Abstract

Prior to the drilling of the depleted Kisimul sand in the Barra well, we were asked to investigate:

- The use of micronized cellulose fibers to prevent mud loss to the Kisimul sand;
- Vendors’ claims of high return permeabilities when these fibers are used as lost circulation material;
- The depth of invasion of micronized cellulose fibers used as a lost circulation material in a synthetic-based mud, particularly as related to the anticipated 5-inch perforations in the Kisimul sand; and
- The ability to dissolve filtercake with the vendor-suggested alkaline hypochlorite (33% sodium hypochlorite and 15% KOH) in the Kisimul sand.

Our investigation indicates that:

- The fine cellulose fibers suggested by the OU lessen fluid loss due to seepage into a permeable zone, but do not provide protection against mud loss in even a 0.02-inch wide fracture, though a mixture of the fine and coarse products would do so;
- The presence of fine micronized cellulose fibers in synthetic-based mud does not adversely affect permeability (up to 2 D);
- The depth of invasion of these fibers is only a few pore volumes, at most; and
- The hypochlorite treatment is not a viable option for clean-up of the fibers in a synthetic-based mud environment.

Experimental Work

A 13.5 lb/gal SBM weighted up from the field value of 12.5 lb/gal with barite was obtained from the mud company; this weight is in the range expected to be used for drilling the Kisimul sand. We used 8 lb/bbl LCM; this means that the LCM is only 2%, on a weight basis, of the total weight of the solids (8 lb/bbl out of a total of 8 lb/bbl + 310 lb/bbl of barite, the latter number obtained from the recipe supplied by the service company). Thus, on a purely statistical basis, the probability of significant invasion by the LCM is very low.

The micronized cellulose LCM product recommended for testing was Highland Glen Fine, which, according to measurement results provided by the manufacturer has the following particle size distribution:

<table>
<thead>
<tr>
<th>Particle Size (microns)</th>
<th>Highland Glen Fine</th>
<th>Barite</th>
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<tbody>
<tr>
<td>d10</td>
<td>6.7</td>
<td>1.6</td>
</tr>
<tr>
<td>d50</td>
<td>35</td>
<td>15</td>
</tr>
<tr>
<td>d90</td>
<td>126</td>
<td>50</td>
</tr>
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</table>
(i.e., 10% of the Highland Glen Fine particles are smaller than 6.7 microns, while 10% of the Barite particles are smaller than 1.6 microns)

In contrast, the barite used by the mud company has a considerably larger fraction of fine material; thus, more barite would be expected to enter the formation than would be the LCM material.

Ceramic disks of nominal pore sizes 5 and 10 microns, obtained from the manufacturer, were used for the permeability tests. According to a representative of the distributor of the disks, the 5- and 10-micron disks could be expected to have permeabilities around 500 mD and 2 D, respectively; these values are in the range expected for the Kisimul sand. Prior to use, the disks were saturated with seawater, in accord with standard practice.

Microscopic examination of the 5-micron disks showed that they were uniform in composition and color (white). In contrast, the 10-micron disks were macroscopically uniformly gray but with islands of white crystals, small black particles, and patches of yellowish color of unknown origin scattered randomly throughout the disk; in addition, these disks had the appearance of an oil impregnation that extended through about half their thickness.

Filtercakes were prepared using standard HTHP (high temperature, high pressure) cells, the disks, and a differential pressure of 500 psi over 30 minutes at 150°F. Return permeabilities in the production direction were measured using standard procedures, with 5 psi differential pressure.

Solubility of the micronized cellulose in HCl was stated by the manufacturer as 35%. We weighed 0.5 g of Highland Glen Fine in 10 ml of acid. Our tests were carried out by heating the LCM/acid at 176°F for 3½ hours in a 5-gallon oil bath and leaving the samples to cool off in the bath overnight. The samples were vacuum filtered through a Whatman 42 filter.

Results and Discussion

Fracture Sealing. Highland Glen Fine and Highland Glen Coarse were added individually to synthetic-based muds at about 40 lb/bbl and placed above a 3/8-in. thick steel disk into which had been cut a slit 0.02 in. wide and about 2 in. long. The sample containing the fine LCM did not plug the “fracture” at all, while the sample containing the coarse LCM plugged the “fracture” within about 30 seconds and did not allow more than a few tenths of a cc of additional fluid loss over the remainder of the 30-minute test.

We infer from these results that the SBM with only Highland Glen Fine will not seal even a 0.02-in. wide fracture and so will allow the loss of whole mud if such a fracture occurs. In contrast, we infer that the use of the Highland Glen Coarse material will substantially prevent whole-mud loss.

Best Practices on LCM by service companies recommend the use of an equal blend of fine, medium, and coarse materials, since the addition of too many fine particles may affect the drilling fluid rheology. In addition, larger particles may be more effective at sealing larger fractures or formation openings.

Permeability Changes. Measured values of the production permeabilities, relative to the initial values, were 105% for the 5-micron disk after cake deposition and 77% for the 10-micron disk. The equivalence of the initial and final values for the 5-micron disk indicates that the cake simply lifted off the disk and suggests that there was no formation damage. The slightly reduced production permeability of the 10-micron disk after deposition of the cake indicates that the cake did not entirely lift off and suggests the possibility of some formation damage; the source of any damage could not be identified with certainty from this experiment.

Invasion. The following approaches were used to establish whether any of the cellulose passed through the disk and whether any lodged in the disk:

- The presence of particulate matter in the effluent from the HTHP test would indicate that either barite or cellulose had passed through the disk and, therefore, that we might anticipate invasion of at least ¼ in. in the Kisimul sand. To test whether any particulate matter was in the effluent, we made use of the Tyndall effect, in which a very bright light is placed at right angles to the angle of observation; floating particulate matter would be detectable as bright spots in the liquid. No bright spots were observed in the effluent from either disk. We have attempted to capture this with a photograph; unfortunately, since placing the bright light normal to the observation angle yielded a very bright spot in the middle of the picture, we chose to illuminate the fluids from four different angles. Figure 1 shows the result.
• A direct test of the invasion of the cellulose into the disks (into the formation) involved breaking the disks, adding a few drops of concentrated sulfuric acid to the broken surfaces, and observing the result through a microscope. (Concentrated acid is known to dehydrate sugars, leaving residual carbon. The effect on solid sugars is similar to the action of “snakes” used to celebrate July 4th.) A control test involved adding sulfuric acid to a sample of cellulose; the fluid immediately turned dark brown, and the residual solid turned black. Figure 2 shows the acid-treated cellulose. Figures 3 and 4 show a blank 10-micron disk (Figure 3) and a 10-micron disk on which a cake had been deposited (no invasion at all, as evidenced by the absence of any dark color except at the surface, Figure 4). Figures 5 and 6 show a detailed view of the two disks. No difference is observed between the two 10-micron disks.

• Thin sections of the two 10-micron disks were analyzed microscopically, with a sample of Highland Glen Fine for comparison. No difference was detected between the two disks, and no LCM was detected in the disks.

We conclude from these experiments that no significant invasion of either the 5-micron or the 10-micron disk took place.

The above interpretation is in accord with expectations based on the sizes of the LCM particles. Less than 2% of the LCM has particle size less than \( \frac{1}{3} \) the pore diameter in the 5-micron disk; for the 10-micron disk, the value is 5%

**Clean-Up of Micronized Cellulose Fibers.**
The choice of a procedure was complicated considerably by the manufacturer’s statement that some of the fibers were oil-wet and some were water-wet. In order for decomposition to take place, the (water-soluble) agent causing the decomposition had to get to the surface of the fibers. This might or might not be straightforward for the water-wetted portion, but would be extremely difficult for the oil-wetted portion.

The manufacturer of the fibers indicated a maximum solubility in HCl of only 35%. Our results show a somewhat higher solubility:

<table>
<thead>
<tr>
<th>Wt% Acid</th>
<th>Wt% Solubility of Highland Glen Fine</th>
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<tbody>
<tr>
<td>0</td>
<td>23</td>
</tr>
<tr>
<td>5</td>
<td>47</td>
</tr>
<tr>
<td>10</td>
<td>47</td>
</tr>
<tr>
<td>20</td>
<td>57</td>
</tr>
</tbody>
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Filtration of the cellulose in de-ionized water took much longer (4 hours), while the acid-treated samples filtered in about 15 minutes. It is likely that the cellulose in water gels and passes through the filter during the extended filtration. The residual filtercakes after the filtration show coarsely textured fibers on the filter paper for the acid-treated sample, while the particles on the untreated cake appear finer. Apparently the acid has dissolved the smaller fraction of the LCM.

In a filtercake made from a synthetic-based mud, the expectation would be that more than a small fraction of the fiber would be oil-wetted, making it inaccessible to acid. In any case, acid will have no effect whatsoever on the barite present as the major solid component of the filter cake.

In work on other projects, we have found that filtercakes made from laboratory synthetic-based muds can be broken most readily by mixtures of mutual solvents (solvents which dissolve in both hydrocarbons and water), very specific surfactants, and concentrated acid (to remove the acid-soluble weighting agent). There is no chemical reason for the mud containing the LCM to behave any differently from the mud without the LCM; the LCM would act largely as an inert ingredient since its solubility is around 35%.

As part of another project, we have conducted tests of a variety of nonionic and ionic surfactants in water for breakup of filtercakes. None of the tests has so far been unequivocally successful.

The manufacturer suggested use of a cocktail of 33% NaOCl (sodium hypochlorite) and 15% KOH, which had been shown in lab tests of only the Highland Glen Fine in water to solubilize up to 97% of the cellulose fiber material. This approach, too, was rejected. Olefins are well known to be susceptible to oxidation, far more so than are polysaccharides. Thus, the chance of a runaway reaction was too great to allow even a laboratory test of this approach. In the field, the well would have to be cleaned thoroughly of ANY deposits of whole mud before a reaction such as this was even attempted.

Our conclusion is that neither acid solubility nor treatment with hypochlorite is a viable method for removal of the LCM in a filtercake made from a synthetic-based mud. Further, such a cake has been found to be very stable under treatment with surfactants in water, the standard procedure for cleaning a hole prior to cementing. In our experience, cake breakup can be accomplished only under the action of a swiftly moving fluid consisting of an expensive mutual solvent, preferably containing a surfactant specific to the particular synthetic-based mud, and acid. As noted above, even if the majority of the LCM were made water-
wet, the probability is that at most only 35% of it would dissolve.

**Size Changes Upon Exposure to water.** We investigated the behavior of this material in water, the concern being that the LCM might expand over time and so block the formation. The experiment involved placing a thin dusting of Highland Glen Fine on a microscope slide, adding a few drops of water, and observing particles in both the water-covered and the dry zones for a couple of hours. We also allowed the LCM to sit in water for a month; the results were the same as for the 2-hour test. In Figure 7, the very bright area on the top right is the dry zone of the slide, and the brown area is the water-covered zone.

Three points are particularly important.

1. The LCM is made up of some fibrous material but also contains a substantial amount of solid, non-fibrous material.

2. The fibers in the water-covered zone are actually about the same size and are present in about the same concentration as those in the dry zone. Unfortunately, the photograph does not show this very well (nor was this shown well in any of the other eleven photographs we took of this slide). We did notice that the fibers did not appear to grow or lose shape over the course of the experiment. Compare the lengths and widths of the few fibers visible in the water-covered zone with those in the dry zone.

3. The solid, non-fibrous particles did not lose their shape during the two hours (or a month) of exposure to water; instead, they remained well defined. In addition, they appear to be about the same size as those in the dry zone. Thus, we do not believe that these particles expanded as the result of their exposure to water.

We do not see any evidence that the Highland Glen Fine material expands significantly upon exposure to water.

**Conclusions**

Our investigation indicates that:

- The depth of invasion of these fibers is only a few pore volumes, at most; and
- The hypochlorite treatment is not a viable option for clean-up of the fibers in a synthetic-based mud environment.

**Acknowledgements**

The authors thank Chris Davis and Jerry Adams for help with the experimental work and the management of SEPTAR for permission to publish.
Fig. 1 – Photograph of the effluents from cake formation on the 5-micron and 10-micron disks, illuminated on four sides by very bright point sources of light. Note the absence of bright spots in the fluid, indicating the absence of particulate matter.
Fig. 2 – Photograph of the test LCM, Highland Glen Fine, after addition of a few drops of concentrated sulfuric acid. Note the dark color of the fluid and the black residue.

Fig. 3 – Overview photograph of the 10-micron control disk (before filtercake was deposited) and addition of a few drops of concentrated sulfuric acid to the exposed broken surface. Note the absence of any dark color other than at the surface. The black deposit on the surface of the control disk (upper left) resulted from contact of the acid with some facial tissue that was adhering to the surface.
Fig. 4 – Photograph of 10-micron disk on which a filtercake had been deposited from SBM, after addition of a few drops of concentrated sulfuric acid to the exposed broken surface. This result shows the absence of significant invasion of cellulose. Note the similarity in appearance of the two surfaces, especially the lack of any unusual concentration of black spots in the photograph on the right.

Fig. 5 – Detail of the 10-micron control disk.
Fig. 6 – Detail of the 10-micron disk after filtercake buildup.
Fig. 7 - Photomicrograph of Highland Glen Fine after two hours of soaking in water. The water is bounded by the brown line, with water being on the left. Note the similarity in particle shape and size in the two zones.