine tank was the same large tank used in the salt core experiments. It was filled with 85% saturated brine, again prepared by dissolving a known mass of Diamond Crystal "Solar Salt" in a known volume of tap water. A sample was then withdrawn for density measurement, and was found to have a density of 1.18 g/mL (86% saturated). The oil-saturated salt pan was then submerged in the brine tank. The resulting oil removal was observed and measured (Figure 6). Visual and video observations were made of the oil removal, and the oil was periodically collected from the free surface using hydrophobic oil collecting pads (Fisher Scientific polypropylene sorbent pads). The pads had each been weighed prior to oil collection, and were again weighed after collection to determine the mass of oil removed per unit time. This mass was converted to a volume by using a crude oil density of 0.8835 g/mL. There was no imposed flow of brine over the crushed salt layer, and the experiment was allowed to run for 380 hours. After 380 hours the experiment was terminated and the residual salt/oil mixture was placed in a large beaker and fresh water was added to the mixture. The last of the salt was dissolved to determine the oil remaining in the salt.

The effects of oil evaporation were not recognized until after the conclusion of this experiment. The oil pads were weighed several days after they were collected. During this time water on the pad evaporated as well as did some of the oil. The total oil mass discrepancy totaled 40% of the oil added to the pan. Because evaporation was not felt to be significant while oil was under the brine surface, the evaporated oil was added back to the total and accounted for as oil removed from the salt during the early part of the experiment.

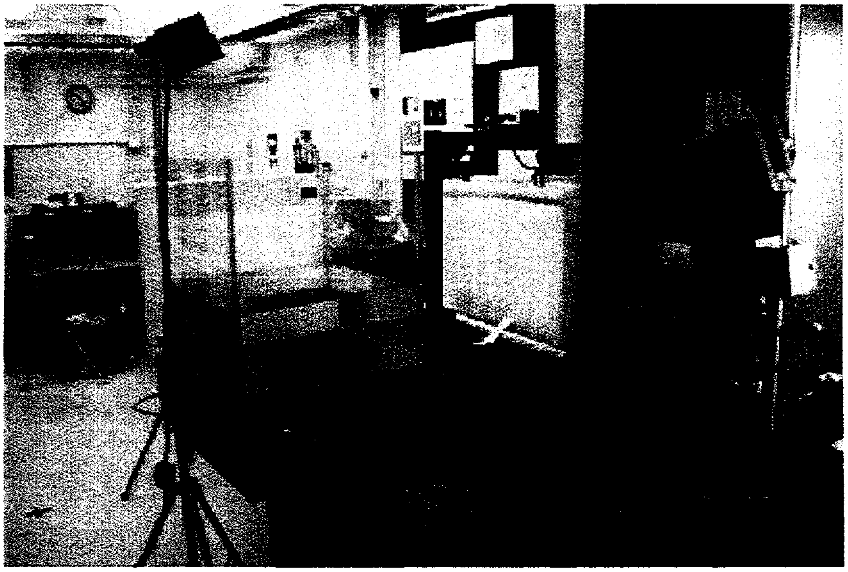

Figure 6. Setup for crushed salt/crude oil shadowgraphy.

4.2 Crushed salt results

As oil was removed from the crushed salt, it rose to the surface of the brine tank where it was collected and weighed. The raw data are plotted in Figure 7. From this plot it is observed that 200 g of the 265 g present after draining the oil was removed in the first few seconds. After this initial period, the rate of oil removal slowed dramatically. After 380 hours, when the experiment was terminated, an additional 34.6 g of oil was released from the remaining salt. The mass of oil removed is plotted on a fractional basis in Figure 8.

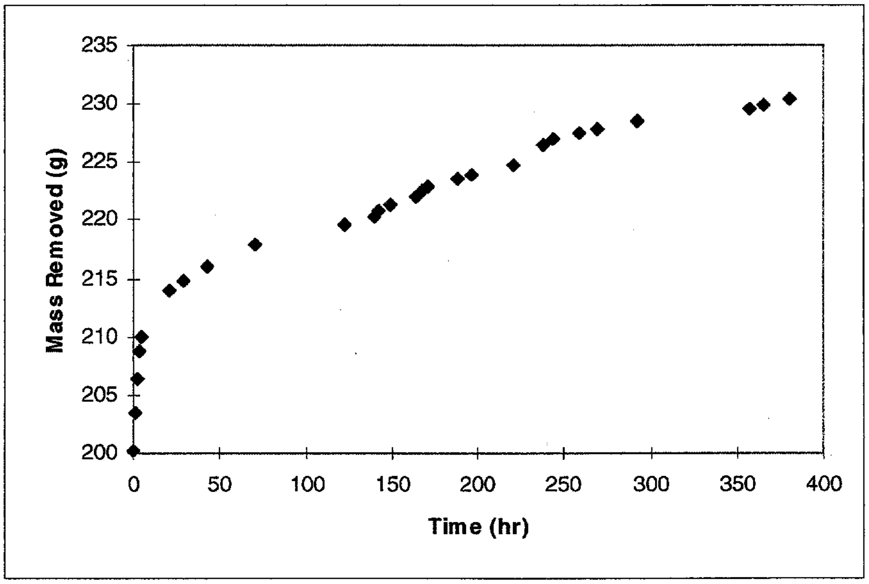

Figure 7. Plot of mass of oil removed from crushed salt as a function of time in the crushed salt experiment.

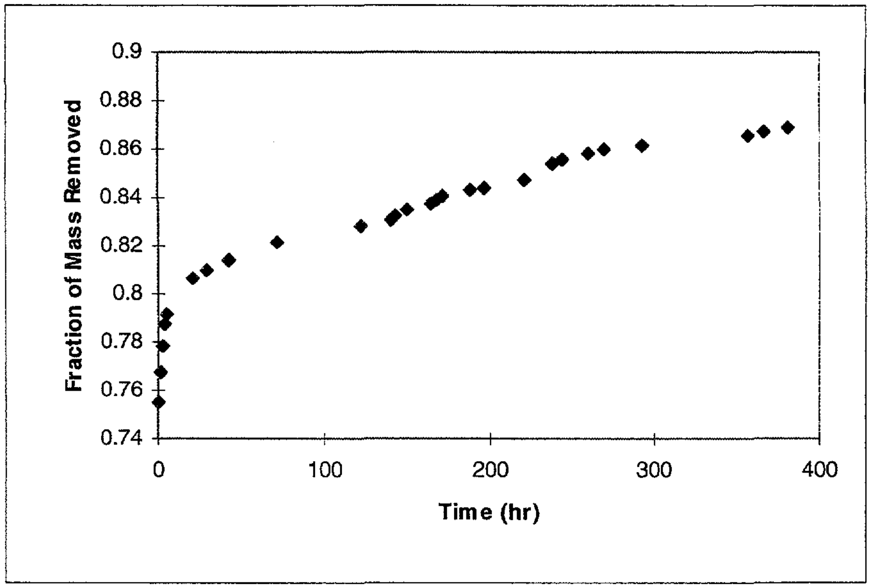

Figure 8. Plot of fractional mass remaining in crushed salt as a function of time.

4.3 Analysis of results for crushed salt experiments

When the crushed salt is flooded with brine, there are two main oil removal processes that occur. First, upon initial contact with brine, non-bound oil immediately floats to the surface. This oil occupies interstitial space in the crushed salt, but capillary forces do not bind it. Consequently, nothing retards the movement of this initial oil outflow. Subsequently, capillary forces within the salt matrix retard the further removal of oil from the crushed salt. The mechanism for this continued loss of oil must be determined from the data.

In order to understand the phenomena of oil removal, the rate of oil removal was plotted in Figure 9. From this figure, the oil removal rate is a linearly decreasing function of time on the log-log plot for times up to 120 hours. This linear behavior is typical of a mass-transfer rate-limiting process.

In order to understand the mass transfer limitation, consider the following mathematical description of mass transfer limited processes.

The slow transfer of oil from crushed salt to the brine is given by

$$R = hA(f - f_c), \qquad (1)$$

where R is the mass-transfer rate of oil from the crushed salt to the brine, f is the fraction of total oil which remains in the salt, f_c is the permanently trapped oil fraction held by capillary forces, h is the mass transfer coefficient, and A is the nominal area of the crushed salt surface.

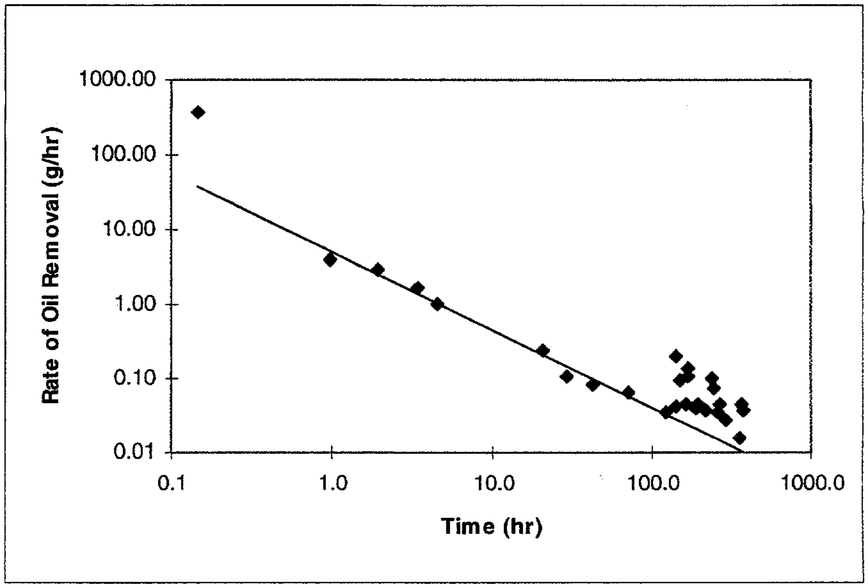

Figure 9. Plot of the rate of oil removal from crushed salt as a function of time. Up until 120 hours, the rate of oil removal showed a linear decrease in this log-log plot. This removal phenomenon is indicative of a mass transfer limited process.

The rate of oil loss from the crushed salt may also be expressed as

$$R = -V\frac{df}{dt}, \qquad (2)$$

where V is the total volume of oil in the crushed salt before flooding with brine, and t is time. Equation (1) and (2) may be combined to give

$$\ln\left(\frac{f - f_c}{f_o - f_c}\right) = -\frac{hA}{V}t, \qquad (3)$$

where f_o is the initial oil fraction after the non-bound oil escapes. This functional dependence is plotted as the solid line in Figure 10. From Figure 8 the non-bound oil represents approximately 80% of the total oil in the salt so that f_o is approximately 0.2. Different values of f_c may be tried for this plot, but the best linear data fit occurs for a permanently trapped oil fraction of 0.1. In the post data-collection time period after 400 hours, very little extra oil was observed on the brine surface to indicate that the ultimate steady state was nearly achieved.

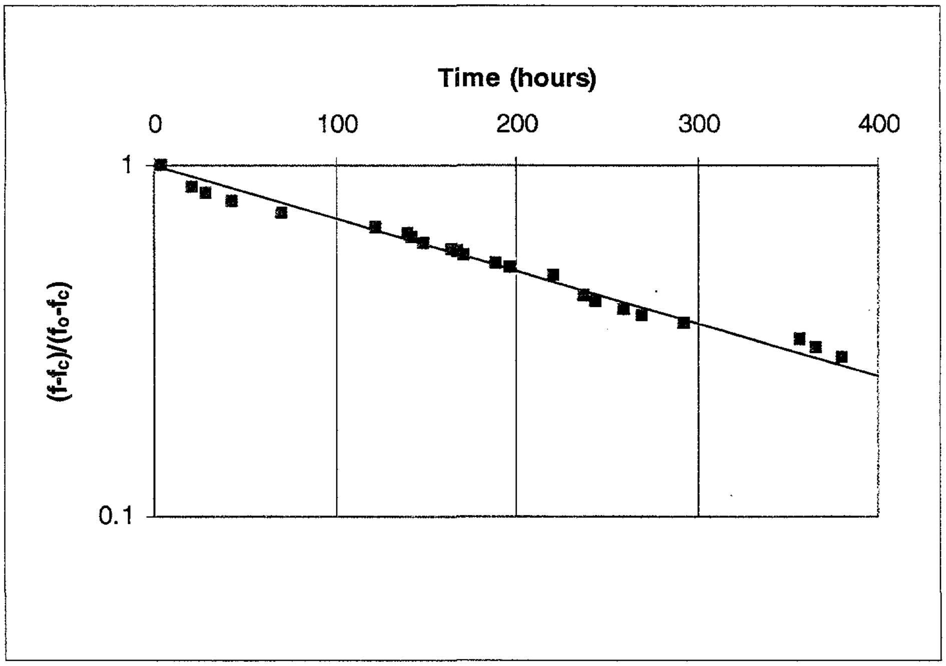

Figure 10. Plot of the oil loss data using the mass transfer model. The individual measured points are shown along with the predicted line from the mass transfer model. In this plot the value of f_0 was taken to be 0.2 while the value of 0.1 was used for f_c.

Constructing the same plot for values of f_c either greater or less than 0.1 shows curvature as shown in Figure 11. Because a linear response is expected by Equation (3), these figures show that the expected permanently trapped oil fraction is approximately 0.1. However, the data do not allow a precise determination of this retained fraction in that all of the curves are very close to linear.

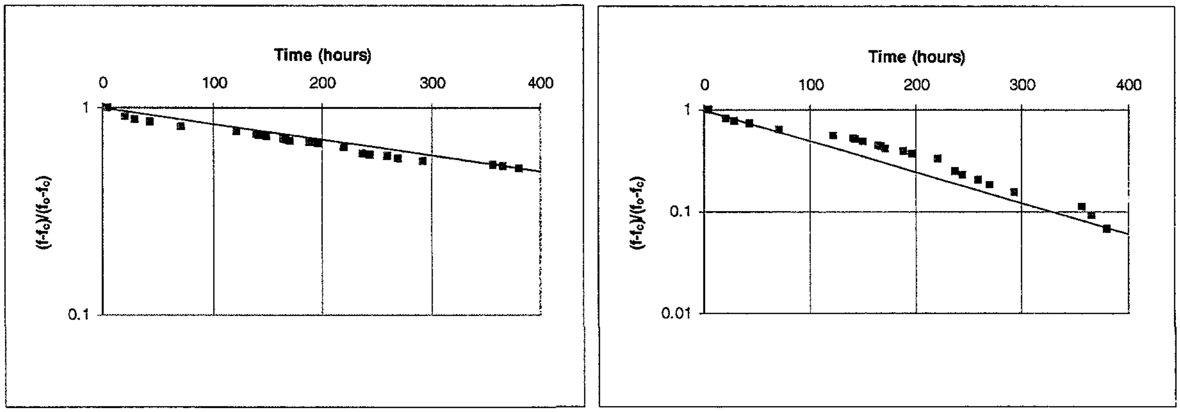

Figure 11. Plots of the fractional available oil loss for a permanently trapped oil fraction, f_c, of 0.05 for the plot on the left and 0.13 for the plot on the right. Deviations of the data points from the linearity required by the mass transfer model indicate a mismatch of the permanently trapped oil fraction.

Applying the best fit of the mass transfer model to the oil removal data shows that the model fit is adequate for prediction purposes.

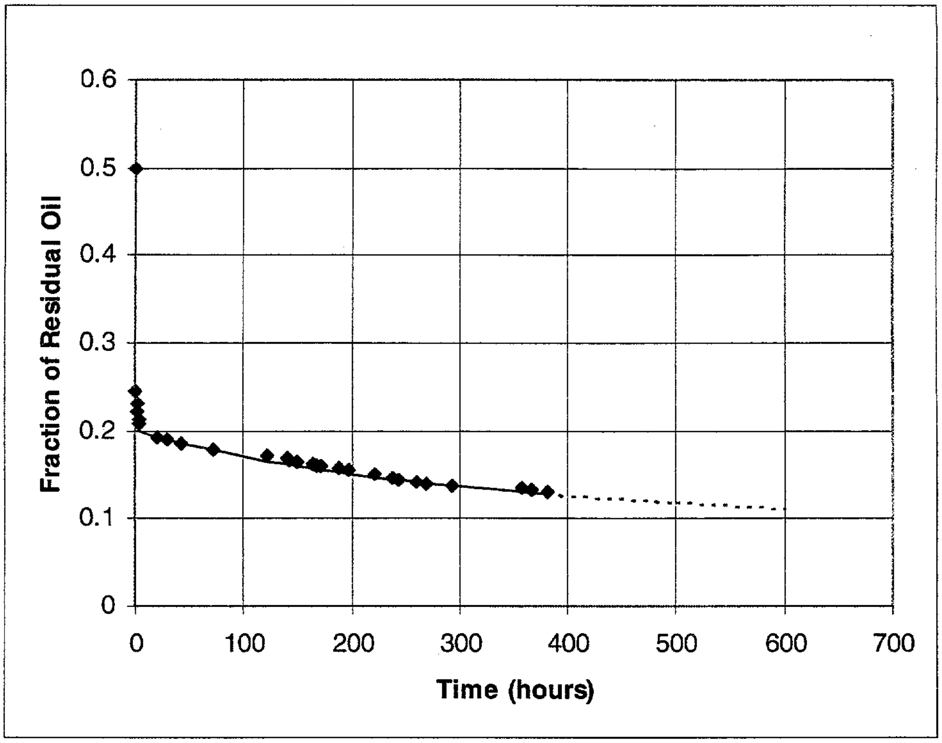

Figure 12. Plot of the fraction of measured retained oil present in crushed salt compared to mass transfer model calculations. For this model, the value of f_o was 0.2 and the permanently bound oil fraction, f_c, was 0.1.

5.0 SUMMARY OF IMPLICATIONS OF THIS STUDY FOR PROJECTIONS OF RETAINED OIL IN CRUSHED SALT AND ON MINE ROOFS

The submerged salt core experiments were performed in the first phase of testing. In these experiments, oil soaked salt cores were immersed in both fresh water and in 85% brine. Visual observation of the hydrodynamic action revealed that oil rapidly separated from the salt by the action of boundary layer flow. In the case of fresh water the boundary layer was turbulent and the oil removal was very rapid. For 85% brine the boundary layer flow was laminar and the oil removal process took approximately 6 times as long. In both cases, this oil removal process was accomplished over the course of several hours. Because this time period is short compared to the brine refill time of the mine, it is concluded that oil removal from the walls of the mine will be instantaneous relative to the brine fill. After tests in both fresh water and 85% brine, the post-test cores always contain some small amount of oil that looks like a stain on the core. This is caused by oil permeating into core fractures. Because the experimental oil soaking process was not done under pressure, the amount of oil penetration in the mine may be greater than what was observed here.

The oil drainage experiments evaluated the amount of oil that clings to salt walls and roofs. In these experiments salt pieces were suspended in air and the oil clinging to the salt was weighed. These experiments were used to estimate the total amount of oil clinging to the roofs of the mine.

In this case the experimental range of values for oil clinging to the roof of the mine is between $153 g/m^2$ and $508 g/m^2$. The roof area estimates for the upper and lower levels of the mine are 227,000 and 473,000 m^2 (2,439,000 and 5,086,000 ft^2). Hence, the total amount of oil clinging to the roof of the lower level is estimated to be between 160 and 270 m^3 (1000 and 1700 BBL). The total amount of oil clinging to the roof of the upper level is estimated to be between 80 and 130 m^3 (500 and 800 BBL). These amounts are very small and their effect on the oil balance is trivial. Based on the results of the first set of experiments, the amount of oil clinging to the drift walls and roofs subsequent to brine flooding will be even less than the small amounts given above.

The crushed salt experiments were used to measure the separation of oil from crushed salt submerged in 85% brine. In the crushed salt experiments, a pan of crushed salt was immersed in 85% brine, and oil removal from the salt was monitored as a function of time. Crushed salt was initially flooded with Weeks Island oil and then allowed to drain. At the start of the experiment, prior to flooding, 16% of the bulk volume of the crushed salt contained oil. The salt used in this study has a particle size distribution which is arguably smaller than that observed in the salt piles by the Acres(1977) report. However, the minimum particle size of the crushed salt used in the test is approximately equivalent to the salt present in the mine. Because the quantity of retained oil is determined more by the minimum particle size than the average particle size, it is felt that the results of this study are meaningful.

After the pan of crushed salt was submerged in 85% brine, 80% of the oil, which resided in the interstitial spaces of the crushed salt, floated to the surface of the brine. This oil was not bound and was immediately released. During the next 380 hours, oil continued to separate from the salt and rate of transfer was governed by a mass transfer rate limitation. At the conclusion of the test 13% of the oil was still present in the salt as was determined by dissolving all of the crushed salt. Data plots using a mass transfer model suggest that the permanently bound oil fraction is 10% of the total interstitial oil. However, for this experiment, the uncertainty of this analysis suggests that the permanently bound oil fraction is between approximately 5% and 15% of the interstitial oil.

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