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New Liquid Microsphere System Simplifies Lightweight and Ultra-Lightweight Cementing

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Abstract

As reservoir targets are increasingly chosen below difficult drilling formations, the need for lightweight cement of higher quality is also increasing. While conventional water-extended cement slurries are often favored because of their low cost, slurry properties or high minimum densities may prevent them from being used in the more challenging wellbores. To obtain slurries of premium quality and lower density, using foam or microspheres is recommended. While foam has certain advantageous mechanical properties, it is not applicable in all well conditions. Because of these reasons, the popularity of microsphere cementing has continued to increase every year over the last decade.

In spite of the growing market, several complications can exist in regard to actually using microspheres to cement oil and gas wells. Microspheres should be homogeneously distributed throughout the bulk material while it is being delivered to the mixing head, otherwise viscosity or density-control issues may arise. Industrystandard cement mixing equipment is designed to mix based on density, but the real concern when mixing a cement slurry (although not monitored) is delivering the slurry at the correct cement:water ratio. During conventional cementing, a heavy bulk material is mixed with a light fluid. When this heavy bulk material is mixed with a light liquid, physics assures that mixing at the correct density also means the slurry is being mixed at the proper cement:water ratio. With the addition of lightweight microspheres to the bulk material, the overall specific gravity of the bulk material is reduced. In some cases where the microspheres are added at high concentrations, the bulk material can actually become as light or even lighter than the mix fluid. As the concentration of the microspheres increases (or the density of the bulk decreases), it becomes hard to mix the resulting slurry at the wrong density. On the surface one might think, not being able to mix at the wrong density a good thing, but it is actually problematic. Remember, with density-based mixing equipment, we are relying on density to control the cement:water ratio. If all cement:water ratios yield a similar density, controlling the ratio by controlling density is no longer possible. Volumetric-based mixing systems that function well when the bulk density—mix fluid density is small have been introduced to the industry, but are not in widespread use.

Some amount of bulk cement is lost every time it is transferred from one pressure vessel to the next. When expensive microspheres are included in those losses, the cost of the job rises. Also, increased fines introduced by the microspheres could cause dusting concerns.

The liquid microsphere system discussed in this paper can help eliminate or minimize the foregoing issues. By minimizing or eliminating the standard microsphere cementing issues, lightweight cement can become easier to deliver successfully. Ultra-lightweight cementing also can be accomplished at most any time and place without requiring (1) specially designed or modified cementing equipment or (2) application of job volumes small enough to batch-mix.

Background

General

When mixed with the recommended concentration of water, cement density ranges from 14.8 to 16.4 lb/gal depending upon the specific surface area or grind of the dry cement powder. When the goal of the cement job is to circulate cement above zones with a low fracture gradient, the cement density is reduced to help prevent losses of bulk cement fluid into the formation. Industry technology allows density reduction through three different mechanisms: (1) addition of extra water, (2) foaming the liquefied cement slurry, and (3) bulk blending microspheres into the dry cement powder.¹

Water-Extended Slurries

The proper amount of water required to slurry dry cement powder is determined by the grind or specific surface area of the dry cement powder plus or minus what is required to slurry any additives that are also being blended with the cement to modify the downhole properties. Most additives will require additional water, but certain additives that disperse the solids can actually reduce the required amount of water needed to generate a normal-viscosity slurry. Water added solely to reduce the slurry density may cause slurry-stability problems. To allow the addition of this extra water while maintaining

slurry stability, more additives are blended in conjunction with the increased water. Examples of these waterextending additives are: sodium and potassium silicates, fumed silica, bentonite, pozzolan, and diatomaceous earth. These materials are often referred to as lightweight additives, but in reality they are waterextending additives. In these slurries it is actually the water that is the lightweight material. Commonly these water-extending additives are inexpensive. When combined with the large increases in slurry yield created by the additional water, this class of cement slurries is typically the most economical solution, especially if viewed only in the short run. This cost reduction is the primary reason why the water-extending choice is selected. While inexpensive, these slurries commonly provide the lowest level of performance of all cementing systems. The second factor that limits the usefulness of these water-extended designs is the lower limit for achievable density reduction of 11 or 11.5 lb/gal. Below this density range, compressive strength development ranges from unacceptably slow to nonexistent.

Foam

Creating a stable foam of the cement slurry is an excellent method of reducing the density of a liquid cement slurry. Nitrogen is normally used as the gaseous phase in foam cementing. Occasionally compressed air is used, but is limited because delivery methods are less reliable. Gas-generating solids have been used in the industry, but the competing chemical reaction with the cementing hydration reaction makes nitrogen the preferred foaming choice. In addition to the primary function of reducing downhole slurry density, foaming the cement slurry has three primary benefits for the well: (1) increased slurry compressibility, (2) increased set cement elasticity,² and (3) capability to change downhole density based on last-minute changes in well conditions. Increased slurry compressibility is beneficial to operators when annular gas-channeling potential exists or in deep water where shallow water flow is a concern³⁻⁵ Increased elasticity of the set cement is desirable in wellbore cementing because it helps the cement maintain its seal in the presence of outside stresses. 6,7 In spite of the above engineering benefits, decreasing slurry density by foaming is often not selected because it requires additional personnel, equipment, and, normally, access to nitrogen.

Microspheres

The third choice for density reduction in wellbore cementing fluids is through the introduction of low specific-gravity microspheres into the cementing slurry. Common industry microspheres typically fall into one of three types: (1) solid plastic beads ~ 1 sg (surface and downhole), (2) hollow pozzolanic spheres 0.7 sg (surface), and 3) hollow, engineered glass bubbles 0.32—0.61 sg (surface and downhole). Lightweight slurries

built with microspheres can create the highest strength:weight ratio and lowest permeability of any lightweight cement design. Rapid compressive strength development associated with high-strength cement is beneficial in wellbore cementing because waiting on cement (WOC) times can be reduced.

The Problem

While microsphere cementing is often selected as being the high-tech or premium lightweight cementing solution, it too can have drawbacks: (1) blending, (2) mixing, and (3) cost. In all blended cements, homogeneity is required. In microsphere blends it is even more important to achieve the highest standards of homogeneity. If the microspheres are not evenly distributed, achieving uniform density and/or slurry stability may be difficult. Provided the correct amounts of all materials are present in the blend and some of blend has an excess of microspheres, less water (than the recommended design concentration) will be used in achieving the design density. If the design was based on an ideal amount of water to make the slurry both thin enough to mix easily and thick enough to ensure slurry stability, this reduced water, required to achieve the correct slurry density, may create a slurry that is too thick to mix on location. Again if all of the material was correctly loaded and a portion of the blend has extra microspheres, another portion of the blend will be short. To mix at the correct density more water would then be required in this scenario. This additional water can cause slurry instability. If mixed with the ideal amount of water for the reduced microsphere concentration, the slurry can be delivered to the wellbore at an excessive density. Densities higher than planned may result in exacerbated lost-circulation problems. To provide required levels of homogeneity, special blending, transport, and delivery guidelines have been established. While following these guidelines has yielded high levels of success, following them requires extra effort. Problems often arise when requesting extra effort from personnel not directly related to the delivery of the cement slurry to the wellhead.

Examples of situations where implementing new best practices might be met with resistance could be marine vessel operators or drilling-rig marine departments that govern offshore rig bulk management. Additionally following these best practice guidelines is very time consuming. Last summer, 1,350 sacks of 9 lb/gal blend were blended in 16 batches over a 5-day period. These same 1,350 sacks could have easily been blended in just a few batches in one afternoon using standard blending procedures if microspheres were not included.

Besides blending issues, the physics of microsphere slurries can present mixing difficulties. Mixing difficulties normally infer problems getting the slurry up to weight. With microsphere slurries, especially those with high concentrations of microspheres, mixing at the correct density is not a problem. As a matter of fact it is very

hard to mix at the wrong density providing you are using the standard industry variance of plus or minus 0.2 to 0.3 lb/gal as meaning constant density. An example of this can be seen in Table 1. In Table 1, four similar slurries have been designed at densities of 9, 10, 11, and 13 lb/gal. In all four cases the required change in mixing water concentration was determined to yield a change in density of plus or minus 0.3 lb/gal, the standard deviation of industry mixing equipment. From this data two observations are readily apparent.

- If one considers staying within a 0.3 lb/gal range of the design density, mixing was done at the correct density, it is hard to mix these slurries at the wrong density.
- 2. As the design density decreases it becomes increasingly difficult to mix these designs at the wrong density. The real problem with mixing these microsphere slurries is mixing them at the correct cement to water ratio. A slurry mixed with an extra 26% water will probably be unstable, one with an extra 150% water could be extreme. Likewise a slurry missing one-fifth of its water will be too thick.

Standard industry practice for preparation of cement slurry involves taking an unmeasured volume of bulk material and adding water until the resulting slurry has the correct density. With conventional systems, using heavy bulk blends, this method can provide a reliable method of mixing the dry bulk material at the correct cement to water ratio. In Table 2, a neat cement mixed at 15.6-lb/gal density and an 18-lb/gal cement mixed with 35% silica and 47-lb/sk hematite were processed with the same variance calculations. These results illustrate that the industry standard method for preparing cements is valid. In Table 3, the three lightweight slurries from Table 1 are revisited. This time the density variation was determined that corresponded with a change in cement to water ratio of 10%. Changes of water ratio in excess of 10% are normally sufficient to yield changes in the laboratory-determined set cement properties. Here we see that density variations from 1.3 to 0.3% are all that is required to push the ratio out of spec and that again the problem intensifies as slurry density decreases. To reiterate, the problem with mixing lightweight microsphere cements is not a matter of being capable of mixing them at the correct density (it is difficult not to mix them at the correct density), but rather supplying a slurry mixed at the ideal cement:water ratio. The point here is not that microsphere slurries cannot be used safely in the field; rather that unconventional mixing equipment may be required as the density decreases.

The final issue with microsphere slurries is cost. In standard bulk operations losses occur every time bulk materials are being transferred from one pressure vessel to the next. A common range for transfers is four to six; this range assumes that every move, including the one to the mixing unit is counted. If the microspheres are

pre-blended with the bulk cement, some of the expensive microspheres will be lost with every move. Since the microspheres are the lowest specific gravity material in the blend they will be lost at the highest rate. If the microspheres are not bulk blended, losses will still occur, but we will only be losing the relatively inexpensive portion of the blend. Because the microspheres are blended in with the cement if all or a portion of the blend is not used the microspheres cannot be reused later unless the exact same blend is requested. If excess material is required for backup, these microspheres may also be wasted.

Current Solutions

Two solutions have been used equally effectively for wellbore cementing.

- 1. In the first solution a batch mixer is partially filled with a known amount of water. An entire batch of bulk material, blended to precisely match the amount of water previously added to the batch tank, is added to the batch mixer and mixed until a homogeneous slurry is obtained. With this method both density and ratio are assured to be correct, providing the dry materials were added to the bulk transport correctly and the water volume was measured correctly. This first solution is the recommended method for delivering a quality ultralightweight microsphere cement slurry to the wellhead provided the job volume is small enough to place the slurry mixing in a batch process. This method requires specialized batch-mixing equipment to be available in the well location and space on location for the additional equipment.
- 2. A second method has proved successful in delivering ultra-lightweight microsphere cement slurries at the correct density and water ratio. The key advantage of this second method is it is a continuous process, thus it works equally well on large-volume jobs and on the smaller ones that could be batch-mixed. In this method the standard cement mixing equipment is modified to mix cement volumetrically. On land, where these modifications can be made to a single unit moved from well to well as needed, this solution works well. Offshore where a single rig has extended plans for microsphere cements, modification to the cementing unit is again cost-effective solution. While conventional equipment can be converted to volumetric mixing for single-slurry applications, the time and money required may prove this solution impractical as well.

New Solution

In an attempt to mitigate (1) the volume and equipment availability issues associated with batch mixing, (2) the equipment modification expense, availability, and time issues tied to volumetric mixing, (3) the cost of lost microspheres associated with bulk

blending, (4) the cost of unused excess, (5) complications related to modified best practices for bulk handling of microspheres, 6) the inability to change the density at the last minute should wellbore conditions change (cannot mix at the wrong density), (7) long lead times required to effectively blend (using all of the new best practices) large volumes of microsphere laden bulk blends, and (8) most importantly the issues that arise from mixing slurry at a less than ideal cement:water ratio a new technique for introduction of microspheres into the cement slurry has been developed. With this new technology a small portion of the mixing water is used to prepare an aqueous slurry containing the required volume of microspheres creating a liquid-bubble additive (LBA). The microspheres can be brought to location in bulk and the LBA prepared on-site or they can be slurried at the yard and brought to location as tanks of LBA. If premium microspheres are used that are both homogenous and not containing extraneous material or broken spheres, the LBA can be made to be very stable. Since hollow microspheres are lighter than water, settling will not occur. Testing has shown that any material that floats to the surface of the LBA can be easily re-suspended with small inputs of mixing energy. Since keeping the material from forming a difficult to resuspend sludge on the bottom of the container is not an issue, additives not required by the wellbore conditions are not required to create this LBA.

phase During the development of LBA. considerations were made for how LBA would affect slurry design and testing. Review of the technique was performed to help assure that field execution would not be too labor-intensive. Quality assurance procedures were put in place to help ensure that lab testing followed certain criteria already established as a best practice for application of beaded slurries. To avoid a repeat of previously stated equipment issues two different techniques were developed for field usage based on existing equipment that could be easily transported to and from location without requiring modifications to the standard mixing equipment.

If location mixing is selected for the LBA, a blender or similar device can be used. If extra microsphere material is requested for backup, it can be left separate until needed, so it can be returned and not be wasted if not required. By waiting until shortly before job time to mix the LBA, the final job volumes can be used, minimizing wasted material. If other liquid additives are required for the job, they can be added to the LBA, provided lab compatibility and viscosity testing was conducted ahead of time. Additional base water may be required if one of the liquid additives acts like a viscosifier to the LBA. It may even be possible to add additives not related to the LBA that are required as part of the cement slurry design, again providing the appropriate stability testing had been performed. This will help minimize either the number liquid additives requiring injection or simplify the

bulk-blending operation. Fluid-loss additives along with cement friction reducers, retarders, or accelerators are examples of additives that one may want to add to the LBA instead of bulking or injecting separately. Remember these additives can cause viscosification or dispersion and compatibility issues so proper water ratio must be confirmed ahead of time. The addition of the dry microspheres, to the small portion of the slurry's mix water, can be done through bulk transfer on land locations or cutting bags over a shear hopper.

Minimal agitation and/or recirculation will be required to initially disperse and may be required to homogenize the LBA immediately before use on the job. Any settling that may occur during static periods will not result in the same problems as those created when normal liquid additives allow solid materials to settle on the bottom. Microspheres in the LBA will not settle on the bottom because they float. Settled material may be problematic and also mean that an improper additive concentration is being delivered. Anyone who has tried to re-suspend material that has caked or clumped at the bottom of any vessel or container knows that the results are often less than satisfactory. If the LBA is premixed and brought to location in tanks as long as other additives are not included, excess can be brought to location and then returned to base for credit as the material is not mixed at a job related concentration.

Once the LBA is prepared, it can be used by injecting it (1) into the mix water line or (2) into the suction of the downhole pump.

- 1. In the first method the LBA is treated similar to other standard liquid additives. With the total required mixing water and the amount of water used to prepare the LBA known, the required injection rate or ratio can be calculated. Existing computerized liquid additive injection technology may be used with this implementation. If the required slurry density is low, the volume of microspheres will be large. In this case, injection rates may be substantially higher than is common for normal liquid-additive injection. This required injection rate can be easily approximated ahead of time so sufficient injection capacity can be brought to location. When using this method, the requirement to use standard densitybased mixing equipment to mix light and ultra-light slurries still exists, although it is no longer an issue. Mixing lightweight cement is not the problem. Trying to mix a cement based on density when the specific gravity of the bulk and mix fluid are similar, is the problem. When the LBA is part of the mix fluid the specific gravity is now even lower and the specific gravity of the bulk material is back in its normal range, providing a substantial density difference between the bulk blend and the mix fluid.
- 2. In the second method, where the LBA is injected directly into the suction side of the downhole pump, only conventional cement is being mixed at standard

or near-standard slurry densities. Again, existing chemical-injection technology may be used with this technique. In foam cementing it has been standard practice for years to inject the foamer/surfactant/ stabilizer mix into the suction side of the downhole pump. If this material is introduced into the mix fluid, sufficient air could be entrained to jeopardize the effective operation of the downhole pumping equipment. Again it is a simple calculation to determine the proper LBA injection rate except this time it will be based on the required microsphere to cement ratio instead of the water ratio.

Unplanned changes in mud weight are common when drilling in new areas or fields with complex lithology. With LBA the slurry density can be changed at the last minute. From the previous discussion we now know that with standard microsphere cementing, especially at the lower slurry densities, we cannot change the density and still maintain good slurry properties. Changing the density at the last minute is not recommended unless the change is planned for ahead of time. If there is any concern that the actual required slurry density and the design slurry may be different, three slurries should be designed ahead of time. These three could be, the initial target slurry and two others, one heavier and one lighter. In some areas it is not uncommon for a well to start making gas while casing is being run. If enough gas comes in that the mud weight is raised, it may be a good idea to also increase the cement density. In other areas it is just as common for losses to occur while running pipe. In this case the mud weight may end up being reduced. If losses are serious enough, chances for cementing success will increase if the cement density can also be reduced. Unfortunately at this point in time the blended cement will already be on location so any change in slurry density would have to be accomplished by altering the cement to water ratio. If the ratio is changed enough to affect the slurry density, the set cement will not have the same properties as those listed in the lab report. If extra water is added to decrease the density, the onset of compressive strength development will be delayed and the ultimate compressive strength will be reduced. If less water is used to increase the slurry density, the thickening time will be decreased, increasing the risk that the slurry may not be displaced all of the way to the shoe track. With an LBA design, having the lightweight additive dispersed in just a small portion of the mix water means that the slurry density can be changed substantially with only small changes in the cement to water ratio. Unlike the results of a new slurry created just by mixing at the wrong cement:water ratio, these new slurries can be mixed at their correct water ratio providing on-site flexibility never before available on non-foamed cement iobs. In this next example we examine the situation where the job was loaded out based on a 10.5-lb/gal

slurry design. In this example we will consider four options. For the first two (with reduced cement density), while running pipe or during pre-job conditioning the well started suffering mud losses requiring the mud weight to be reduced. In the final two cases the well started making enough gas that the mud weight had to be increased. With proper preplanning and the new LBA material on location, one could effectively and safely cement for either of these options.

Table 4 contains the design results based on this contingency planning. If we used the microspheres as LBA, instead of bulk-blending them into the dry cement powder the remaining mix would result in a slurry that is much easier and guicker to blend and then, trivial to mix with standard equipment. The relative ease with which the base portion of the slurry can be mixed is shown with the new water requirement and density (5.5-8 gal/sk water and 13.4-14.7 lb/gal) before addition of the LBA. If the desired downhole slurry (including LBA) rate is 4 bbl/min, the liquid additive rates to yield our ideal mix at densities from 9.5- to 11.5-lb/gal slurries are listed in the final column. Keep in mind we are talking about bringing out to location a single, simple blend designed to generate a production-quality 10.5-lb/gal cement that has the flexibility to deliver, without extra effort, a 9.5-, 10-, 11-, or 11.5-lb/gal slurry at a recipe that would very closely approximate the slurry that would have been designed, had one of those densities been the requested density.

In the past, microspheres have been added to the entirety of mix water in slugging pits, but there are many shortcomings to this solution. The primary one revolves around the issue of ensuring proper microsphere injection rate. Since microspheres are lighter than water, there is a tendency for this material to float. Since the suction from these rig pits is in or near the tank bottom, the initial mix-water may contain less than the design microsphere concentration. If this happens, the final portion of the mix-water will contain excessive beads. In this case, the lead slurry is mixed heavy and the tail light, exactly the opposite from ideal. Since the beads can be prepared ahead of time at the bulk plant, which is the ideal site for this operation, bringing bulk microspheres to location that may or may not be equipped is no longer required. Using LBA technology helps eliminate both of these problems as well.

In the current procedure used to create the LBA mixture, the microsphere material is slurried with just enough fluid to create a pumpable solution, minimizing the opportunity for slurry segregation, helping to ensure ideal microsphere delivery rate. Not only does LBA methodology enable consistent delivery rate, but opens up the possible to ramp the microsphere concentration. With a variable microsphere delivery rate, a single blend is brought to location and a heavier, higher-strength slurry can be placed near the shoe.

To properly prepare LBA and deliver it to the rig site

ready to use requires stricter material quality control than for common microsphere cementing. When slurrying the microspheres, it is recommended that this process only be attempted with engineered microspheres that are completely uniform in size with less than 5% shards. Any agitator or circulating tank should work for bead preparation, storage, and/or delivery. After extended static periods, the microspheres will float and may even form what appears to be a hard crust. Upon resumption of agitation or circulation, the microspheres rapidly resuspend forming a homogeneous mix. Fig. 1 shows the very fluid nature of the material shortly after an 18-hour static period. 9

Conclusions

- The use of LBA can help eliminate the problems associated with blending cements containing microspheres.
- Using LBA helps eliminate the problems associated with mixing a slurry at the proper cement:water ratio when using density-based mixing equipment with high concentrations of microspheres.
- 3. LBA helps eliminate the problems associated with bringing specialty blends to offshore locations.
- Installation of specialized volumetric mixing equipment is not required.
- LBA can enable job volumes, in excess of what can be batch mixed, to be easily mixed at the correct cement:water ratio when using high concentrations of microspheres.
- 6. LBA can enable last-minute changes in slurry density to be delivered to the wellhead without sacrificing the correct cement:water ratio.
- Using LBA can save money by helping eliminate most of the losses associated with the bulk-transfer process for expensive bulk blends containing microspheres.
- Using LBA can save money by enabling reuse of excess microspheres brought to location not part of a pre-blended cement mix.
- 9. Microsphere-slurried mix-water can be effectively delivered to standard oilfield cementing equipment.
- Required lead-time could be reduced with LBA by eliminating the time-consuming, specialty-blending process required to delivery a homogenous blend containing microspheres.
- 11. Concentration inaccuracies associated with adding

- microspheres to the entirety of the mix fluid may be eliminated with LBA.
- 12. Microspheres can be easily slurried into a long-term, stable mix.

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Table 1—Effect of Minor Density Differences on Cement:Water Ratio with a Lightweight Blend.

Slurry No.	Density, lb/gal	Water, gal/sk	Yield, ft ³ /sk	% Change Density	% Change Water	
1a	12.7	7.2	1.68	-2.3	12	
1	13	6.4	1.58	•	-	
1b	13.3	5.7	1.48	2.3	11	
2a	10.7	8.7	2.16	-2.7	26	
2	11	6.9	1.91	•	-	
2b	11.3	5.5	1.72	2.7	-20	
3a	9.7	11.3	2.67	-3	47	
3	10	7.7	2.19	•	-	
3b	10.3	5.25	1.87	3	-32	
4a	8.7	29.3	5.29	-3.3	155	
4	9	11.5	2.91	•	-	
4b	9.3	4.7	2	3.3	-59	

Table 2—Effect of Minor Density Differences on Cement:Water Ratio with a Conventional Cement.

Slurry No.	Density, lb/gal	Water, gal/sk	Yield, ft ³ /sk	% Change Density	% Change Water	
1a	15.3	5.6	1. 23	-1.9	7.7	
1	15.6	5.2	1.18	=	-	
1b	15.9	4.9	1.13	1.9	-5.8	
2a	17.7	6.8	1.74	-1.6	6.2	
2	18	6.4	1.69	=	-	
2b	18.3	6	1.64	1.6	-6.2	

Table 3—Effect of Cement:Water Ratio on Density with a Lightweight Bulk Blend.

Slurry No.	Density, lb/gal	Density, lb/gal Water, gal/sk		% Change Density	% Change Water	
1a	10.88	7.59	2	-1.1	10	
1	11	6.9	1.91	=	-	
1b	11.14	6.21	1.82	1.3	-10	
2a	9.93	8.47	2.3	-0.7	10	
2	10	7.7	2.19	-	=	
2b	10.09	6.93	2.09	0.9	-10	
3a	8.97	12.65	3.06	-0.3	10	
3	9	11.5	2.91	=	=	
3b	9.04	10.35	2	0.4	-10	

Table 4—LBA Slurry Modifications.

Density, lb/gal	Water, gal/sk	Yield, ft³/sk	Mix-water, gal/sk	Base Slurry Density (W/O LBA), lb/gal	Slurry Rate, bbl/min	LBA, gal/min
9.5	10.1	2.63	7.93	13.4	4	71.57
10	8.2	2.25	6.34	14.2	4	71.64
10.5	7.3	2.02	5.75	14.6	4	66.41
11	6.8	1.85	5.52	14.7	4	60.15
11.5	6.5	1.71	5.46	14.8	4	52.91



Fig. 1—Surface of LBA mix under agitation.