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An Assessment of Laboratory Sample Splitting Techniques

Nenuwa, Olushola B. and Adetiloye, Adeola
Department of Minerals & Petroleum Resources Engineering,
The Federal Polytechnic, P.M.B. 5351, Ado-Ekiti, Ekiti state, Nigeria

ABSTRACT

Two different laboratory sample splitting methods were used to prepared representative sub-samples. Performance evaluation tests were conducted using the coning and quartering sampling technique and the Jones riffle sample splitter, the bulk sample was iron chips and sand mixture. The sample splitting exercise was repeated thrice for each method and the results obtained were recorded. On completing the performance evaluation tests, the percentage content of the iron chips and sand in the parent sample were compared with the percentage contents in the splitted samples. It was observed that the value of the absolute difference between the percentage content of sand in the parent sample and splitted sample for the Jones riffle sample splitter is lower compared to that of coning and quartering technique. It was also observed that the value of the absolute difference obtained for iron content in the parent and splitted sample is also lower for sampling with Jones riffle splitter than when coning and quartering technique was used. Jones riffle sample splitter is therefore recommended for splitting dry granular samples whenever a cheap, efficient, accurate and less sophisticated technique for sample splitting is desired.

Keywords: *Absolute Difference, Riffler, Sampling, Splitting, Sub-Sampling*

1. INTRODUCTION

Sampling is the means whereby a small amount of material is taken from the main bulk in such a manner that it is representative of that larger amount. Great responsibility rests on a very small sample, so it is essential that samples are truly representative of the bulk (Homles, 1991). Wherever possible samples should be taken of the material when it has been reduced to the smallest particle size consistent with the process. For instance, the ground ore pulp will be easier to sample, and will give more accurate results than the feed to the primary crusher (Wills, 2006).

Sampling procedures cover the practice of selecting representative quantities of test material in the field, to evaluate bulk materials. Examples of the test materials are bulk granular solids, slurries, sludge, grains, and solid fuels. It is necessary to be able to sample bulk materials during shipment and during processing operations. Taggart (1945) defined sampling as: "The operation of removing a part convenient in size for testing, from a whole which is of much greater bulk, in such a way that the proportion and distribution of the quality to be tested (e.g. specific gravity, metal content, recoverability) are the same in both the whole and the part removed (Sample)." In a laboratory before performing a size analysis, chemical analysis, or any other determination of material properties, it is necessary to take a representative sample from the lot being analyzed. For most metallurgical or environmental operations, the methods of sampling includes: Grab sampling, Coning and Quartering, Chute-type sample splitters and Rotary riffles. Sampling can be a significant source of error in the measurement process. The characterization and cleanup of hazardous waste sites require data that meet site-specific levels of acceptable quality if scientifically supportable decisions are to be made (Gerlach *et al*, 2003).

The act of sampling may introduce more uncertainty than all of the subsequent steps in the measurement process (Jenkins *et al*, 1997; Crumbling, 2001; Allen, 1996). This is especially true for heterogeneous particulate samples. The goal of most sampling procedures is to obtain a representative sample. In terms of characterizing the level of contaminants, the ideal representative sample would have the same concentration of contaminants as the original sample. Procedures for obtaining representative samples from particulate matrices have been studied for some time in the minerals industry and a comprehensive sampling theory originated by Pierre Gy has been developed (Pitard, 1993; Gy, 1998; Myers, 1997; Smith, 2001).

The need for sample homogeneity prior to laboratory analyses has been long recognized by geologists, chemists, and members of other scientific disciplines. Homogeneity is the degree that the material under investigation is mixed resulting in the random distribution of all particles in the sample. Completely homogenous materials are so rare that they may be considered non-existent (Ingamells and Pitard, 1986). Yet, scientists must strive to obtain a homogenous sample in order to obtain data exhibiting minimal error attributable to sample heterogeneity. Care must be taken during the sub-sampling phase of soil preparation, during sample transport, unpacking, and transfer to other containers in the laboratory to avoid particle sorting via different particle densities, shapes, sizes, and resistance of certain minerals to mixing, such as magnetite (Muller,1967; Ingamells and Pitard, 1986; Reeves and Brooks, 1978). Numerous methods have been used to obtain homogeneous soil samples. The various methods range from simple grinding and sieving of the sample to a desired particle-size to various mixing and splitting devices and machines (Schumacher *et al*, 1990).

Ingamells and Pitard (1986) expressed serious doubts as to the value of mechanical splitters. They stated that: 'In general, machines that use mechanical violence and look and sound as though they are efficient are most likely to cause segregation of heavy and light, large and small, and flat and round particles'. Further concerns have been expressed concerning sample breakdown to finer particle sizes due to the violent tumbling in the machines. The riffle splitter (also called a chute splitter, Jones splitter, or sample splitter) is perhaps the most common mechanical method for sample homogenization and/or sample size reduction (Ingram, 1971; Mullins and Hutchison, 1982). The riffle splitter also provides one of the best general methods of sample mixing to obtain bulk sample homogeneity (Ingamells and Pitard, 1986). A riffle splitter is a device having an equal number of narrow sloping chutes with alternate chutes discharging the sample in opposite directions into two collection bins (Fig. 1). Sample homogenization is achieved by repeated pouring of soil through the splitter and combining the halves between passes. The use of the riffle splitter as a sub-sampling device is done in a similar manner with the exception that after the sample is passed through the splitter, one collection pan is replaced with a clean pan. The material in the 'replaced" pan, which contains about one-half of the original sample, is then passed through the riffle splitter again thereby reducing the volume in the clean pan to one-quarter of its original sample volume. This process of sample reduction is repeated until the desired weight or sample size is obtained (Schumacher *et al*, 1990).

Perhaps the best known sample splitting method is the classical coning and quartering technique. This technique involves pouring the sample into a cone, flattening the cone, dividing the flattened cone into four equal divisions (quartering), and then removing two opposite quarters (Fig. 2). The remaining two quarters are repiled into a cone and the process is repeated until the desired sample size is obtained. Variations on the process are possible which can enhance the speed of sample size reduction by using just one quarter (chosen at random) to continue the splitting process or which allow this method to be modified to homogenize large sample. The use of the coning and quartering method to homogenize a sample involves the removal of the first quarter and repiling it into a cone followed by the subsequent repiling of the opposite quarter and then the remaining two quarters to reform a single cone (Raab *et al*, 1990). This process is repeated several times until sample homogeneity is achieved. Several sources of error for this method have been identified. Van Johnson and Maxwell (1981) reported that during the cone and quarter process on large samples (several kilograms), there is the danger of unequal segregation of heavier materials during the flattening and coning of the sample. Similar to the riffle splitting techniques, dusting is also a possible source of error during cone formation. Sample loss from the inability to recollect all the soil from the underlying material, the ability of the sample to "cling" to the underlying material via static charges, and sample embedment are all further sources of error.

Selecting a reduced sample mass from the quantity available for analysis is one the most common procedures performed in an analytical laboratory. However, little attention is usually given to this procedure and the effect it may have on the quality of the data (Gy, 1998). From a laboratory analyst's point of view, a reported sample concentration is an estimate of the average level of analyte in the original mass of sample received for analysis. For particulate or soil samples, this value is often based on the analysis of an aliquot selected from the soil sample. Sub-sampling practices have an important effect on the reported results. Improper sub-sampling procedures can lead to results that have significant biases and large imprecisions (Pitard, 1993; Myers, 1997).

Several studies have been conducted comparing the effectiveness of the riffle splitter to other homogenization techniques. The aim of this study is to assess and compare the performance of a mechanical sampler (the Jones riffle splitter) and a traditional sampling method (coning and quartering sampling). This work will help a mineral analyst to choose an appropriate sample splitting technique that will produce the best homogenised test sample which will introduce the smallest level of inaccuracy.

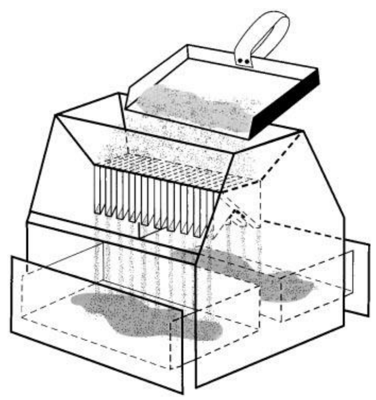

Figure 1: The basic components of a riffle splitter include the scoop, an even number of chutes, or riffles, and a pair of collection pans (Source: Gerlach *et al*, 2002).

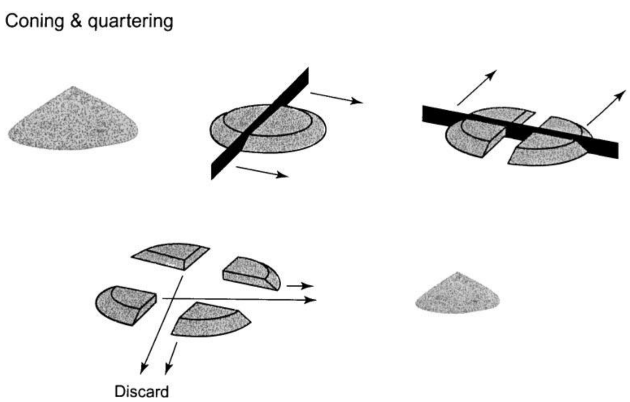

Figure 2: Coning and quartering repeatedly divides a sample into halves until the desired sample size is achieved (Source: Gerlach *et al*, 2002).

2. METHODOLOGY

i. Materials and Equipment

The materials and equipment required for this study are: iron chips, sand, bar magnet, Jones riffle splitter, cutting knife and electronic weighing balance.

ii. Sample Collection

8kg quantity of sand and 4kg quantity of iron chips were collected inside containers at Abuja boys hostel and the Mechanical Engineering workshop respectively both located within the campus of the Federal Polytechnic, Ado-Ekiti, Ekiti state.

iii. Sample Preparation

1000g of iron chips and 2500g sand were weighed using the electronic weighing balance. The two samples were thoroughly mixed to produce a homogenous sample of 3500g iron chips and sand mixture. This was used as the test sample.

iv. Experimental Procedure for Coning and Quartering Sampling Technique

3500g of the test sample was poured on a flat table to form a cone shaped pile, and the pile was uniformly flatten until the diameter is approximately equal to four times the thickness of the pile. The sample was divided into half by vertically passing the cutting knife through the center of the pile. In a similar manner, the halved pile was divided into another half, thus quartering the sample. The sample was combined diagonally with opposite quarters of the materials forming two splitted samples. Then one of the two halves was stored. The other half was spread out on the table and a bar magnet was moved over it in order to separate the iron chips from the sand. The iron chips that were collected by the magnet were then placed inside a container. The two samples were thereafter weighed separately and the result was recorded. This procedure was repeated using the other splitted sample as the test sample in order to do a re-splitting. The re-splitting was done thrice in order to obtain a total of four different sampling tests. The results obtained were also recorded.

v. Experimental Procedure for the Jones Riffle Sample Splitter

The test sample was prepared and 3500g was weighed as earlier done on the weighing balance. The mixture of the sand and iron chips was poured evenly into the hopper of the Jones riffle splitter. The samples then flowed through the alternately arranged passages in the opposite directions (chute/riffle bank) into the two collecting pan under the dividing head outlet. The feed sample was then divided into two representative sub-samples. One of the sub-samples was then spread on the table and the bar magnet was placed very close to it in order to separate the iron chips from the sand. The iron chips that were collected by the magnet was placed inside a container and the sand was placed in another container. The two samples were thereafter weighed separately as earlier done and the results were recorded. This procedure was also repeated thrice in order to re-split the other portion of the splitted sample. All the results obtained were recorded.

vi. Determination of the Percentage Composition of the Sample Constituents

The percentage composition of the constituents in the parent and splitted samples were calculated using Equation 1 and 2:

Percent (%) of iron in the parent or splitted sample = $\frac{Mi}{M} \times 100\%$ ………..….. 1

Percent (%) of sand in the parent or splitted sample = $\frac{Ms}{M} \times 100\%$ …………………. 2

Where, Mi = Mass of iron in the parent or splitted sample
Ms = Mass of sand in the parent or splitted sample
M = Mass of the mixture of sand and iron chips in the parent or splitted sample

3. RESULTS AND DISCUSSION

After conducting the performance evaluation test for both coning and quartering technique and the Jones riffle sample splitter, the results of the tests were recorded and the data obtained was used to calculate the percentage content of the iron chips and sand in the test samples as described earlier. The absolute difference between the percentage content of sand and iron in the parent and splitted sample for both sampling techniques were also obtained. The results obtained were tabulated and presented graphically in fig. 3 – 9.

Fig. 3 is a bar chart showing the percentage of materials in the parent sample and splitted sample for the coning and quartering sampling technique. From the graphical analysis shown on fig. 3, for test 1, the percentage composition of iron and sand in the parent sample is 28.6% and 71.4% respectively. The percentage composition of iron and sand in the splitted sample is 71.4% and 27.8% respectively. The difference between the percentage content of iron and sand in the parent sample and splitted sample for test 1 is large, which indicates that this result is not reliable. For test 2, the percentage composition of iron and sand in the parent sample is 72.0% and 27.3% respectively. The percentage composition of iron and sand in the splitted sample is 61.55% and 27.5% respectively. The difference between the percentage content iron and sand in the parent and splitted sample for test 2 is small which indicates that it is a reliable result. For test 3, the percentage composition of iron and sand in the parent sample is 65.1% and 30.9% respectively while the percentage composition of iron and sand in the splitted sample is 72.2% and 28.1% respectively. The difference between the percentage content of iron and sand in the parent and splitted sample for test 3 is also small which also indicates a reliable result. For test 4, the percentage composition of iron and sand in the parent sample is 72.0% and 28.0% respectively, the percentage composition of iron and sand in the splitted sample also is 75.4% and 23.4% respectively. Hence, the difference between the percentage composition of iron and sand in parent and splitted sample for the test 4 is low and this further confirms the efficiency of the coning and quartering technique.

Fig. 4 is a bar chart showing the absolute difference of the percentage content of sand in the parent sample and splitted sample for coning and quartering splitting technique, from the chart, test 2 gives the lowest absolute difference of sand content in the parent and splitted sample (0.22%) which means that test 2 is the most reliable result. The absolute difference obtained for test 3 and 4 are also low, 2.78% and 6.58% respectively, this further assures the efficiency of this technique. The value obtained for test 1 is high (44.2%), thus, the result of test 1 is erroneous and so re-splitting the sub-sample is necessary.

Fig. 5 is a bar chart showing the absolute difference of iron content in the parent sample and splitted sample for the coning and quartering splitting technique, from the chart, test 4 gives the lowest absolute difference of iron content in the parent and splitted sample (3.39%) which means that test 4 results are very reliable. The value obtained for test 3 is also low (7.04%). The value obtained for test 1 and 2 are high (42.8% and 34.5% respectively). The parent sample must thus, be re-splitted at least twice in order to obtain sub-samples that will adequately represent the content of the parent samples.

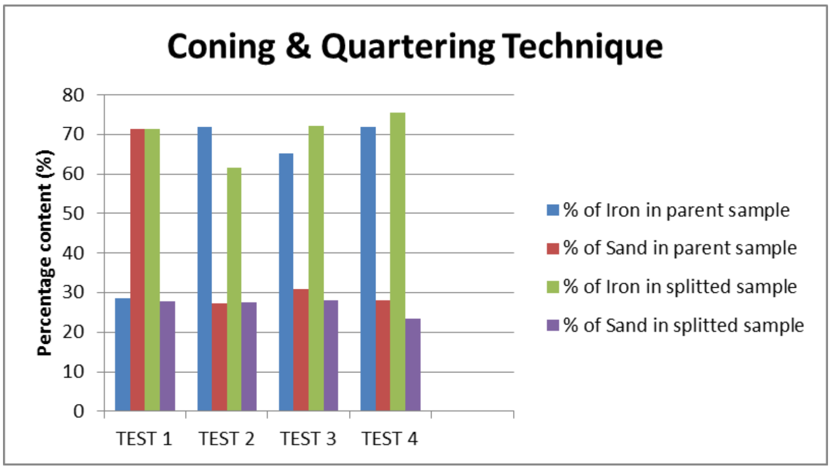

Fig. 3: Bar chart showing the percentage of materials in the parent sample and splitted sample for coning and quartering technique

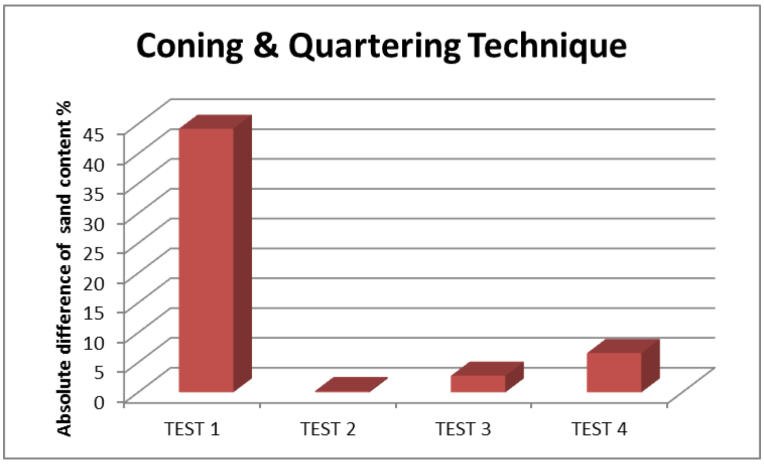

Fig. 4: Bar chart showing absolute difference of sand content in the parent sample and splitted sample for Coning and Quartering splitting technique

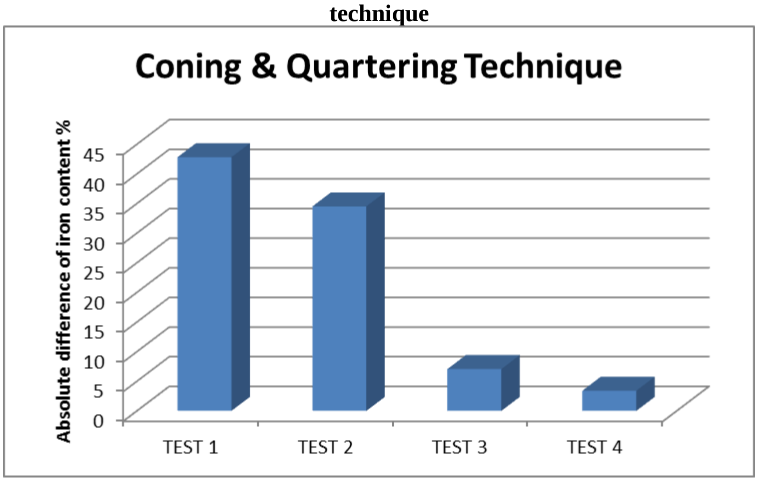

Fig. 5: Bar chart showing absolute difference of iron content in the parent sample and splitted sample for Coning and Quartering splitting technique

Fig. 6 is a bar chart showing the percentage of materials in the parent sample and splitted sample for Jones riffle sample splitter, from the chart, test 1 shows that the percentage content of iron and sand in the parent sample is 28.6% and 71.4% respectively while that of iron and sand in splitted sample is 26.1% and 73.4% respectively, hence the difference between the percentage composition of iron and sand in parent and splitted sample for test 1 is small. For test 2, the percentage content of iron and sand in parent sample is 26 .2% and 73.8% respectively and that of the percentage content of iron and sand in splitted sample is 26.2% and 73.6% respectively. For test 3, the percentage content of iron and sand in parent sample is 26.2% and 73.8% respectively, while that of the splitted sample is 26.9% and 73.2% respectively. For test 4, the percentage composition of iron and sand in parent sample is 26.9% and 73.2% respectively, while for the splitted sample, it is 26.1% and 73.9% respectively for both iron and sand. The difference in the percentage composition of iron and sand in parent and splitted sample for all the four tests are small and these results showed that the sample splitting method is efficient for all the four tests conducted.

Fig. 7 is a bar chart showing the absolute difference of the percentage sand content in the parent sample and splitted sample for Jones riffle sample splitter, from the chart, test 2 gives the lowest absolute difference of the percentage sand content in the parent sample and splitted sample (0.15%) which means that test 2 sub-sample is accurately representing the bulk sample. Test 1 gives absolute difference of 1.92% while test 3 and 4 gives absolute difference of 0.65% and 0.72% respectively. These results confirmed that Jones riffle sample splitter is an efficient sampling device.

Fig. 8 is a bar chart showing the absolute difference of the percentage iron content in the parent sample and splitted sample for the Jones riffle sample splitter, from the chart, test 2 gives the lowest absolute difference (0.09%). The value of the absolute difference for test 1 is 2.47% while that of test 3 and 4 are 0.66% and 0.72% respectively. These results further confirmed the efficiency of the Jones riffle sample splitter.

Fig. 9 is a bar chart comparing the absolute difference of the sample contents in both coning and quartering technique and the Jones riffle sample splitter, from the chart, test 1, 2, 3 and 4 gives lower value of the absolute difference of sand content in the parent sample and splitted sample for Jones riffle sample splitter when compared to that of coning and quartering sampling technique. The absolute difference of iron content in the parent sample and splitted sample is also lower when Jones riffle sample splitter was used than when coning and quartering technique was used for sampling. These implied that sampling using the Jones riffle sample splitter gives more accurate and reliable results than sampling using the coning and quartering sampling technique.

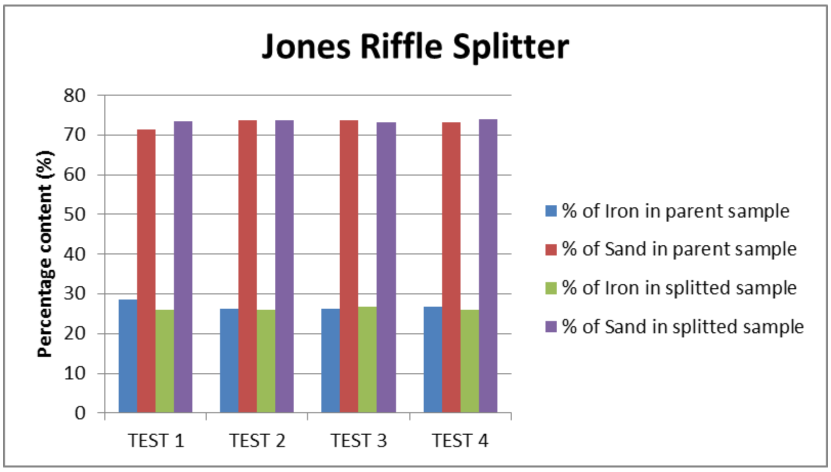

Fig. 6: Bar chart showing the percentage of materials in the parent sample and splitted sample for Jones Riffle Sample Splitter

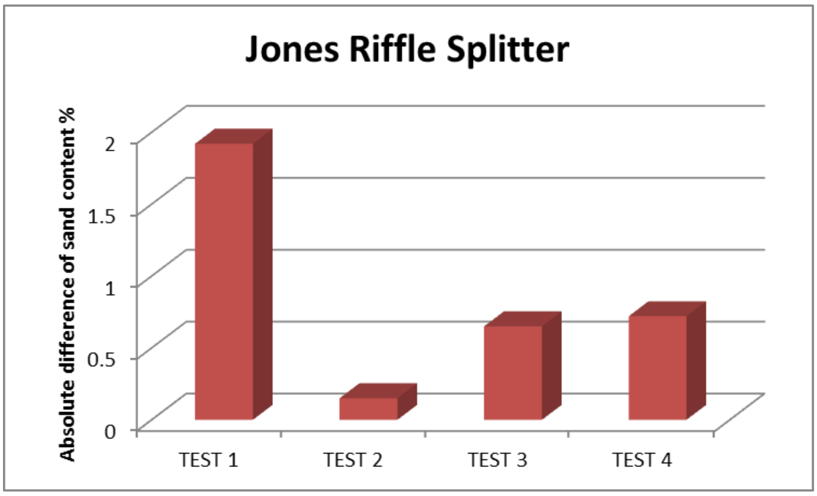

Fig. 7: Bar chart showing absolute difference of sand content in the parent sample and splitted sample for Jones riffle sample splitter

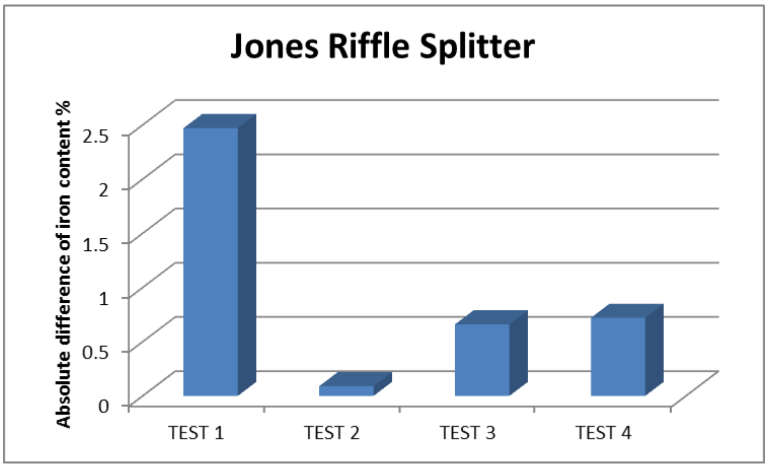

Fig. 8: Bar chart showing absolute difference of iron content in the parent sample and splitted sample for Jones riffle sample splitter

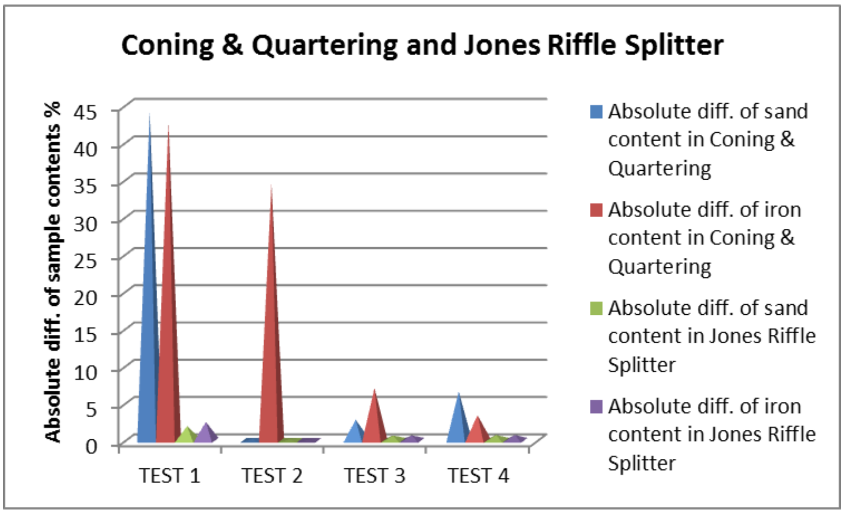

Fig 9: Bar chart comparing the absolute difference of sample contents in both the coning and quartering technique and the Jones riffle sample splitter.

4. CONCLUSIONS

The coning and quartering sampling technique and sampling using the Jones riffle sample splitter was actually conducted by observing all necessary precautions. From the performance evaluation results obtained, the value of the absolute difference between the percentage content of sand in the parent sample and splitted sample for the Jones riffle sample splitter is lower compared to that of coning and quartering technique. It was also observed that the value of the absolute difference obtained for iron content in the parent and splitted sample is also lower for sampling with Jones riffle splitter than when coning and quartering technique was used. It can therefore be concluded that using Jones riffle sample splitter for sampling procedure will give more accurate results with higher efficiency than when the coning and quartering sampling technique is being used, the sub-samples that will be obtained will also be more homogeneous and will more closely represent the bulk or parent sample than when coning and quartering technique is adopted. Although, the coning and quartering technique is more economical, it does not assure accuracy. The Jones riffle sample splitter is therefore recommended to be used for mineral sample splitting whenever a cheap, mechanized and less sophisticated technique for sample splitting is desired. Further researches should however be encouraged in order to evaluate the performance of other sample splitting techniques.

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