

# ATTRITION EXPERIMENTS FOR THE BENEFICIATION OF UNCONSOLIDATED SAND SOURCES OF POTENTIAL HYDRAULIC FRACTURE SAND, NORTHEASTERN BRITISH COLUMBIA

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## ABSTRACT

*Sand for use as a hydraulic fracture proppant is critical in the development of shale gas. The cost of transporting proppants from distant suppliers has prompted the evaluation of local sand sources to meet or subsidize demand in northeastern British Columbia. Several sand deposits have been identified as potential proppant sources; however, in their natural state, these sand deposits do not meet industry standards. Several experiments are presented in this paper that focus on washing, sizing and attrition to improve the quality of a promising aeolian sand deposit. These beneficiation methods were selected because they are the least expensive processing steps for improving silica sand proppants.*

*Results suggest attrition will reduce the content of deleterious sand grains; however, success was limited and attrition alone is insufficient to elevate the SiO<sub>2</sub> content to industry standards. Other physical, physiochemical or chemical treatments will be required. The most substantial improvement in the silica content was achieved early in the experiments (i.e., in the first 5 minutes), after which results were negligible. A short period of attrition is a beneficial processing step, but prolonged attrition will be of little advantage. Prolonged attrition may, in fact, be detrimental to the quality of the proppant because grain breakage may result in an abundance of undesirable angular grains. Aggressive milling tended to produce the greatest increase in SiO<sub>2</sub> and greatest decrease in Al<sub>2</sub>O<sub>3</sub> and Fe<sub>2</sub>O<sub>3</sub>.*

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**Key Words:** frac sand, proppant, attrition, beneficiation, silica sand

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## INTRODUCTION

The demand for hydraulic fracture proppants ('frac sand') in northeastern British Columbia will increase as shale gas resources development in this area continues. The BC Ministry of Energy and Mines, with its partner, the Geological Survey of Canada (through the Geo-mapping for Energy and Minerals [GEM] Program), is currently investigating surface sand and bedrock deposits to attract industry evaluation of the commercial potential of northeastern British Columbia frac sand sources (Hickin et al., 2010; Huntley and Hickin, 2010; Huntley and Sidwell, 2010; Huntley and Hickin, 2011; Huntley et al., 2011).

Hickin et al. (2010) identified a number of unconsolidated and bedrock sand sources in northeastern British Columbia that may hold some potential for use as a frac sand source. They indicated, however, that without processing or

beneficiation, none of these deposits would meet industry standards in their natural state. To be used as a proppant, sand must meet very stringent requirements, which are outlined in several industry standards, such as API RP 56 (American Petroleum Institute, 1995a), API RP 60 (American Petroleum Institute, 1995b) and International Organization for Standards (ISO) report number ISO 13503-2 (International Organization for Standards, 2006). If sand sources from northeastern British Columbia are to be considered as viable exploration targets, it is necessary to evaluate the processing and cost required to upgrade these deposits to meet industry standards.

Northeastern British Columbia sand sources might be considered economic for use as a proppant if the cost of beneficiation is less than the cost of transporting the products from established sources. Most frac sand in North America is produced from the Middle to Late Ordovician St. Peter

sandstone of the northeastern United States (also known by the industrial names of ‘Ottawa’ or ‘White’ sand), the Cambrian Hickory Member or Riley Formation (‘Brown’ or ‘Brady’ sand) or more recently, the Middle Ordovician Black Island Member in Saskatchewan (Dumont, 2007; Zdunczyk, 2007). To supply the British Columbia market, the product must be moved substantial distances from the mine to the well site. In addition to the cost associated with transportation, current frac sand production may not be able to keep pace with growing North American markets. The increased demand on the current supply may result in escalating costs. Processing sources from northeastern British Columbia may partially alleviate this supply gap if raw sand can be satisfactorily processed and/or blended with traditional products.

Beneficiation is the process of separating ore from gangue. It is achieved through physical or chemical processing (Taxiarchaou et al., 1997; Dumont, 2007; Sundararajan et al., 2009). Silica sand requires the washing and separation of quartz sand grains from other mineral grains (e.g., feldspar and mica) and lithic fragments. The cost of beneficiation increases as processing becomes more elaborate or intensive. Simple washing, screening and attrition scrubbing are the least expensive methods. Chemical separation and treatment would add significant cost and

regulation to processing. This study explores rudimentary beneficiation to elevate silica content through washing, sieving and attrition (through light milling), thereby providing the industry some sense of the processing requirement. The experimental method and design described in this report is not intended to be a substitute or alternative to established industrial processing (e.g., attrition scrubbing, other physical and chemical treatment). The most comparable industrial-scale process to that presented here is attrition scrubbing, where the action of one high-speed slurry flow impacts on another. The equipment involved in attrition scrubbing generally consists of two opposed impellers mounted on a shaft in a cell (cf. Freeman et al., 1993). In the experiments presented here, attrition is achieved by lightly milling the sample slurry with small stainless steel balls.

## METHOD

Three experiments were conducted on splits from a homogenized sample from the Fontas Eolian Sand Dune Field located along Dazo Creek, a tributary of the Fontas River (Hickin et al., 2010; Figure 1). A Fontas dune sample was selected because Hickin et al. (2010) reported this deposit as having relatively high potential as a frac sand source.

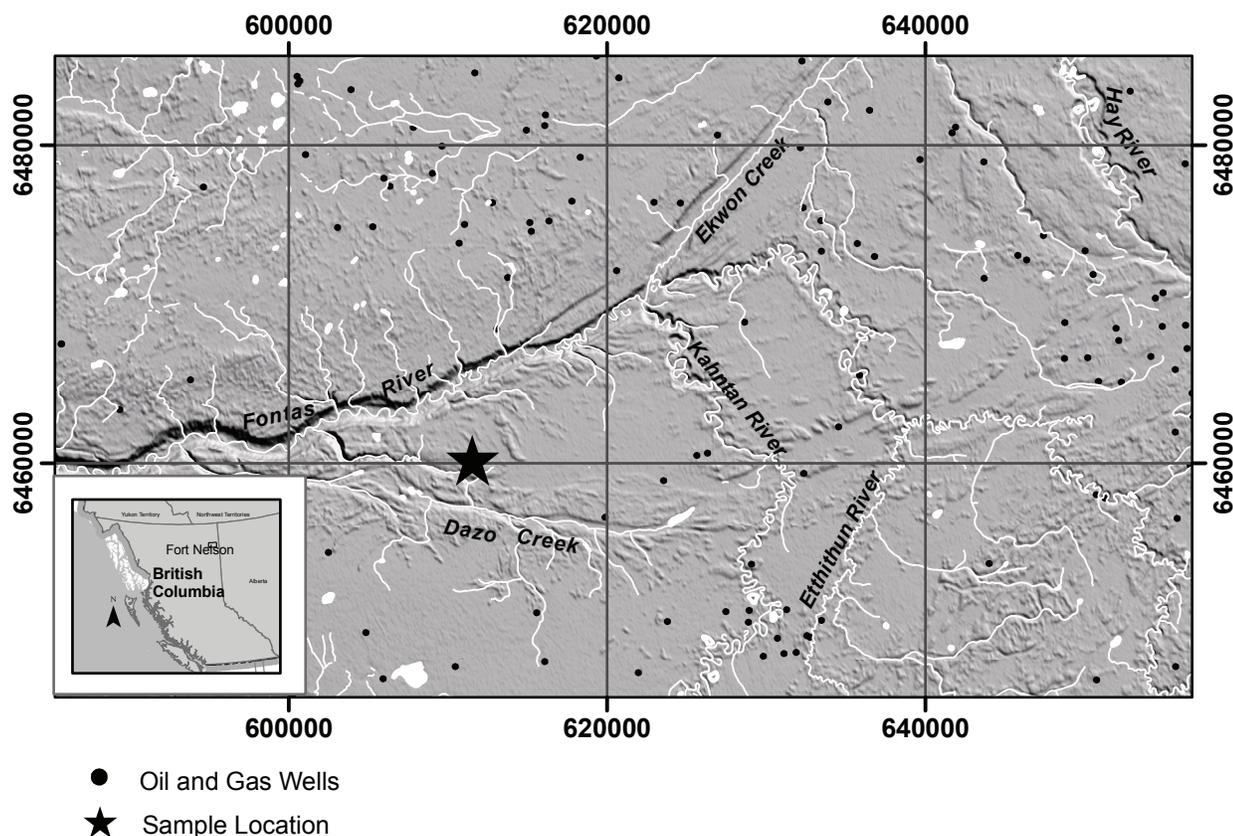


Figure 1. The sample used in the experiments is from the Fontas Dune Field located along Dazo Creek in northeastern British Columbia.

The sample is dominantly fine sand (Figure 2); therefore, a high proportion of material is in the 70/140 size fraction (<0.21 to >0.105 mm). Samples were initially washed using standard ASTM-approved sieves to remove buoyant organic material, silt and clay, preserving the sand-sized fraction (<2.0 to >0.063 mm; Wentworth, 1922). Although the majority of grains in this sample are quartz, there are also grains of other silicate minerals (e.g., feldspar) and lithic fragments of local Cretaceous rock (e.g., siltstone, chert and ironstone; Figure 3a).

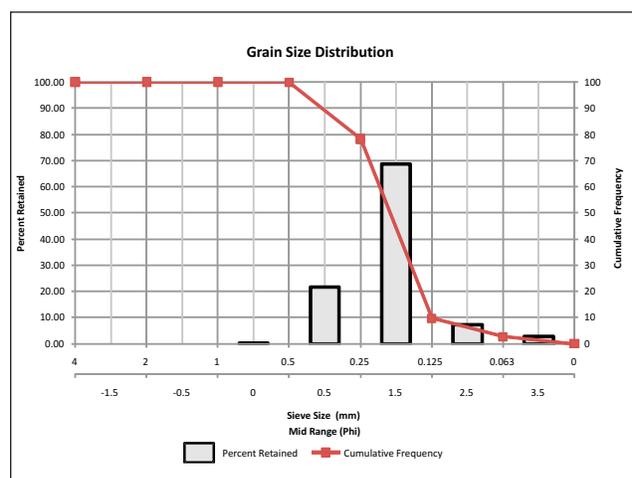


Figure 2. The original unwashed sample from the Fontas Dune Field consists mainly of fine-grained sand.

The equipment used in these experiments is listed and shown in Figure 4. In general, a given number of small steel balls, each 10 mm in diameter and weighing 3.6 g, were used to mill a predetermined weight of dry sample and water. The sample, water and steel balls were placed in a 250 mL plastic bottle and mounted in a table shaker to agitate the mixture for a predetermined period. The processed sample was then screened in standard ASTM-approved sieves to the desired size fraction, then dried in an oven and weighed to determine weight percent loss. This loss is assumed to be the result of the removal of grain coatings and the breakdown of sand grains into silt- and clay-sized material, which was washed out of the sample after agitation. The washed and processed samples were sent to Acme Laboratories (Vancouver, BC) for major oxides analysis. Total abundances of the major oxides and several minor elements were reported on a 0.2 g sample milled to <200 mesh (<0.074 mm). Samples were fused and analyzed by inductively coupled plasma–emission spectrometry (ICP-ES) following a lithium metaborate/tetraborate fusion and dilute nitric digestion. Loss on ignition (LOI) was determined from the weight difference after ignition at 1000°C. Oxide analysis was normalized to the major oxides to account for the organic content assumed to be equivalent to the material lost on ignition.

## Quality Control

Two blind duplicates were prepared and submitted to the laboratory. The laboratory also randomly duplicated one sample. The analytical data for these samples were used to establish precision error. The standard error was determined for three duplicate pairs for the three most abundant oxides (i.e., SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub> and Fe<sub>2</sub>O<sub>3</sub>). The percent error of each pair of analyses was determined by taking the standard error of all three pairs and dividing it by the mean of each pair and reported as a percent. The three percent errors were then averaged for a mean percent error for each of the abundant oxides.

## Experiment 1

This experiment was designed to determine the effects of the duration of attrition on a raw sand sample. Bottles were prepared with 50 g of raw sample, 50 g of water and ten steel balls. The mixture was agitated for 2, 5, 10 and 20 minutes. The processed samples (after agitation) in part 1 of the experiment were washed and screened to the complete sand fraction (<2.0 to >0.063 mm) and in part 2, were washed and screened to 70/140 mesh (<0.21 to >0.105 mm). Samples were then dried, weighed and sent for analysis.

## Experiment 2

This experiment was designed to determine the effects of the duration of attrition on a prescreened (before processing) 70/140 mesh (<0.21 to >0.105 mm) split of the raw sand sample. Bottles were prepared with 50 g of 70/140 mesh sample, 50 g of water and ten steel balls. The mixture was agitated for 2, 5, 15 and 20 minutes. Sets of samples were washed and screened to 70/140 mesh (<0.21 to >0.105 mm), then dried, weighed and sent for analysis.

## Experiment 3

This experiment was designed to determine the effects of increasingly more aggressive attrition on a raw sand sample over several periods of time. Bottles were prepared with 20 g of raw sample, 20 g of water and 10, 15 and 20 steel balls. The mixture was agitated for 2, 5, 10 and 20 minutes. Samples were washed and screened to the complete sand fraction (<2.0 to >0.063 mm), then dried, weighed and sent for analysis.

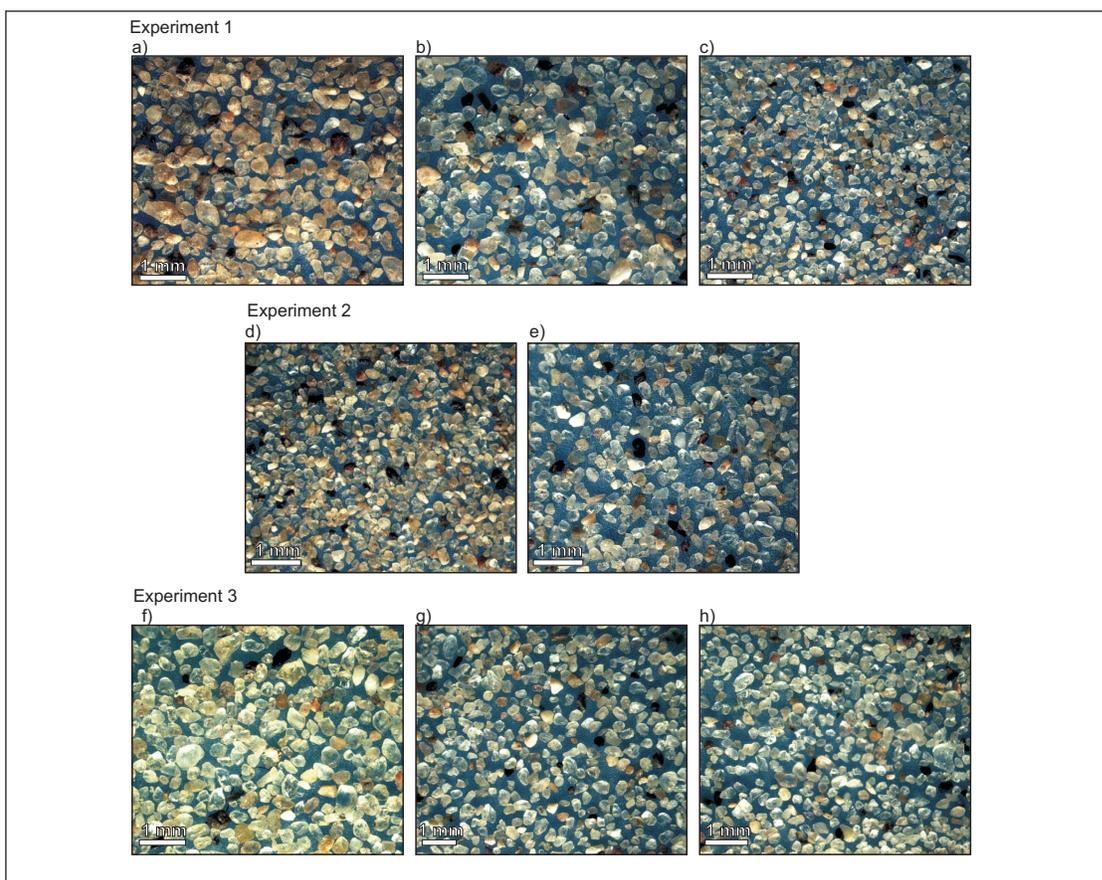


Figure 3. Photograph of sand grains before and after the experiments: a) Unprocessed, washed sand-sized fraction; b) Sand-sized fraction from experiment 1, part 1, after 20 minutes; c) 70/140 mesh-sized fraction from experiment 1, part 2, after 20 minutes; d) Unprocessed, washed 70/140 mesh size fraction used in experiment 2; e) 70/140 mesh-sized fraction from experiment 2, after 20 minutes; f) Sand-sized fraction from experiment 3, after 20 minutes of attrition using 10 steel balls; g) Sand-sized fraction from experiment 3, after 20 minutes of attrition using 15 steel balls; h) Sand-sized fraction from experiment 3, after 20 minutes of attrition using 20 steel balls.



Figure 4. Equipment used in the experiments consisted of a) 250 ml plastic bottles; b) 10 mm, 3.6 g stainless steel balls; c) table shaker; d) scale; e) sieves.

## RESULTS

All analyses are provided in Table 1. In each experiment, the results of the three main oxides ( $\text{SiO}_2$ ,  $\text{Al}_2\text{O}_3$  and  $\text{Fe}_2\text{O}_3$ ) are explored.

### *Quality Control*

The three duplicate analyses offer some measure of variability (Table 2). The quantification of the error gives some assurances that the changes reported in each experiment are significant. Correlation of  $\text{SiO}_2$  and  $\text{Fe}_2\text{O}_3$  in the duplicate pairs is poor, with  $R^2$  values of 0.3289 and 0.0004, respectively (Figure 5). Correlation of  $\text{Al}_2\text{O}_3$  is very good, with an  $R^2$  value of 0.9573. Error bars for each analysis are determined from an average percent error calculated for a standard error and are 0.6663%, 3.04% and 14.33% for  $\text{SiO}_2$ ,  $\text{Al}_2\text{O}_3$  and  $\text{Fe}_2\text{O}_3$ , respectively (Table 3).

TABLE 1. NORMALIZED MAJOR OXIDE GEOCHEMISTRY

| Sample       | SiO <sub>2</sub> | Al <sub>2</sub> O <sub>3</sub> | Fe <sub>2</sub> O <sub>3</sub> | MgO         | CaO         | Na <sub>2</sub> O | K <sub>2</sub> O | TiO <sub>2</sub> | P <sub>2</sub> O <sub>5</sub> | MnO         | Cr <sub>2</sub> O <sub>3</sub> |
|--------------|------------------|--------------------------------|--------------------------------|-------------|-------------|-------------------|------------------|------------------|-------------------------------|-------------|--------------------------------|
|              | %                | %                              | %                              | %           | %           | %                 | %                | %                | %                             | %           | %                              |
| <b>*D.L.</b> | <b>0.01</b>      | <b>0.01</b>                    | <b>0.04</b>                    | <b>0.01</b> | <b>0.01</b> | <b>0.01</b>       | <b>0.01</b>      | <b>0.01</b>      | <b>0.01</b>                   | <b>0.01</b> | <b>0.002</b>                   |
| 1            | 90.21            | 4.57                           | 2.26                           | 0.31        | 0.52        | 0.78              | 1.01             | 0.20             | 0.10                          | 0.03        | 0.005                          |
| 2            | 87.71            | 4.76                           | 4.13                           | 0.38        | 0.65        | 0.82              | 0.99             | 0.36             | 0.13                          | 0.05        | 0.009                          |
| 3            | 91.15            | 4.19                           | 1.93                           | 0.26        | 0.45        | 0.75              | 0.97             | 0.18             | 0.09                          | 0.02        | 0.004                          |
| 4            | 89.46            | 4.71                           | 2.63                           | 0.33        | 0.60        | 0.82              | 1.02             | 0.26             | 0.11                          | 0.03        | 0.017                          |
| 5            | 91.22            | 4.09                           | 1.96                           | 0.25        | 0.47        | 0.74              | 0.96             | 0.19             | 0.09                          | 0.02        | 0.006                          |
| 6            | 89.56            | 4.54                           | 2.72                           | 0.32        | 0.60        | 0.82              | 1.02             | 0.26             | 0.11                          | 0.03        | 0.017                          |
| 7            | 91.57            | 3.93                           | 1.84                           | 0.23        | 0.44        | 0.74              | 0.98             | 0.17             | 0.08                          | 0.02        | 0.004                          |
| 8            | 90.12            | 4.35                           | 2.46                           | 0.29        | 0.57        | 0.82              | 1.01             | 0.23             | 0.10                          | 0.03        | 0.016                          |
| 9            | 91.47            | 3.94                           | 1.89                           | 0.23        | 0.45        | 0.76              | 0.97             | 0.18             | 0.08                          | 0.02        | 0.004                          |
| 10           | 90.11            | 4.31                           | 2.55                           | 0.28        | 0.56        | 0.84              | 1.01             | 0.22             | 0.09                          | 0.03        | 0.006                          |
| 11           | 91.73            | 3.77                           | 1.86                           | 0.22        | 0.44        | 0.75              | 0.95             | 0.17             | 0.08                          | 0.02        | 0.014                          |
| 12           | 89.31            | 4.59                           | 2.91                           | 0.33        | 0.61        | 0.80              | 0.98             | 0.31             | 0.12                          | 0.03        | 0.008                          |
| 13           | 89.42            | 4.57                           | 2.79                           | 0.32        | 0.61        | 0.82              | 1.01             | 0.30             | 0.11                          | 0.03        | 0.016                          |
| 14           | 90.01            | 4.28                           | 2.61                           | 0.30        | 0.59        | 0.80              | 0.97             | 0.29             | 0.11                          | 0.03        | 0.004                          |
| 15           | 89.76            | 4.34                           | 2.78                           | 0.31        | 0.59        | 0.80              | 0.94             | 0.33             | 0.10                          | 0.03        | 0.018                          |
| 16           | 90.98            | 4.13                           | 2.18                           | 0.24        | 0.45        | 0.76              | 0.97             | 0.17             | 0.08                          | 0.02        | 0.003                          |
| 17           | 91.35            | 3.97                           | 2.00                           | 0.22        | 0.45        | 0.76              | 0.96             | 0.17             | 0.08                          | 0.02        | 0.019                          |
| 18           | 91.59            | 3.82                           | 1.99                           | 0.21        | 0.43        | 0.75              | 0.96             | 0.15             | 0.07                          | 0.02        | 0.003                          |
| 19           | 91.94            | 3.70                           | 1.79                           | 0.20        | 0.42        | 0.75              | 0.95             | 0.14             | 0.07                          | 0.02        | 0.019                          |
| 20           | 90.85            | 4.08                           | 2.32                           | 0.24        | 0.46        | 0.76              | 0.97             | 0.19             | 0.09                          | 0.02        | 0.003                          |
| 21           | 91.56            | 3.87                           | 1.99                           | 0.21        | 0.43        | 0.75              | 0.95             | 0.15             | 0.07                          | 0.02        | 0.004                          |
| 22           | 91.68            | 3.82                           | 1.88                           | 0.20        | 0.44        | 0.76              | 0.95             | 0.16             | 0.07                          | 0.02        | 0.017                          |
| 23           | 92.11            | 3.59                           | 1.83                           | 0.18        | 0.41        | 0.73              | 0.91             | 0.14             | 0.07                          | 0.02        | 0.003                          |
| 24           | 91.35            | 3.98                           | 2.00                           | 0.23        | 0.44        | 0.75              | 0.97             | 0.16             | 0.08                          | 0.02        | 0.017                          |
| 25           | 91.62            | 3.86                           | 1.92                           | 0.21        | 0.42        | 0.76              | 0.96             | 0.15             | 0.07                          | 0.02        | 0.004                          |
| 26           | 91.73            | 3.77                           | 1.86                           | 0.21        | 0.43        | 0.74              | 0.97             | 0.17             | 0.07                          | 0.02        | 0.017                          |
| 27           | 92.38            | 3.46                           | 1.76                           | 0.17        | 0.39        | 0.71              | 0.91             | 0.13             | 0.06                          | 0.02        | 0.003                          |

\*Detection Limit

### Experiment 1

Experiment 1 was designed to examine the changes in composition during progressively longer periods of agitation and therefore attrition. Both parts of this experiment were conducted on the raw sand-sized fraction. In part 1, the processed samples were washed to the sand-sized fraction and in part 2, the processed sample was washed to 70/140 mesh size fraction.

In part 1, there was a 4% mass loss in the raw sand-sized fraction over 20 minutes (Figures 3b and 6). During this period, the SiO<sub>2</sub> content increased from 90.21 to 91.73%, a difference of 1.52%. After 20 minutes, the Al<sub>2</sub>O<sub>3</sub> content was reduced from 4.57 to 3.77%, a change of 0.80%, and the Fe<sub>2</sub>O<sub>3</sub> content was reduced from 2.26 to

1.86%, a change of 0.40%. The largest change in the oxide composition happened within the first minute of the experiment. For example, 61.8% of the total increase in SiO<sub>2</sub> content occurred within 1 minute.

In part 2, the 70/140 mesh fraction was separated from the attrition-processed sample. The unprocessed sample experienced a 60.0% loss of mass, indicating that 40% of the sand sample consists of grains in the 70/140 size fraction (Figure 7). When processed, the sample initially lost an additional 4.2% of its mass after 1 minute. Interestingly, the percent mass loss decreased at 5 minutes to a level similar to the unprocessed sample. After 5 minutes, the percent loss fell to below the unprocessed level, indicating the 70/140 size fraction was gaining mass, likely from crushing larger grains. This is consistent with the abundance of angular grains in the sample after 20 minutes (Figure 3c). The SiO<sub>2</sub>

TABLE 2. NORMALIZED MAJOR OXIDE GEOCHEMISTRY OF DUPLICATE SAMPLES FOR QUALITY CONTROL

| Sample       | SiO <sub>2</sub> | Al <sub>2</sub> O <sub>3</sub> | Fe <sub>2</sub> O <sub>3</sub> | MgO  | CaO  | Na <sub>2</sub> O | K <sub>2</sub> O | TiO <sub>2</sub> | P <sub>2</sub> O <sub>5</sub> | MnO  | Cr <sub>2</sub> O <sub>3</sub> |
|--------------|------------------|--------------------------------|--------------------------------|------|------|-------------------|------------------|------------------|-------------------------------|------|--------------------------------|
|              | %                | %                              | %                              | %    | %    | %                 | %                | %                | %                             | %    | %                              |
| *D.L.        | 0.01             | 0.01                           | 0.04                           | 0.01 | 0.01 | 0.01              | 0.01             | 0.01             | 0.01                          | 0.01 | 0.002                          |
| 3            | 91.15            | 4.19                           | 1.93                           | 0.26 | 0.45 | 0.75              | 0.97             | 0.18             | 0.09                          | 0.02 | 0.004                          |
| 3 Duplicate  | 90.66            | 4.29                           | 2.10                           | 0.29 | 0.56 | 0.78              | 1.00             | 0.19             | 0.09                          | 0.02 | 0.015                          |
| 11           | 91.73            | 3.77                           | 1.86                           | 0.22 | 0.44 | 0.75              | 0.95             | 0.17             | 0.08                          | 0.02 | 0.014                          |
| 11 Duplicate | 91.21            | 3.92                           | 2.14                           | 0.23 | 0.47 | 0.77              | 0.95             | 0.20             | 0.08                          | 0.02 | 0.018                          |
| 9            | 91.47            | 3.94                           | 1.89                           | 0.23 | 0.45 | 0.76              | 0.97             | 0.18             | 0.08                          | 0.02 | 0.004                          |
| 9 Duplicate  | 91.71            | 3.92                           | 1.78                           | 0.22 | 0.43 | 0.76              | 0.93             | 0.16             | 0.07                          | 0.02 | 0.003                          |

\* Detection Limit

TABLE 3. SUMMARY OF DUPLICATE ANALYSIS FOR CONSTRUCTING AVERAGE % ERROR BARS

| Oxide                          | Duplicate Sample | Sample 1 | Sample 2 | mean  | % Error | Standard Error | Average % Error |
|--------------------------------|------------------|----------|----------|-------|---------|----------------|-----------------|
| SiO <sub>2</sub>               | 3                | 91.15    | 90.66    | 90.91 | 0.669   | 0.608          | 0.666           |
|                                | 11               | 91.73    | 91.21    | 91.47 | 0.665   |                |                 |
|                                | 9                | 91.47    | 91.71    | 91.59 | 0.664   |                |                 |
| Al <sub>2</sub> O <sub>3</sub> | 3                | 4.19     | 4.29     | 4.24  | 2.87    | 0.122          | 3.04            |
|                                | 11               | 3.77     | 3.92     | 3.85  | 3.16    |                |                 |
|                                | 9                | 3.94     | 3.92     | 3.93  | 3.09    |                |                 |
| Fe <sub>2</sub> O <sub>3</sub> | 3                | 1.93     | 2.10     | 2.02  | 13.85   | 0.279          | 14.33           |
|                                | 11               | 1.86     | 2.14     | 2.00  | 13.95   |                |                 |
|                                | 9                | 1.89     | 1.78     | 1.84  | 15.20   |                |                 |

content increased from 87.71% in the unprocessed sample to 90.11%, a change of 2.40%. The majority of the increase (72.9% of the total change) occurred within the first 2 minutes. The Al<sub>2</sub>O<sub>3</sub> was reduced from 4.76 to 4.31%, a meagre reduction of only 0.45%. Much of this change occurred between 2 and 10 minutes. The maximum Fe<sub>2</sub>O<sub>3</sub> reduction was from 4.13 to 2.46% (a change of 1.67%) after 10 minutes. This indicates that approximately 40.4% of the Fe<sub>2</sub>O<sub>3</sub> in the unprocessed sample was removed in the experiment, 89.8% of which occurred during the first two minutes.

### Experiment 2

Experiment 2 was designed to examine the changes in composition if the 70/140 mesh size fraction was separated from the raw sand sample before processing and was milled over progressively longer periods of time. The sample experienced 7.8% mass loss over 20 minutes (Figure 8). The SiO<sub>2</sub> content increased from 87.71% in the unprocessed

sample to 90.01% at 15 minutes and dropped to 89.76% after 20 minutes, showing a maximum change of 2.30%. The apparent decline in SiO<sub>2</sub> content from 15 minutes to 20 minutes is within the acceptable margin of error, so is not considered significant. The majority of the increase (53.5% of the total change) occurred in the first minute. The Al<sub>2</sub>O<sub>3</sub> values dropped from 4.76% to a low at 4.28% (15 minutes), a change of only 0.48%. Initially, there was a relatively rapid drop in Al<sub>2</sub>O<sub>3</sub> (35% total reduction), but the majority (60.4% total reduction) of loss occurred between 5 and 15 minutes. The Fe<sub>2</sub>O<sub>3</sub> content decreased from 4.13% to a low of 2.61%, with the most significant drop (80.9% of the total reduction) occurring in the first 2 minutes.

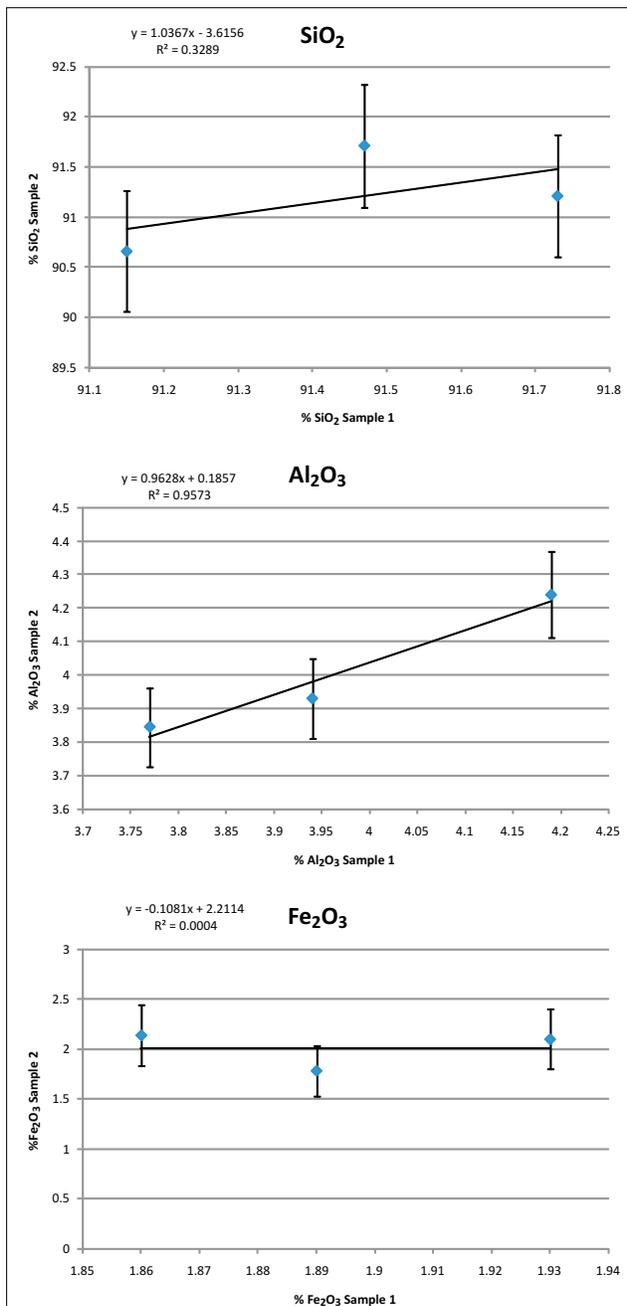


Figure 5. Three duplicate samples were used to assess variation expected in SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub> and Fe<sub>2</sub>O<sub>3</sub>, the three dominant major oxides.

### Experiment 3

Experiment 3 was designed to evaluate progressively more aggressive attrition. After 20 minutes, the 10, 15 and 20 ball experiments lost 4.5%, 4.0% and 6.5% of their mass, respectively (Figure 9). The shape of the percent loss curves suggest that the 10 and 20 ball experiments would continue to lose mass if attrition were continued. The 15 ball experiment, however, shows the curve flattening, indicating that only minor loss would be expected if the experiment was

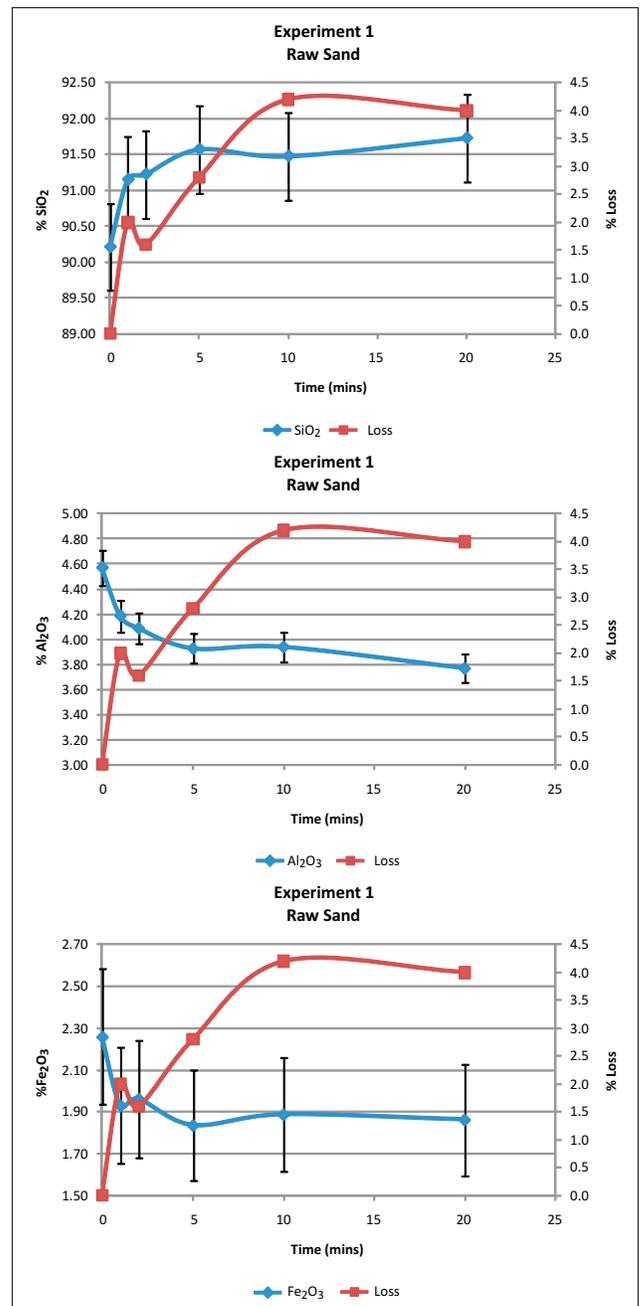


Figure 6. Chart of the percentage mass lost and the SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub> and Fe<sub>2</sub>O<sub>3</sub> analysis versus time from experiment 1, part 1.

continued. The 10 ball experiment showed the SiO<sub>2</sub> content increased by 1.73% from 90.21 to 91.94%. The 15 ball experiment showed only slightly better results, with the SiO<sub>2</sub> content increasing from 90.21 to 92.11%, a change of 1.9%. The 20 ball experiment showed the SiO<sub>2</sub> content increasing from 90.21 to 92.38%, a change of 2.17%. All the curves show a rapid increase in SiO<sub>2</sub> in the early part of the experiment before flattening.

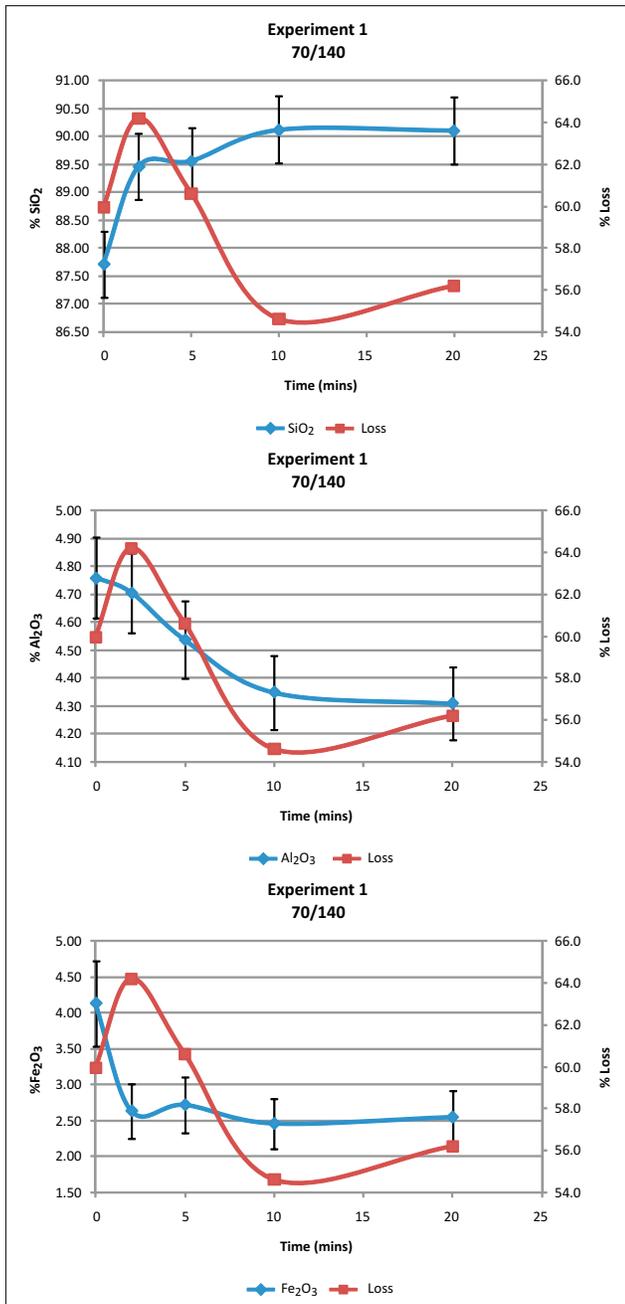


Figure 7. Chart of the percentage mass lost and the SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub> and Fe<sub>2</sub>O<sub>3</sub> analysis versus time from experiment 1, part 2.

## DISCUSSION

The duplicate samples used in the quality-control portion of this experiment show that the precision of the SiO<sub>2</sub> and the Fe<sub>2</sub>O<sub>3</sub> data is relatively poor; therefore, there is a large margin of error associated with the precision of the analyses (indicated by the error bars in Figure 5). There must be a relatively large change in the proportion of these two oxides to consider the change significant. The experiments do show a significant change from the initial measurement to the final measurement, but much of the variability in the later time values are within acceptable error.

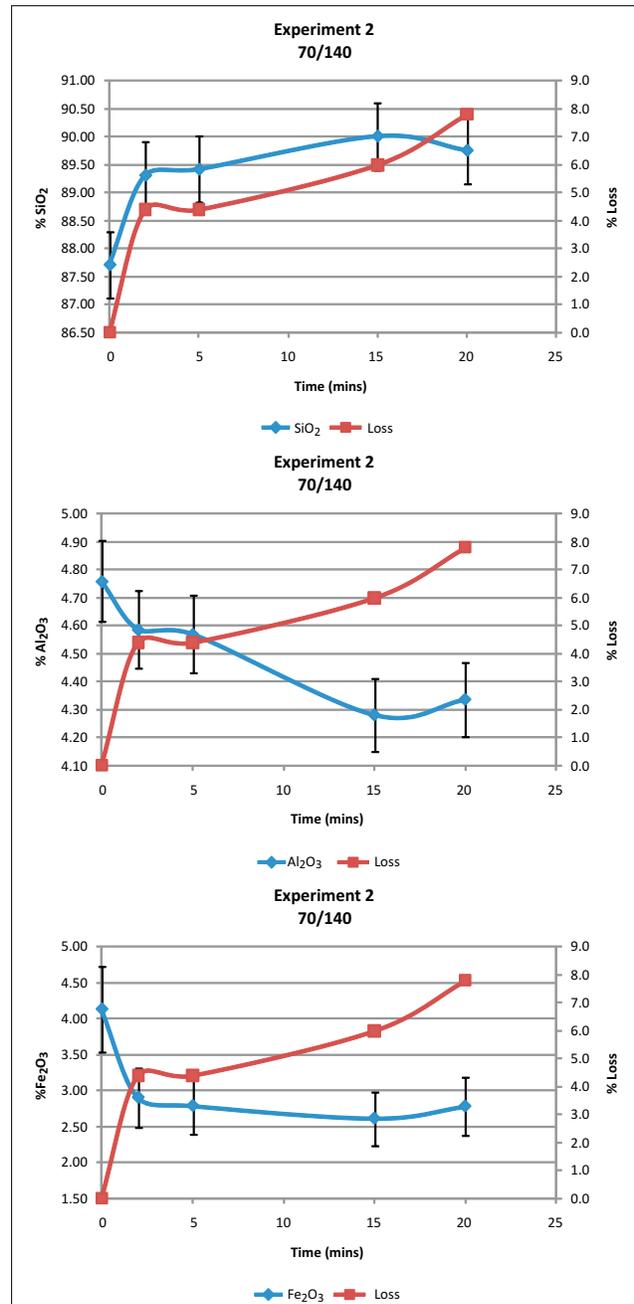


Figure 8. Chart of the percentage mass lost and the SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub> and Fe<sub>2</sub>O<sub>3</sub> analysis versus time from experiment 2.

Experiment 1, part 1 shows approximately 4% mass loss over 20 minutes, indicating grains were breaking down; however, SiO<sub>2</sub> increased to a maximum of 91.73%, 1.52% more than the unprocessed sample (Figure 6). The reduction of Al<sub>2</sub>O<sub>3</sub> and Fe<sub>2</sub>O<sub>3</sub> indicate that grains of undesirable minerals and lithic fragments (e.g., feldspar, mica, clay) were partially removed, but a substantial proportion remained. Most of the SiO<sub>2</sub> increase and Al<sub>2</sub>O<sub>3</sub> and Fe<sub>2</sub>O<sub>3</sub> decrease occurred in early stages of the experiment, so it is unlikely that increasing the duration of attrition would improve the SiO<sub>2</sub> content of the sample under these experimental conditions.

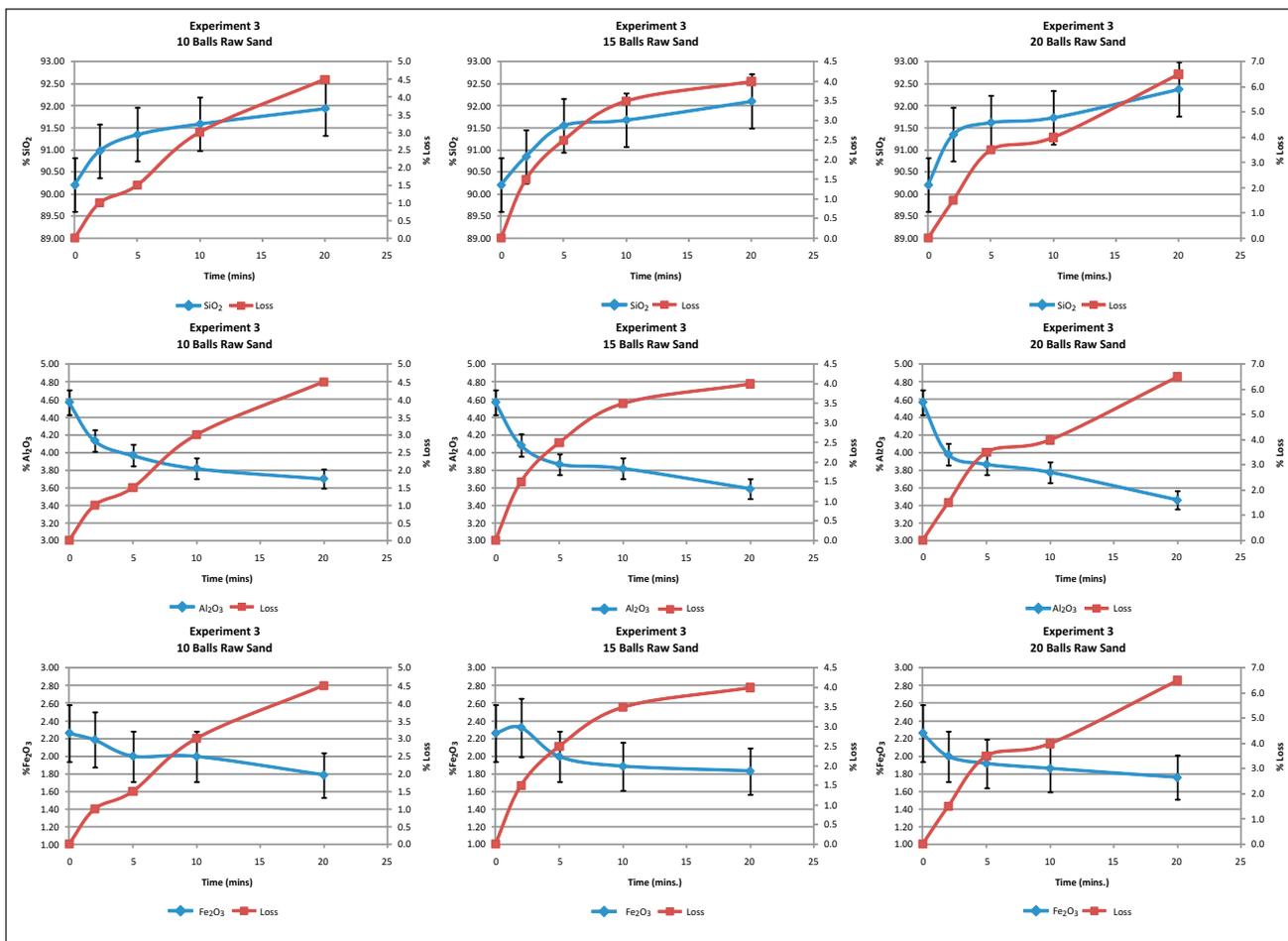


Figure 9. Chart of the percentage mass lost and the  $\text{SiO}_2$ ,  $\text{Al}_2\text{O}_3$  and  $\text{Fe}_2\text{O}_3$  analysis versus time from experiment 3.

Experiment 1, part 2 focused specifically on the impact of attrition on the 70/140 mesh size fraction within a raw sand sample. The experiment shows the addition of mass after 5 minutes of attrition, indicating the generation of grains in the 70/140 size fraction (Figure 7). This, presumably, was from the breakage of larger grains. The inflection in the  $\text{SiO}_2$  curve at 5 minutes (the time when the mass began to increase) suggests larger quartz grains began to fracture into smaller grains at this time. This is significant because angular grains are not desirable for frac sand, so although attrition may improve silica content, the fracturing of grains may degrade the suitability of the sand. The relatively limited and slow decrease in  $\text{Al}_2\text{O}_3$  suggests that  $\text{Al}_2\text{O}_3$ -bearing grains, possibly feldspar and hard (silicified) clay-rich lithic grains, resisted degradation during the course of the experiment. The rapid reduction in  $\text{Fe}_2\text{O}_3$  after 2 minutes suggests that Fe-bearing grains degraded early in the experiment. Although there was a relatively significant increase in the proportion of  $\text{SiO}_2$  from 87.71% in the unprocessed sample to 90.12%, it is well below the suggested  $\text{SiO}_2$  content used in frac sand.

Experiment 2 was designed to test if separating the 70/140 fraction from the raw sand sample would improve

results. The experiment produced a loss of 7.8% and the curve suggests that if the experiment were continued, there may be additional loss of material. The proportion of  $\text{SiO}_2$  in the unprocessed 70/140 size fraction is 87.71% and after processing, the sample had a maximum  $\text{SiO}_2$  content of 90.01%, an increase that mostly occurred in the first 2 minutes. Although this represents a relatively large improvement, it fails to achieve content that meets industry specifications. The flattening of the  $\text{SiO}_2$  curve signifies that continuing the experiment would likely not increase the silica content significantly, probably because quartz grains are being milled in the later stages of the experiment. The  $\text{Al}_2\text{O}_3$  shows an initial rapid decline and then a second drop between 5 and 15 minutes; however, the total reduction of  $\text{Al}_2\text{O}_3$  from 4.76% to 4.28% represents a small improvement. The percentage of  $\text{Fe}_2\text{O}_3$  dropped rapidly, in the first 2 minutes, followed by negligible improvement over the remaining 18 minutes.

Experiment 3 was aimed at testing if the experimental design was more aggressive, there would be an appreciable improvement in the  $\text{SiO}_2$  content. In the 10 ball scenario, there was a 4.5% mass loss and the percent loss curve suggests that additional material would be lost if the

experiment was prolonged. Although most of the increase in  $\text{SiO}_2$  and decrease in  $\text{Al}_2\text{O}_3$  occurred in the first 2 minutes, the curves suggest that if the experiment persisted, there may have been additional improvements. Nonetheless, the maximum  $\text{SiO}_2$  value of 91.94% is only marginally better than the unprocessed sand. The 15 ball experiment was slightly more successful with the  $\text{SiO}_2$  proportion, reaching 92.11%. The percent loss curve flattened over 20 minutes, although material would continue to be lost if the experiment was allowed to continue. Similarly,  $\text{SiO}_2$  would increase and  $\text{Al}_2\text{O}_3$  would decrease if time were extended. The  $\text{Fe}_2\text{O}_3$ , however, shows very little change between 5 and 20 minutes. The 20 ball experiment, considered the most aggressive in these tests, caused the greatest improvement in the sample, and the curves suggest that continuing the experiment may result in additional enhancement. The  $\text{SiO}_2$  content reached 92.38% and the  $\text{Al}_2\text{O}_3$  and  $\text{Fe}_2\text{O}_3$  were reduced to 3.46% and 1.76%, respectively; however, the experiment did produce an abundance of broken, angular quartz grains. Either the time duration should be limited or additional processing would be required to round the grains to meet the well rounded/spherical shape requirements suggested by the industry specifications.

In general, the experiments resulted in limited beneficiation. This, in part, is attributed to the limited scope of the experimental design. Nonetheless, the findings suggest that attrition alone is not sufficient to improve the content of the sand samples to an acceptable level and other beneficiation (magnetic separation, floatation, acid dissolution, etc.) is likely required. Attrition may offer some economic benefit early in processing, initially reducing the highly deleterious grains before more expensive and intensive physical, physiochemical or chemical processing is conducted. In these experiments, the unprocessed sample was relatively high in  $\text{SiO}_2$ , so the improvements were meagre. Attrition may be an appreciable process where the sand has a lower initial  $\text{SiO}_2$  content. Regardless, some form of attrition and washing should be included in any processing circuit. The duration of attritions will need to be optimized for industrial-scale beneficiation. In these experiments, the majority of improvement occurred in the early stages of the experiments (<5 minutes). Extending the attrition process may have adverse affects on the grain shape because of breakage.

Although silica content is an important factor in the suitability of sand for use as a proppant, other parameters, such as crush strength, acid solubility and grain shape are also critical. Silica content may not meet standards, but if the sand is satisfactory in the other parameters, it should still be considered for use as a proppant or proppant blend. Ultimately, further evaluation of the deposits' qualities is necessary to design and optimize a beneficiation process.

## CONCLUSION

- These attrition experiments show that light milling of the sand sample will result in the degradation of deleterious sand grains; however, attrition alone was insufficient to elevate the  $\text{SiO}_2$  content to industry standards. Attrition may be an effective early step in a processing circuit, removing highly deleterious grains in lower-grade samples. Other physical, physiochemical and chemical processing will be required to upgrade deposits for use as industrial proppants.
- Much of the improvement in the silica content was achieved early in the experiments. Consequently, a short duration of attrition would benefit the quality of the sample and would only add a brief processing step.
- Aggressive milling and longer durations tended to produce the greatest increase in  $\text{SiO}_2$  and the greatest decrease in  $\text{Al}_2\text{O}_3$  and  $\text{Fe}_2\text{O}_3$ . Aggressive milling and extending the experiments beyond approximately 5 minutes produced abundant angular grains that are detrimental to the quality of the proppant. Optimizing the time and intensity of attrition must be explored at the industrial scale to maximize the effectiveness of the processing step.

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