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# Upgraded Analytical Protocols in Bauxite Refining Industry Using Composite Sampling Approach to Minimize Laboratory Analysis Load

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## Abstract

The laboratories in the bauxite processing industry are always under a heavy workload of sample collection, analysis, and compilation of the results. After size reduction from grinding mills, the samples of bauxite are collected after intervals of 3 to 4 hours. Large bauxite processing industries producing 1 million tons of pure aluminium can have three grinding mills. Thus, the total number of samples to be tested in one day reaches a figure of 18 to 24. The sample of bauxite ore coming from the grinding mill is tested for its particle size and composition. For testing the composition, the bauxite ore sample is first prepared by fusing it with X-ray flux. Then the sample is sent for X-ray fluorescence analysis. Afterwards, the crucibles are washed in ultrasonic baths to be used for the next testing. The whole procedure takes about 2 - 3 hours. With a large number of samples reaching the laboratory, the chances of error in composition analysis increase. In this study, we have used a composite sampling methodology to reduce the number of samples reaching the laboratory without compromising their validity. The results of the average composition of fifteen samples were measured against composite samples. The mean of difference was calculated. The standard deviation and paired t-test values were evaluated against predetermined critical values obtained using a two-tailed test. It was found from the results that paired test-t values were much lower than the critical values thus validating the composition attained through composite sampling. The composite sampling approach not only reduced the number of samples but also the chemicals used in the laboratory. The objective of improved analytical protocol to reduce the number of samples reaching the laboratory was successfully achieved without compromising the quality of analytical results.

**Keywords**

Composite Sampling, Analytical Methods, Sampling Technique, Bauxite Composite Sampling, Sampling Protocols, Aluminum Ore Sampling Technique, Sampling Methods

## 1. Introduction

Bauxite ore is mined, crushed, and transported to a refining plant for the production of alumina ($Al_2O_3$), the primary raw material for producing pure aluminium. At the refining plant, the crushed ore is first ground to increase the surface area for further processing. Laboratory samples after grinding mills are subsequently taken for bauxite ore analysis. These samples are tested in the laboratory for physical tests such as particle size and moisture. The elemental analysis is usually carried out using the industrial standard X-ray fluorescence (XRF) method [1] [2]. Larger plants, producing 1 million tons of aluminium per annum, normally have 3 grinding mills. The samples are taken from each grinding mill in regular intervals such as after every 4 hours depending upon the quality of the source of bauxite. The number of samples coming from 3 grinding mills for XRF analysis can reach as high as 30 - 40 samples per week which results in an excessive load on laboratory equipment.

The XRF machine requires the samples to be prepared before analysis. The preparation step involves the fusion of the sample with flux (lithium or sodium borate) at a temperature of 1100˚C [3]. The fusion step can be manual or automated depending upon the setup available in the laboratory. The sample is then cooled. The sample preparation step takes about 35 minutes. The XRF analysis is performed after the preparation step. Once the analysis is complete, the used crucibles are subjected to a 30-minute ultrasonic bath at 60C for cleaning. Further cleaning, washing, and drying take time. Thus, 5 - 7 samples for analysis can take a total of over 2 - 3 hours of laboratory time. The XRF in the industries is always operating at high load conditions. Increasing the number of XRF analysis machines also requires an increase in the technical workforce which results in additional laboratory operational expenses.

The purpose of the present study is to reduce the number of samples reaching the laboratory while maintaining their validity. This is the basic requirement of correct sampling *i.e.* all the individual samples have an equal representation is the final gross sample. The general method employed for this purpose is taking the samples, analysing them and then taking averages of their constituents. The method is considered standard in the industry because it can accommodate any periodic variation in the ingredients of bauxite samples. However, it is noticed that the samples coming from a single source have minor composition variations for longer periods. Such small variations do not impact production quality or quantity and require minimal or no change in process parameters. However, the

overall load on the laboratory for such samples is very high. The purpose is to propose an analytical methodology that is precise and accurate in terms of composition variation representation of bulk material (bauxite) being ground. For this purpose, we conducted the research to compare the compositional analysis of bauxite by using average and composite sampling methods.

Composite sampling significantly decreases the analytical costs because the number of samples reaching the laboratory for analysis is reduced [4]. In composite sampling, several small samples are composted into one representing the composition of all the collected samples. The collected samples are homogenized to make one composite sample which is analysed. The conventional statistical method allows the reduction of uncertainty or cost/analysis load on laboratory equipment. The problem is that the reduction of one of the aforementioned factors increases the other. In composite sampling, either uncertainty or cost is maintained to the allowed limit while reducing the other factor (uncertainty or cost). The composite sample involves the physical mixing of a number of samples as shown in **Figure 1**.

The major benefit of composite sampling is that only one analysis has to be made for the composite sample which is a representation of each of the individual samples [5]. Composite sampling increases the representativeness of the measurement of individual samples thus this method reduces the costs of estimating a total or an average value. Another advantage of this method is that a composite sample can be extended to classify original individual sample units. The conditions where analytical costs dominate over sampling costs, composite sampling mitigates the problem [6].

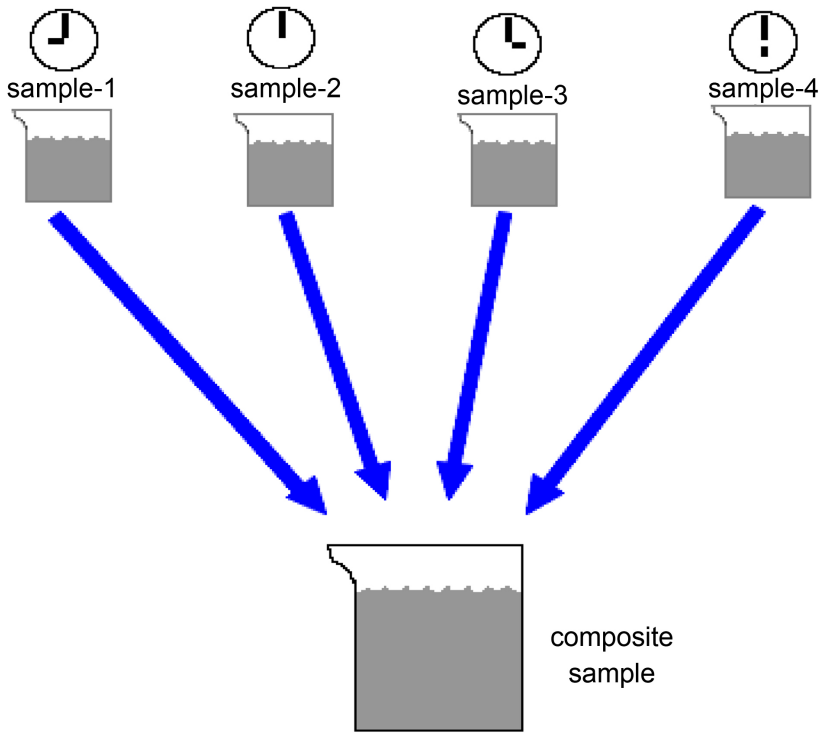


**Figure 1.** Representation of composite sampling.

The bauxite refining industry needs to continuously analyse the composition of raw materials. The analytical costs always dominate over the sampling cost because several steps are involved in the analysis of the ground ore [7]. The critical issue is how to determine, validate and compensate for the information lost during composite sampling. This question is more pronounced in cases such as the presence of a single component (such as some contaminant) above the threshold in the individual sample. The process should avoid the dilution of such a sample when mixing it with others to make a composite sample. Another requirement is the measurement of variables *i.e.* concentrations of elements/compounds in individual samples of bauxite ore. In case, there is a major variation noted in the composite sample, the individual samples need to be preserved, so that their composition can be obtained. The composite sampling process has a fair advantage over the average sampling method because, in the latter mentioned, there is no possibility of determining the individual composition of the samples. Composite sampling provides a rigorous process for the determination of the composition of individual samples when needed.

In this study, we have established a comprehensive methodology that helps reduce the analytical load on the laboratory by reducing the number of samples. Another objective is to reduce the number of testing chemicals (X-ray flux) in the laboratory. Moreover, a strategy has been proposed for storing the individual samples, in case of the requirement for an individual sample composition. The rest of the paper has been organized into the subsequent section. The second section provides a comprehensive literature survey of the use of composite sampling techniques in industrial sampling setups including the bauxite refining industry. Section 3 describes the methodology of the experiment. This section also explains how composite samples are derived and measures the difference between compositions obtained through the average sampling method and composite method. Section 4 presents the results of the experiment and the variance in the results from the average sampling method and composite method. Section 5 highlights the limitations of the current study. Finally, Section 6 concludes the study.

## 2. Literature Review

The idea of composite sampling was first proposed in 1935 by Yates according to which when the weights of several samples are to be ascertained, one can increase precision and reduce the cost by weighing the samples in combination rather than separately [8]. This concept of uniting the samples before some measurement is taken was then reintroduced by Dorfman and Hotelling [9], [10]. In their research, Hotelling tried to minimize the variance of least square estimates. They laid the foundation for weighing design and introduced the concept of optimal designs. Dorfman experimented on blood samples where composite samples were prepared using the combinatorial sampling technique. This method remained of particular interest because of reduced analysis costs.

In the recent past, the upsurge in environmental protection and minimizing laboratory waste prompted the demand for composite sampling techniques. Moreover, environmental regulatory bodies have mandated the safe disposal and cleanup of hazardous laboratory wastes [11]. A large amount of testing in laboratory setups generates considerable waste. Reducing the laboratory samples reduces waste production as well as the cost of sampling and analysis [12]. The true applications of composite sampling are less in the cases of environmental studies and more in soil analysis because of the nature of analysis *i.e.* detection of a particular element or compound.

## 2.1. Composite Sampling Applications

The broad classification of composite sampling applications includes the cases of classifying or identifying individual samples with certain properties or estimation of the mean of a stochastic process. The later application involves analysis of the variance of the components. The other applications include the classification of individual samples for continuous and discrete measurement cases, increasing analytical sensitivity, and maintaining confidentiality in the estimation of a certain property. The potential advantages of composite sampling in the aforementioned applications include reduced costs, variance, and a number of false-positive results. Another advantage is an increase in the total amount/quantity of samples to be measured. The disadvantages include increased sampling costs, as more number of small samples have to be collected, though the analytical cost is reduced. There is a possibility of false-negative results in case of over-dilution due to mixing. Uneven mixing can also alter the final analytical results.

Composite sampling has been recommended for applications such as estimating the mean concentration of constituents of the composite sample collected during the site investigation process. An example of such applications is the sampling of ores during the refining process. The Environmental Protection Agency (EPA) recommends composite sampling techniques when there is a high degree of certainty in the site history and a low possibility of an extremely large variation in compositions. While measuring concentrations of constituents of a composite sample, first a criterion value or action level is set. At or above/below this level individual measurements need to be taken to ascertain the ore composition. The composite sample with values within the predefined action levels is classified as not exceeding the action level. Moreover, the composite sampling data can be applied without any modifications, especially in cases where information on individual samples is not critical to make changes in process parameters. This is of particular interest when sampling for the composition of ores because the refining process takes the input in bulk quantity. Therefore the composite sampling better represents the bulk composition because a number of samples are taken from the material at different intervals at different locations.

In one study, researchers compared the composite sampling technique with

conventional grab sampling for contaminations of superfund sites in the USA [13]. The objective of the research was to find the spatial extent of contaminated soil. It was deduced that the individual samples were highly variable in composition when compared to composite samples. The laboratory analytical error was also small for composite samples. The author recommends further study of spatial variability in composite sampling. The author recommends that composite sampling is the most effective way to reduce measurement errors as well as the number of samples.

In similar research, the chemical properties and lateral variability of the forest floor were examined across LF and H horizons [14]. The variables measured in the research were N, P, S, C, Zn, K, Cu, Mn, Al, Fe, Ca, lipids and pH. Evaluated using mean values, the lateral variability of K, Mn, and Cu was high in the lateral direction. In the second section, composite samples were prepared analysed and compared for depth and bulk density. It was evaluated that the values from the composite deviated from one standard from the mean. The exceptions were Cu in LF horizons. The author concluded that composite sampling provides an adequate estimate of the mean value of subsamples analysed individually. The author also recommended composite sampling as the only feasible method to obtain the estimate of the mean.

Laboratory analysis costs are reduced using composite sampling as concluded in a study carried out on the soil samples collected at depths of 0 - 15 and 15 - 30 cm under the sugar maple trees [15]. Two composite samples containing ten core samples were compared to averages of four individual samples collected beneath a total of 10 trees. It was found from the study that the coefficient of variation of measurements was low for composite samples when compared with individual samples. The author concluded that this parameter indicates potential savings in laboratory costs. Another similar study measured the soil pollution data using composite sampling [16]. The author used analysis of variance to measure the homogeneity of composite samples. The study also demonstrated the applicability of statistical methods for small spatial distributions in soil. The author recommended composite sampling for soil pollution assessment, however, advised that the method should not be directly applied to other use cases because the final results highly depend on the spatial distribution of the analyte.

Bauxite ore sampling protocols have been set and applied all over the industry as standard procedures. Research describes the experimental procedures for the calibration sampling parameters *i.e.* segregation-free analysis, heterogeneity test, and sampling tree experiments [17]. The calibration is necessary to reduce the sampling error caused by minimum representative sample masses. Once the experimental calibration is complete, the data can be used in Gy's formula for calculating variances of fundamental sampling error. This procedure can be applied to individual, mean, and composite samples to compare the final results. **Table 1** lists some other studies highlighting the advantages, disadvantages, and guidelines for composite sampling.

Table 1. Guidelines, advantages and disadvantages of composite sampling.

| Proclamation | Literature sources |
|---|---|
| **Advantages** | |
| Composite sampling reduces analytical costs | [18] [19] [20] [21] |
| Composite sampling provides a better estimate of the mean concentration of the samples | [22] [23] |
| It helps in identifying the units with the highest level of constituents of interest | [24] [25] [26] |
| When a benchmark is set for appropriately adjusted composition levels, Composite sampling can help detect major variations in composition due to the increased number of samples. | [27] [28] |
| **Disadvantages** | |
| Some information is lost when individual samples are mixed with each other. Dilution can occur. This loss of information is a concern when one has to determine the threshold of a constituent in the samples. | [29] [30] |
| Composite sampling is not suitable in cases where activity levels are close to analytical detection limits. | [26] [31] |
| For non-homogenous composite samples the spatial variability or temporal information is lost. | [20] [32] |
| Composite sampling should not be used when the integrity of individual samples alters due to physical mixing such as loss of volatile components. | [25] [33] |
| Surrogate ratios cannot be established using composite sampling | [34] [35] |
| **Composite sampling guidelines** | |
| If there is a large variation in compositions caused by the heterogeneous nature of some contaminant, the researcher needs to account for potential large errors. | [36] [37] |
| Composite sampling is very helpful in reducing analysis costs for cases where the size of pattern sampling is smaller than the spacing between statistically necessary random sampling areas. | [29] [38] |
| In order to make the composite samples, the individual samples must be of equivalent volume/weight. The individual samples must be homogenized properly to make a composite sample. | [39] [40] |
| The user must develop a strategy to re-test the individual samples, in case the threshold value set for a composite level is reached. This will make it possible to retrieve the potentially lost information during the compositing the samples | [41] [42] |
| The users must define the threshold and modify the investigation level for the composite samples. Moreover, the user must account for the dilution factor when calculating the final results. | [43] [44] [45] |

## 2.2. Unit Estimation and Retesting of Composite Samples

In this paper we are proposing the sample unit classification and retesting schemes, therefore, this section of the literature review is dedicated to the aforementioned topics as well as unit estimation methodologies.

Two major queries are answered while creating a composite sample and unit estimation *i.e.* will composite sampling reduces the analysis and cost, and what will be the optimal composite size? The initial work done by Dorfman answers these questions, however, been modified by several researchers and created numerous extensions. In one research, three different composite designs were evaluated against simple sampling [46]. The objective of the experiment was to classify the samples according to the detection of HIV (human immunodeficiency virus) antibodies. The independent variables were the specificity of the analytical procedure, three prevalence, and sensitivity. The dependent variables were costs and classification variables. Experiment results show a false positive predictive value of 0.02 for the composite sampling method at a prevalence of 0.004 while for non-composite samples, its value was 0.98. When the prevalence rate was 0.024, the false-positive predictive value remained stable at 0.02 while for non-composite samples, it reduced to 0.45. The authors concluded that the choice of composite design depends on whether the objective is to decrease the number of fall negatives or positives. The authors recommended composite sampling due to cost savings and an increase in estimation accuracy.

Retesting the individual samples from composite samples is necessary when finding the maximum value of a trait. In one study researchers proposed a model, the hypothesis of which was based on the sample units which exhibit a high degree of correlation [47]. They developed a sequential composite sampling design. In this methodology, the composite sample with the maximum value was identified and the individual sample units were retested. The author compared their sequential sampling method with random sampling and found the former one better in terms of detection.

Sample unit estimation is one of the major hurdles towards composite sampling because of the uncertainty of the impact of unit size, location, and the number of units. Two theories can be applied for sample unit estimation *i.e.* weighing design theory and inverse theory. In weighing design theory the individual objects are weighted assuming measurement errors unrelated. The following relation has been devised for estimating the individual unit weights:

$$y = Cx + \sigma_m^2 I$$

In this equation, $y$ is $C$ weighing operations, *and* $x$ represents any number of unknown weights. $\sigma_m^2 I$ is the covariance matrix of errors in measurement. As the value of $C$ is greater than n, thus least square estimation techniques can be used to solve for individual weights ($x$). Although there are some differences in composite sampling and weighing design theory, however, it can be adopted by changing the lower bound parameters of variance in each estimate. Moreover, the authors also claimed that the theory can be applied to retesting schemes.

This technique also reduces the measurement error in situations to quantify the general soil constituents. The inverse theory is applied to the conditions where the analysis or characterization of a site of interest is. The objective of sampling is to estimate the proportion of constituents at different locations. The sampling design will be collecting $c$ samples. The numerical regularization methods are applied to estimate the concentrations of the constituent.

This literature survey of composite sampling was aimed to find out the best-devised techniques which can be applied to bauxite ore samples in our study. The literature provided a basis for our research design, methodology, experimental strategy, and finalizing the results. The fore coming sections describe the details of our research methodology.

## 3. Methodology

The methodology section has been divided into five subsections *i.e.* EPA composite sampling protocols, current sampling methods, composite sampling approach, development of the hypothesis, and paired t-test statistics that have been adopted in this research for evaluation of the difference between results of the average composition against composite sample composition. The objective is to establish the relationship between the results of composite samples with the daily average of compositions of samples and observe the deviation.

### 3.1. EPA Composite Sampling Protocols

EPA has laid down a detailed methodology for collecting composite samples of soil [48]. The recommended guiding principles recognized by EPA have been modified to accommodate bauxite sampling which is more homogeneous in its composition than soil samples. Following are the principles followed while collecting bauxite ore samples after grinding mill:

- Discrete samples were taken of equal size from a predefined sampling point (after grinding mill) and were composited laterally.
- Each discrete subsample was thoroughly homogenized in the laboratory for testing purposes.
- It was ensured that each discrete subsample was contributing an equal amount of material to the composite sample.
- The discrete subsamples were of similar characteristics *i.e.* particle size, and type of material (bauxite).
- The composite sampling is affected by highly volatile substances present in subsamples. In the case of bauxite, there are no such volatile substances.
- During the homogenization and composting of samples, it was ensured by taking equal-weight samples that the target analyte are not compromised.
- A clear record of discrete subsample that made composite sample was maintained until the ore is processed and the final product is tested.
- The sub-samples placed for possible future examination were 10 grams each.
- One in 10 samples was randomly selected for additional analysis of its

sub-samples. This information is useful for monitoring the homogeneity of the composite samples.

### 3.2. Current Sampling Method

Sampling protocols are consistently requiring the operator to take the sample at the exit of each grinding mill after every four hours. The current sampling protocols involve taking 5 - 7 samples from the exit of each grinding mill. The samples are sent to the laboratory for analysis. The total number of samples coming from three grinding mills can reach a figure of 21. Thus only 50% of samples are subjected to an XRF test to reduce the laboratory load and the other 50% are only analysed for physical properties such as particle size and moisture content etc. The samples for elemental analysis are first prepared for XRF in AFT Phoenix 6000 Bead maker. Once the beads are ready, they are subjected to the XRF test for composition analysis. Figure 2 shows the current sampling protocols followed in the industry.

The objective is to devise a more comprehensive approach that not only reduces the load on laboratory analysis equipment but also represents a better inclusion of unit population in a composite sample.

### 3.3. Composite Sampling Approach

The composite sampling approach consists of collecting individual bauxite samples at regular intervals over 24 hours. The bauxite ore is collected in a common container and sub-samples are maintained in individual containers for future analysis if required. The objective here is to compare the results of an average of five samples with the results of a single composite sample containing individual bauxite samples. For calculating the average, five samples were taken each from mill-A, mill B, and mill C. The samples were prepared for XRF analysis to find the composition of each sample. Once the compositions are found, the average composition of each constituent was calculated for the five samples. This average composition is then compared with the constituent components of the composite sample. Figure 3 shows the difference between average sampling composition and composite sampling.

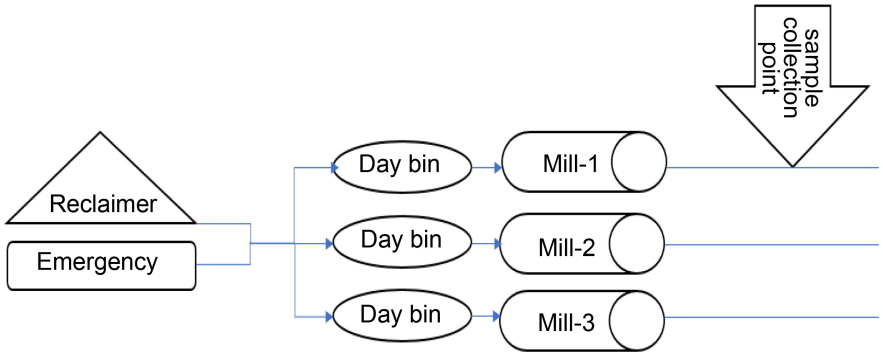


Figure 2. Current sampling protocols.

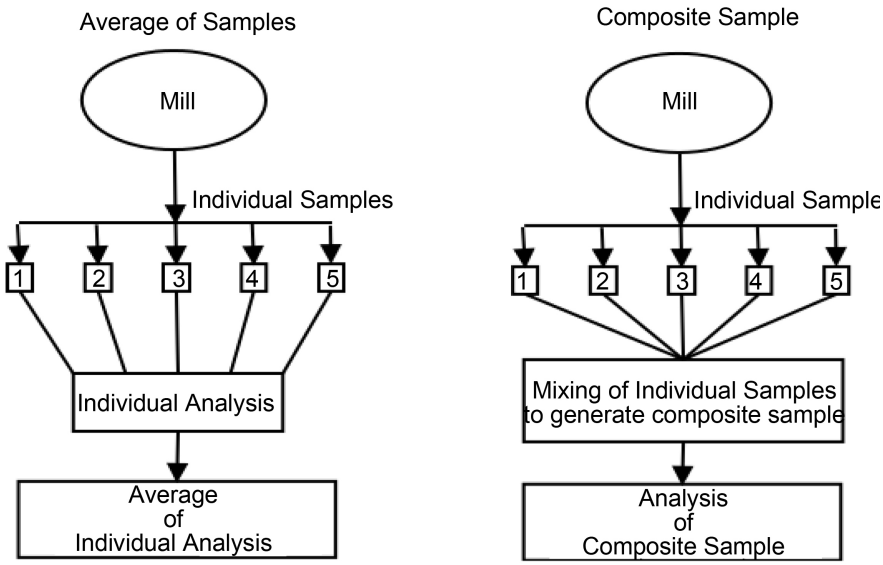


**Figure 3.** Methodology of the average composition of samples vs. composition of the composite sample.

### 3.4. Experimental Setup and Conditions

The bauxite ore samples were prepared using AFT Phoenix 6000 automatic bead maker using the following procedure.

1) A mixture of flux and an unknown sample is weighed and fused in a platinum crucible at 1100˚C, then cast into a platinum mould to produce a glass disc for XRF analysis.

2) Sampling and Sample Preparation: Bauxites, Mud and Sands are dried at 110˚C for at least two hours, and then weighed into a stainless steel scoop.

3) The sample is transferred to a crucible containing flux, stirred with a plastic stirring rod, and then fused in the machine.

4) Once the cycle is complete, the bead was cooled and labelled before being removed from the mould and transferred to an XRF sample cup or clean plastic bag.

5) Discs were handled on their edges to avoid contamination, and if the disc "sticks" to the mould it can be released by dropping the mould onto a granite block.

The beads are now ready for XRF sampling. For XRF, we used industrial standard CTX Benchtop/CounterTop XRF Analyzer—Model 800. The beads were subjected to a CTX XRF analyzer to get the compositions.

### 3.5. Development of Hypothesis and Paired T-Test Statistics

The null and alternative hypotheses for this study are stated below:

Null hypothesis—There is no significant difference between the two sets of data. (Average composition of sum samples and Composite of Day samples).

$$H_o : u_1 = u_2$$

where $H_o$ represents the null hypothesis, $u_1$ is the population means of average

compositions and $u_2$ is the mean of composite sample composition.

The alternative hypothesi—There is a significant difference between the two sets of data. (Average composition of sum samples and Composite of Day samples).

$$H_o : u_1 \neq u_2$$

In order to validate the hypothesis and compare the mean of two measurements (composition of average samples vs. composition of composite samples), the paired t-test is used here. It is a parametric test and is used to compare the means of two measurements taken from the same sample (bauxite). The purpose of this test is to determine whether the alternative hypothesis is valid *i.e.* the mean difference between paired-sample compositions are significantly different from zero. This procedure helps in determining whether the means of two samples are different knowing the variance. The following relation is used to calculate the Paired t-test statistics:

$$\text{Test statistic} - t = \frac{d - \Delta 0}{SDd/\sqrt{n}}$$

where $d$ is the mean of sample differences, Δ0 is the mean of the population, SDd is the standard deviation of the differences and n is the sample size.

Following are the data requirements for paired t-test which were met before applying the statistics:

- The dependent variable was defined (5 intervals).
- The subject (bauxite) in each sample was the same *i.e.* the bauxite sample in the first group is also present in the second group.
- The samples were random because of grabbing samples from the continuous flow of milled bauxite ore on the belt.
- There was a normal distribution of the difference between the paired values.
- There were no outliers in the difference between average and composite samples.

Once the data has been arranged and paired t-test results are evaluated, the next step is to validate the null hypothesis and test the statistical significance. A two-tailed test (two-sided test) is selected which gives a measure of the value greater or less than a critical range of values. If the sample results are less than the critical value, the null hypothesis is valid else the alternative hypothesis is accepted. With a 95% confidence level and 16 degrees of freedom, the critical value = 2.12 as shown in the following Table 2.

In the next step, the paired t-test values are measured against the critical value to test the null hypothesis.

## 4. Results and Discussion

5 samples from three mills (A, B & C) were analysed for the constituents. The average of 5 samples is calculated. A composite sample was made by collecting and homogenizing five 50 g sub-samples. Figure 4 illustrates the composite

Table 2. Two-tailed test for valuation of critical value.

| Degree of freedom | Significance level | | | | |
|---|---|---|---|---|---|
| | Two-sided test | | | | |
| | 90% | 95% | 99% | 99.9% | 90% |
| | 0.1 | 0.05 | 0.01 | 0.001 | 0.1 |
| 1 | 6.31 | 12.71 | 63.66 | 637 | 3.08 |
| 2 | 2.92 | 4.30 | 9.92 | 31.6 | 1.89 |
| 3 | 2.35 | 2.18 | 5.84 | 12.9 | 1.64 |
| 4 | 2.13 | 2.78 | 4.60 | 8.61 | 1.53 |
| 5 | 2.02 | 2.57 | 4.03 | 6.87 | 1.48 |
| 6 | 1.94 | 2.45 | 3.71 | 5.96 | 1.44 |
| 7 | 1.89 | 2.36 | 3.50 | 5.41 | 1.41 |
| 8 | 1.86 | 2.31 | 3.36 | 5.04 | 1.40 |
| 9 | 1.83 | 2.26 | 3.25 | 4.78 | 1.38 |
| 10 | 1.81 | 2.23 | 3.17 | 4.59 | 1.37 |
| 11 | 1.80 | 2.20 | 3.11 | 4.44 | 1.36 |
| 12 | 1.78 | 2.18 | 3.05 | 4.32 | 1.36 |
| 13 | 1.77 | 2.16 | 3.01 | 4.22 | 1.35 |
| 14 | 1.76 | 2.14 | 2.98 | 4.14 | 1.35 |
| 15 | 1.75 | 2.13 | 2.95 | 4.07 | 1.34 |
| 16 | 1.75 | 2.12 | 2.92 | 4.01 | 1.34 |
| 17 | 1.74 | 2.11 | 2.90 | 3.97 | 1.33 |

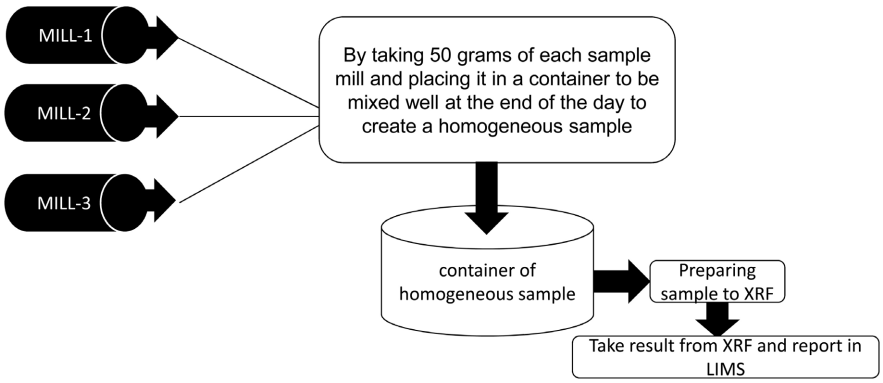


Figure 4. Composite sampling.

sampling technique.

The composite sample was analysed for the compositions. The difference in composition from the average sample was calculated against composite samples. The mean of the difference "d" was calculated. The standard deviation of difference "d" was calculated. Finally paired t-test was applied to find the t-value. The Figure 5 represents the composition values of samples taken from mill A. It can

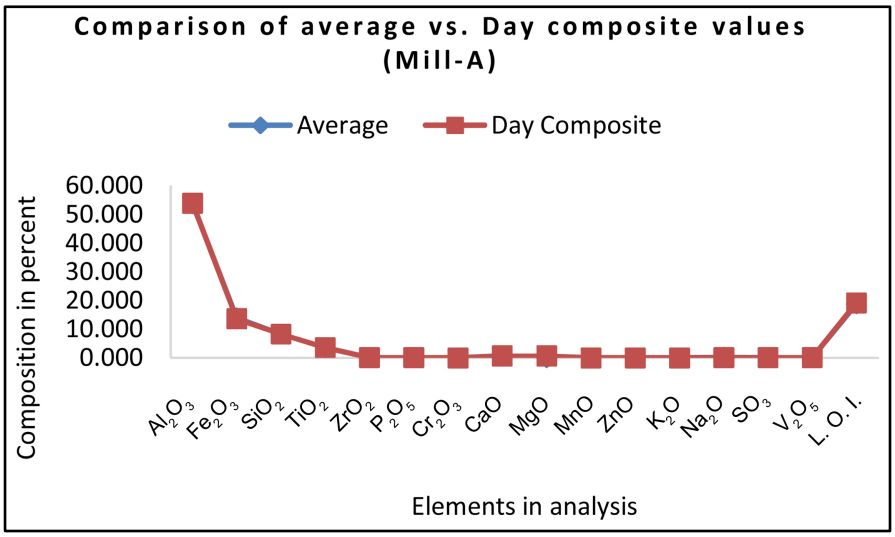


Figure 5. Comparison of average vs. day composite values for mill-A.

be observed that two values *i.e.* average and day composite are highly overlapping which means there is a negligible difference, later validated through statistical techniques. It can be observed from the results that the paired test statistics value calculated in **Appendix A** is −0.8022 which is less than the critical value of 2.12. Thus null hypothesis is valid in this case (Figure 5).

Figure 7 represents the comparison of the composition of samples for which their average was taken vs. day composite sample composition for Mill-B. It can be observed that two values *i.e.* average and day composite compositions are highly overlapping which means there is a negligible difference which was later validated through statistical techniques.

It can be observed from the results that the paired test statistics value provided in **Appendix B** is 0.1544 which is less than the critical value of 2.12. Thus null hypothesis is valid in this case (Figure 6).

Figure 7 represents the comparison of the composition of samples for which their average was taken vs. day composite sample composition for Mill-C. It can be observed that two values *i.e.* average and day composite compositions are highly overlapping which means there is a negligible difference which was later validated through statistical techniques.

It can be observed from the results that the paired test statistics values provided in the **Appendix C** which is 0.009 which is less than the critical value of 2.12. Thus null hypothesis is valid in this case as well (Figure 7).

From the above results it can be evaluated that instead of analyzing 5 samples, one composite sample representing all 5 samples suffices for the purpose of analysis. The paired t-test statistics values compared with critical values are much less and thus validate the null hypothesis. This procedure reduces the load on the laboratory as well as increases the sample accuracy.

The benefits of composite sampling evaluated from this study can be summarized below:

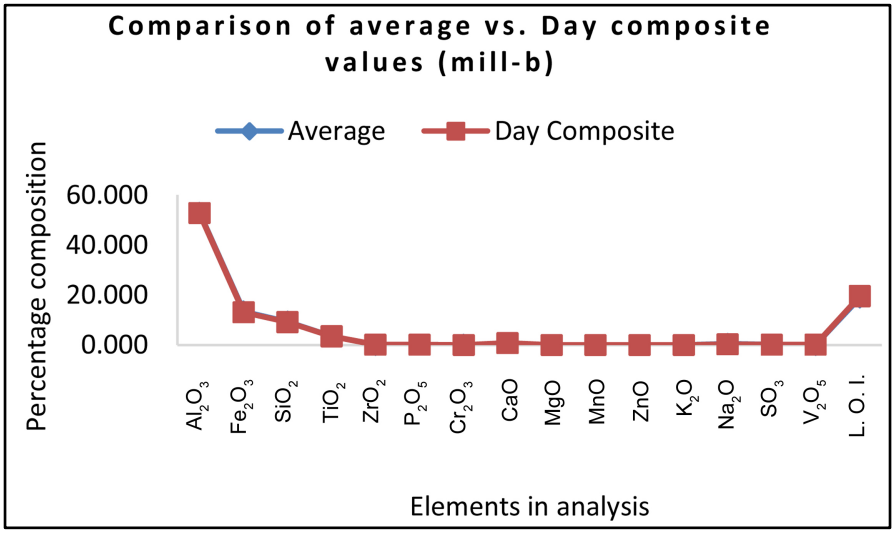


**Figure 6.** Comparison of average vs. day composite values for mill-B.

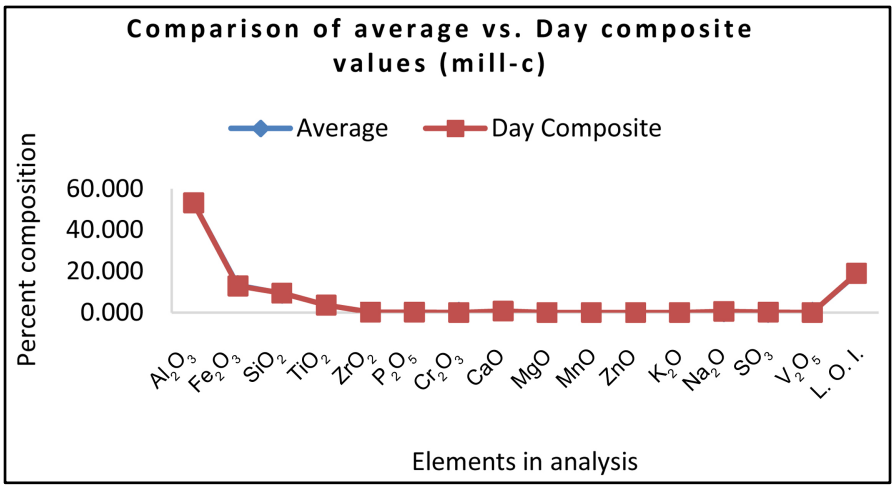


**Figure 7.** Comparison of average vs. day composite values for mill-C.

1) The composite sample represents all of the sub-samples and is thus more accurate and comprehensive.

2) Composite sampling reduces the load on the XRF and AFT machines.

3) Composite sampling reduces the consumption of chemicals and gas used in mill samples. In the present conditions, composite sampling helped reduced the use of X-ray Flux by 600 grams per month.

The protocol helps reduce working hours for the staff in the laboratory.

## 5. Limitations of This Study

The general limitations of composite sampling apply to the bauxite samples. The following are the limitations of this study:

1) The samples taken after the mill are sufficiently dry, however, in some cases if the moisture level is high, there are chances of the formation of agglomerates. These agglomerates can affect the homogeneity which is the core requirement of

a composite sample.

2) There are chances of reduction in the information of variability. When bauxite ore composition changes significantly, the reduction in information will affect the data quality objectives and statistical power requirements for hypothesis testing.

3) If the integrity of an individual sub-sample changes while making a composite sample, there are higher chances of false analysis. For example, high moisture content can affect the samples because the composite sample will be analyzed after 12 or 24 hours during which moisture will be reduced. Consideration must be taken while composting bauxite samples.

The sub-samples must be preserved until the processing of ore and final testing. This can result in some extra expenses which should be considered in the overall cost comparison.

## 6. Conclusion

This study was aimed at devising a composite sampling protocol for ore testing in the bauxite refining industry. The conventional method of sampling is linear and generates a large number of samples which not only overloads the laboratory equipment and staff but also increases the chances of false analysis and errors. It has been observed that the average composition of samples remains the same for bauxite ore and little variation does not impact the process parameters. The composite sampling technique proposed in this study helped to reduce the analysis load on the laboratory without affecting the quality of the analysis. Statistical techniques applied in this study such as paired t-tests and two-tailed tests have shown that the null hypothesis is valid, *i.e.* there is no significant difference between composite sample composition and average composition of samples. It can also be concluded that industries can improve the quality of the mill sample results to be more comprehensive and with less time and effort by taking a small amount of each sample to create a homogeneous sample from the samples of the three mills four times.

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## Author Contributions

We strongly encourage authors to include author contributions and recommend using CRediT for standardised contribution descriptions. Please refer to our general author guidelines for more information about authorship.

## Conflicts of Interest

There are no conflicts to declare.

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## Appendix A

**The comparison of average vs. composite composition values for Mill-A.**

| Mill A | | Sample Number | | | | | Average | Value of day composite | Difference d |
|---|---|---|---|---|---|---|---|---|---|
| | | 1 | 2 | 3 | 4 | 5 | | | |
| 1 | $Al_2O_3$ | 53.800 | 53.670 | 53.690 | 53.900 | 53.250 | **53.662** | **53.790** | **−0.128** |
| 2 | $Fe_2O_3$ | 14.020 | 13.580 | 13.170 | 14.080 | 15.060 | **13.982** | **13.640** | **0.342** |
| 3 | $SiO_2$ | 8.180 | 8.480 | 8.660 | 8.260 | 7.940 | **8.304** | **8.320** | **−0.016** |
| 4 | $TiO_2$ | 3.557 | 3.537 | 3.592 | 3.630 | 3.539 | **3.571** | **3.581** | **−0.010** |
| 5 | $ZrO_2$ | 0.121 | 0.125 | 0.132 | 0.127 | 0.131 | **0.127** | **0.128** | **−0.001** |
| 6 | $P_2O_5$ | 0.172 | 0.169 | 0.178 | 0.177 | 0.162 | **0.172** | **0.175** | **−0.003** |
| 7 | $Cr_2O_3$ | 0.050 | 0.049 | 0.048 | 0.051 | 0.049 | **0.049** | **0.049** | **0.000** |
| 8 | CaO | 0.689 | 0.748 | 0.676 | 0.715 | 0.832 | **0.732** | **0.714** | **0.018** |
| 9 | MgO | 0.074 | 0.069 | 0.068 | 0.074 | 0.083 | **0.074** | **0.714** | **−0.640** |
| 10 | MnO | 0.028 | 0.032 | 0.033 | 0.030 | 0.034 | **0.031** | **0.033** | **−0.002** |
| 11 | ZnO | 0.004 | 0.004 | 0.004 | 0.004 | 0.005 | **0.004** | **0.004** | **0.000** |
| 12 | $K_2O$ | 0.017 | 0.014 | 0.014 | 0.014 | 0.007 | **0.013** | **0.008** | **0.005** |
| 13 | $Na_2O$ | 0.335 | 0.305 | 0.295 | 0.161 | 0.100 | **0.239** | **0.163** | **0.076** |
| 14 | $SO_3$ | 0.114 | 0.122 | 0.106 | 0.110 | 0.108 | **0.112** | **0.112** | **0.000** |
| 15 | $V_2O_5$ | 0.092 | 0.091 | 0.092 | 0.093 | 0.091 | **0.092** | **0.092** | **0.000** |
| 16 | L.O.I | 18.740 | 19.000 | 19.240 | 18.580 | 18.610 | **18.834** | **19.120** | **−0.286** |
| | | | | | | | | **Mean Of d** | −0.040 |
| | | | | | | | | **SD of d** | 0.200942019 |
| | | | | | | | | **Test statistic** | −0.802296110 |

## Appendix B

**The comparison of average vs. composite composition values for Mill-B.**

| Mill B | | Sample Number | | | | | Average | Value of Day composite | Difference d |
|---|---|---|---|---|---|---|---|---|---|
| | | 1 | 2 | 3 | 4 | 5 | | | |
| 1 | $Al_2O_3$ | 52.900 | 52.950 | 52.700 | 52.980 | 53.170 | **52.940** | **52.820** | **0.120** |
| 2 | $Fe_2O_3$ | 13.900 | 13.670 | 12.960 | 12.890 | 13.420 | **13.368** | **13.110** | **0.258** |
| 3 | $SiO_2$ | 9.140 | 8.700 | 9.320 | 10.100 | 9.540 | **9.360** | **9.200** | **0.160** |
| 4 | $TiO_2$ | 3.558 | 3.516 | 3.507 | 3.610 | 3.570 | **3.552** | **3.524** | **0.028** |
| 5 | $ZrO_2$ | 0.127 | 0.124 | 0.123 | 0.132 | 0.119 | **0.125** | **0.123** | **0.002** |
| 6 | $P_2O_5$ | 0.172 | 0.168 | 0.162 | 0.172 | 0.170 | **0.169** | **0.168** | **0.001** |
| 7 | $Cr_2O_3$ | 0.049 | 0.049 | 0.047 | 0.051 | 0.051 | **0.049** | **0.048** | **0.001** |
| 8 | CaO | 0.748 | 0.737 | 0.741 | 0.775 | 0.769 | **0.754** | **0.744** | **0.010** |
| 9 | MgO | 0.088 | 0.080 | 0.074 | 0.090 | 0.081 | **0.083** | **0.077** | **0.005** |
| 10 | MnO | 0.037 | 0.035 | 0.030 | 0.029 | 0.033 | **0.033** | **0.031** | **0.002** |
| 11 | ZnO | 0.004 | 0.004 | 0.004 | 0.004 | 0.004 | **0.004** | **0.004** | **0.000** |
| 12 | $K_2O$ | 0.010 | 0.007 | 0.015 | 0.028 | 0.018 | **0.016** | **0.016** | **0.000** |
| 13 | $Na_2O$ | 0.219 | 0.243 | 0.388 | 0.795 | 0.453 | **0.420** | **0.362** | **0.058** |
| 14 | $SO_3$ | 0.101 | 0.101 | 0.103 | 0.139 | 0.107 | **0.110** | **0.112** | **−0.002** |
| 15 | $V_2O_5$ | 0.091 | 0.090 | 0.090 | 0.093 | 0.092 | **0.091** | **0.091** | **0.001** |
| 16 | L.O.I | 19.370 | 19.520 | 19.730 | 18.120 | 18.400 | **19.028** | **19.570** | **−0.542** |
| | | | | | | | | **Mean Of d** | 0.006 |
| | | | | | | | | **SD of d** | 0.16410186 |
| | | | | | | | | **Test statistic** | 0.154416287 |

## Appendix C

**The comparison of average vs. composite composition values for mill-C.**

| Mill C | | Sample Number | | | | | Average | Value of Day Composite | Difference d |
|---|---|---|---|---|---|---|---|---|---|
| | | 1 | 2 | 3 | 4 | 5 | | | |
| 1 | $Al_2O_3$ | 53.140 | 52.880 | 53.680 | 53.640 | 53.260 | 53.288 | 53.220 | 0.068 |
| 2 | $Fe_2O_3$ | 13.600 | 13.640 | 12.000 | 13.490 | 13.480 | **13.178** | **12.820** | **0.358** |
| 3 | $SiO_2$ | 8.920 | 9.090 | 10.010 | 9.270 | 9.140 | **9.358** | **9.370** | **−0.012** |
| 4 | $TiO_2$ | 3.549 | 3.501 | 3.684 | 3.564 | 3.576 | **3.578** | **3.577** | **0.001** |
| 5 | $ZrO_2$ | 0.123 | 0.129 | 0.128 | 0.128 | 0.125 | **0.127** | **0.125** | **0.002** |
| 6 | $P_2O_5$ | 0.165 | 0.159 | 0.175 | 0.170 | 0.174 | **0.169** | **0.169** | **0.000** |
| 7 | $Cr_2O_3$ | 0.049 | 0.049 | 0.050 | 0.049 | 0.049 | **0.049** | **0.051** | **−0.002** |
| 8 | CaO | 0.735 | 0.706 | 0.746 | 0.775 | 0.742 | **0.743** | **0.712** | **0.031** |
| 9 | MgO | 0.074 | 0.086 | 0.074 | 0.073 | 0.083 | **0.079** | **0.084** | **−0.005** |
| 10 | MnO | 0.033 | 0.032 | 0.024 | 0.032 | 0.033 | **0.031** | **0.029** | **0.001** |
| 11 | ZnO | 0.005 | 0.005 | 0.004 | 0.004 | 0.004 | **0.004** | **0.004** | **0.000** |
| 12 | $K_2O$ | 0.012 | 0.012 | 0.029 | 0.011 | 0.012 | **0.016** | **0.017** | **−0.001** |
| 13 | $Na_2O$ | 0.285 | 0.188 | 0.979 | 0.080 | 0.222 | **0.394** | **0.521** | **−0.127** |
| 14 | $SO_3$ | 0.112 | 0.090 | 0.136 | 0.100 | 0.101 | **0.110** | **0.116** | **−0.006** |
| 15 | $V_2O_5$ | 0.091 | 0.090 | 0.094 | 0.092 | 0.092 | **0.092** | **0.092** | **0.000** |
| 16 | L.O.I | 19.110 | 19.350 | 18.190 | 18.520 | 18.910 | **18.785** | **19.090** | **−0.305** |
| | | | | | | | | **Mean Of d** | 0.0003 |
| | | | | | | | | **SD of d** | 0.127383087 |
| | | | | | | | | **Test statistic** | 0.009289564 |