Alternative Method of Moisture Determination in Bauxite Residue from Press Filters

Alípio C. Junior[1], Luciano L. Santana[2], João V. Monteiro[2] and Éricles D. Moraes[3]
1. Process Engineer
2. Laboratory Analyst
3. Laboratory Manager
Hydro Alunorte, Barcarena, Brazil
Corresponding author: alipio.junior@hydro.com

Abstract

The moisture in bauxite residue is the main metric for filter performance and a key parameter in a variety of refinery key performance indicators (e.g. processing capacity, dried residue factor, alumina recovery, etc.). In day to day operations, the moisture must be regularly monitored to guarantee filtration stability and protect against other process disturbances. This parameter is typically monitored by thermogravimetric analysis to evaluate if the filters are delivering the residue according to design. Although this method provides good accuracy, it takes a long time to provide any information, delaying the operation's ability to respond to a process upset. This paper will present the results of the development of an alternative procedure, the infrared weighting method, which has reduced processing time by approximately 87 %, while preserving accuracy based on standard statistical methods. This reduction in processing time has allowed the operations team to respond to process disturbances in a timely manner, guaranteeing more stable operations.

Keywords: moisture, bauxite residue, infrared weighting, thermogravimetric analysis.

1. Introduction

The moisture control of bauxite residue from an alumina refinery is critical to several operational and strategic decisions. From this value, it is possible to estimate the capacity, consumption factors, scheduling labor on disposal, etc. Since August 16, the Hydro Alunorte Refinery started the commissioning phase of its new techolonogy for filtration of Bayer process residue, replacing the existing drum filters. The new Press Filters provide a number of advantages related to soda recovery and reduced cake moisture, which will be an important enabler for residue disposal safety. A good knowledge of the material properties is one of the important prerequisites to make good decisions about residue area operations [1].

The most common method to determine moisture in solids is thermo-gravimetric, which consists of weighing the wet solids, followed by drying under controlled conditions, and re-weighing after the drying period. The weight difference is considered to be a measure of free water (or 'moisture'). This techinique allows good accuracy, however, it does demand a delay (typically several hours or more) to provide results. This can cause delays in identifying and rectifying operational and equipment performance issues and/or decision-making.

For other applications, determining moisture using micro-wave weight scales has been adopted, and provides the moisture value in a much shorter time [2]. Determination of moisture content in freeze-dried solids is typically done by Karl Fischer (KF) titration, thermogravimetry (TG) or gas chromatography (GC). These methods are all time-consuming, invasive and destructive. Moreover, the sample to be analyzed may suffer contamination if the measurement is not carried out under appropriate conditions [3].

Until 2013, Hydro Alunorte Refinery used the gravimetric method for all solids in the process. Several studies were done to replace this method due to the differents characteristics of the solids. It was verified that infrared heating was interesting due to the speed of analysis, particularly for product quality of shipments. The method has been applied to hydrate solids, due to the advantages regarding the time of analysis.

The aim of this paper is to present an alternative to the standard thermo-gravimetric method with the minimum difference between the results.

2. Experimental

To evaluate the differences between both methods, the equipment from Hydro Alunorte's Lab were used. The samples used for this study were collected during to the daily routine. Subsamples were taken for analysis by both methods. Figures 1 and 2 show, the dryer used for the gravimetric method and the infrared scale, respectively.

Figure 1. Hot air blower dryers.

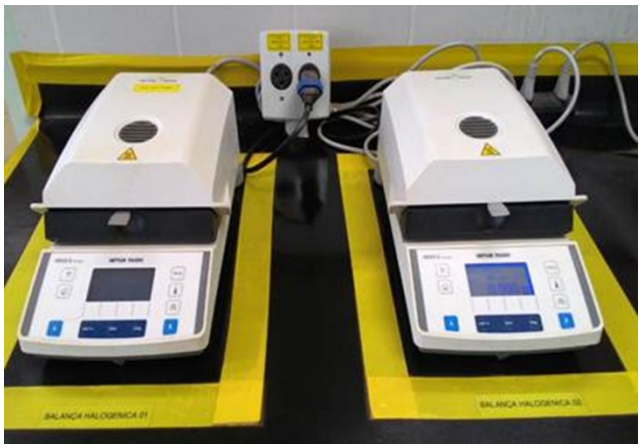

Figure 2. Infrared balance.

For both methods the mosture is calculated using the classical equation for moisture as follows:

$$\% M = \left(\frac{w_f - w_i}{w_f}\right) * 100$$

Where:

w_i Initial weight of sample, g

w_f Final weight of sample, g
M Moisture, %

3. Results and Discussions

After six months of using both methods to determine the moisture of residue from press filters, it was possible compare both methods and measures of their accuracy. Table 1 presents the results of this comparison.

Table 1. Moisture results by both methods.

Aug/16		Sep/16		Oct/16		Nov/16		Dec/16		Jan/17	
Dryer	Infrared Scale	Dryer	Infrared Scale	Dryer	Infrared Scale	Dryer	Infrared Scale	Dryer	Infrared Scale	Dryer	Infrared Scale
26,31	26,04	20,20	20,95	20,40	20,37	21,89	21,62	19,11	19,23	23,38	23,57
24,59	25,18	23,41	23,09	20,00	20,16	15,19	21,65	19,75	19,81	24,43	25,06
24,87	25,11	20,20	20,36	22,90	22,96	21,77	21,70	21,30	21,34	21,78	21,73
24,76	25,15	21,50	21,75	21,10	20,70	22,91	22,88	21,38	21,38	22,42	22,06
23,27	22,90	20,90	20,63	20,20	20,00	22,38	22,36	21,65	21,37	23,27	23,23
21,69	21,49	20,30	19,83	21,90	21,83	23,93	23,71	21,91	22,11	23,59	23,77
24,94	24,72	20,50	21,11	22,06	22,57	22,47	23,01	22,39	22,16	21,20	20,64
24,71	24,65	20,60	21,51	20,76	20,28	23,35	24,33	22,80	23,35	22,21	22,07
21,73	21,79	20,10	20,89	25,00	25,01	23,40	23,45	18,34	18,58	21,18	21,11
21,86	21,96	21,10	21,74	24,40	24,36	24,38	23,94	19,98	19,78	23,66	24,07
24,26	24,06	20,31	20,30	20,30	20,46	24,80	24,76	24,31	23,73	23,01	22,97
25,29	25,31	21,09	22,47	24,60	24,68	23,05	23,05	23,81	23,47	23,65	23,80
24,78	24,96	22,67	22,16	21,40	22,80	23,32	22,95	20,25	22,32	22,10	22,45
24,20	24,38	21,10	21,75	22,10	22,37	25,30	24,35	22,88	22,82	25,41	24,73
25,37	25,18	21,50	21,73	20,20	20,43	22,90	24,13	23,16	22,95	21,21	21,05
24,17	24,11	22,15	21,86	22,10	22,33	23,70	23,81	19,41	23,26	21,47	21,51
24,67	24,96	21,80	21,75	24,70	25,72	25,30	25,00	22,94	22,73	22,97	23,28
24,44	24,42	21,51	21,10	19,00	22,85	24,30	24,35	20,18	21,86	21,20	22,14
22,41	21,19	21,40	21,73	24,80	24,98	19,30	24,41	19,22	21,64	22,70	22,05
23,33	25,56	24,80	24,85	22,70	22,66	22,06	22,10	19,35	19,63	21,80	23,25
24,44	25,01	20,74	21,05	22,38	22,14	22,82	22,91	22,86	23,55	21,90	22,86
24,41	24,43	20,53	20,68	23,80	23,67	22,15	22,79	23,46	23,61	19,48	20,00
24,52	24,27	20,99	21,31	23,40	22,71	23,38	23,31	24,57	24,45	19,64	29,70
23,25	22,35	21,43	21,40	23,00	22,59	23,50	23,30	24,22	24,27	20,02	29,30
22,97	22,15	21,35	21,58	24,90	25,03	25,07	22,14	24,85	24,27	21,30	21,04
23,71	23,90	21,20	22,13	19,34	23,29	23,80	23,51	22,92	23,60	22,30	21,87
20,60	20,51	21,40	22,30	21,84	24,86	21,90	24,66	19,56	18,87	19,80	20,46
22,01	22,07	21,20	21,40	22,38	22,16	21,65	21,90	23,22	23,98	22,80	23,22
21,42	21,46	20,70	20,60	21,03	21,20	23,40	22,80	20,44	20,42	19,70	20,83
21,12	21,23	22,02	22,85	24,47	21,33	21,42	21,50	23,80	23,49	23,60	23,90
20,20	20,84	20,40	25,16	24,10	23,77	22,33	22,25	22,93	19,09	21,00	21,10
20,20	21,36	27,10	26,10	24,39	23,89	21,37	23,76	19,04	21,59	22,70	22,91
21,90	22,59	21,60	21,86	23,96	23,75	23,74	21,69	21,96	22,96	21,60	21,77
21,32	21,57	20,80	21,20	25,98	25,98	24,04	24,00	23,27	23,08	21,70	21,85
22,26	22,11	21,40	21,20	25,00	24,87	24,94	22,82	19,16	19,45	20,62	20,53
22,00	22,52	20,20	20,10	21,96	22,03	21,87	21,96	23,61	23,27	23,13	23,33
21,44	21,92	22,30	22,37	20,33	22,08	23,19	24,62	19,10	19,30	20,80	21,52
22,11	22,12	21,40	21,50	21,13	21,51	23,31	23,20	18,95	19,20	20,17	20,30
23,09	23,24	22,67	22,50	23,81	23,99	23,53	23,46	23,17	23,25	23,00	22,78
19,95	19,50	22,39	22,20	20,17	20,18	21,80	21,50	20,06	20,30	22,40	22,03
21,82	21,49	22,68	22,50	24,62	25,03	21,11	21,57	18,57	18,23	22,30	21,68
20,46	21,12	20,30	20,16	21,45	21,19	24,26	24,15	18,92	18,48	21,80	21,53
19,30	20,61	21,30	21,25	21,40	21,20	22,43	22,73	18,76	18,42	21,90	22,86
20,15	19,42	22,10	23,32	25,20	24,60	20,43	20,46	20,63	21,03	20,74	21,40
20,34	19,34	22,12	21,69	21,47	21,10	22,81	22,35	18,95	19,39	21,48	20,50
21,56	21,08	22,48	21,49	21,33	21,54	24,29	24,14	19,28	19,19	20,90	20,82
20,23	20,42	22,25	22,49	21,46	21,87	21,40	21,78	18,95	18,81	22,60	22,53

Firstly, the time to deliver the results was evaluated and Figure 3 below shows the time of analysis for both methods for the same residue from 7 press filters in operation.

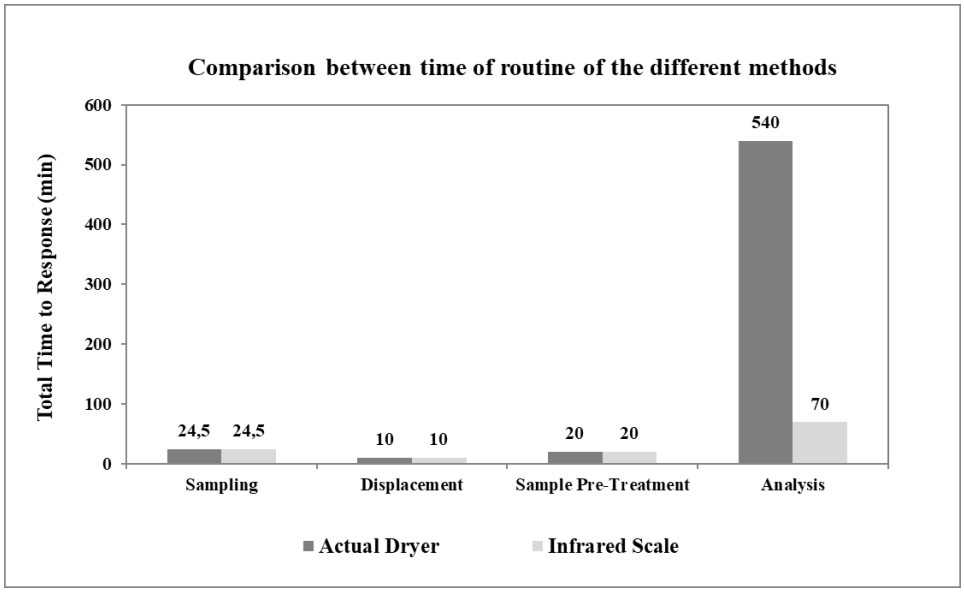

Figure 3. Comparison between routine time of the two moisture methods.

As the results presented below confirm, the infrared balance gives a reduction of over 87 % in total response time. Since this comparison was for the moisture determination step, only the time of analysis is changed in this study and the rest of steps starting from sampling remain the same.

This improved speed to analysis, is however, not enough by itself to justify a change of method. Any method has also to provide acceptable accuracy and precision [4, 5], and must consider charateristics and variability of the samples. To confirm the infrared balance method met these requirements, a statistical analysis was undertaken. In this case, for dependent sampling (i.e, results from differents methods with the same sample source), the most applicable analysis to perform was considered to be a Paired-t Hypothesis Test [6]. As a preliminary observation, Figure 4 shows the Box – Plot of all results to identify any visual deviation or outliers that could impact the monthly averages of moistures from both methods.

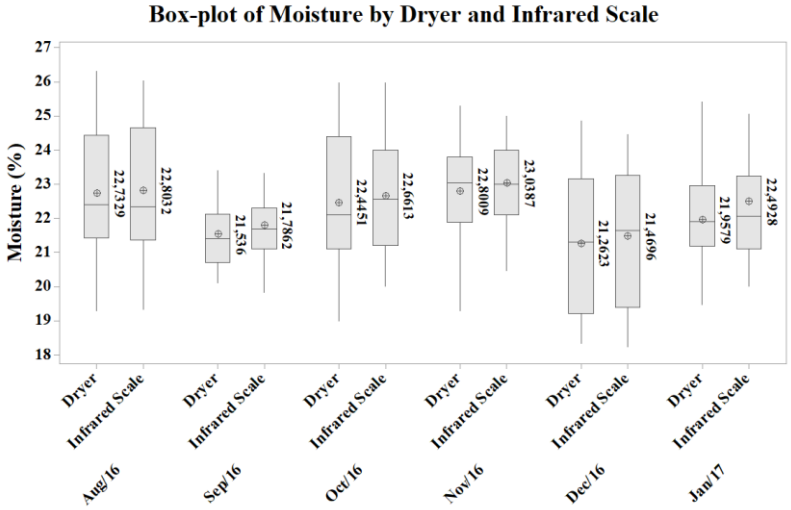

Figure 4. Box-plot of moisture by dryer and infrared balance.

If we take into account only the monthly average values during the study, it is easy conclude that both methods agree acceptably well, and that they mey be interchangable. Neverthless, it is

true only if the p-value for the Paired test is higher than 0.05. Table 2 presents the p-value for the hypothesis test for the monthly averages.

Table 2. Paired-t hypothesis test (p-values)

Period	p-value
Aug/16	0.423
Sep/16	0.074
Oct/16	0.251
Nov/16	0.468
Dec/16	0.291
Jan/17	0.025

Excepting the Jan/17 database, all the months presented acceptable p-values (according to equality) for the moisture averages no matter what the variability. The differences with the other months was due to the re-start of the press filter plant after mechanical improvements were done, and did not represent the normal conditions of the test.

Another observation is regarding the range of moisture that can be accepted in the infrared scale. As reported, Hydro Alunorte used this methodology for hydrate and other materials like bauxite. For example, for sbauxite amples from drum filters which have high moisture, the accuracy is impacted due to the liquor condensation in the chamber of the balance, and this can generate disturbances. Samples from the press filters have lower moisture than from the drum filters, and this problems does not occur with these samples. Figue 5 shows the comparison between typical moistures results of materials used or generated from the Bayer process.

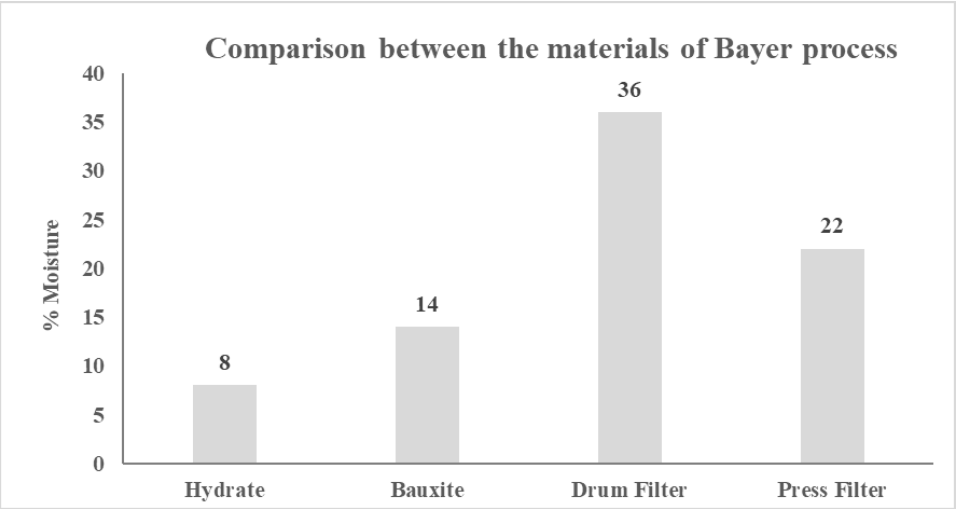

Figure 5. Comparison between Bayer process materials.

4. Conclusions

An evaluation of an alternative method for measuring the moisture of Bayer residue was achieved. The evaluation confirms the reduction in time of analysis is significant (over the 87 % actual method), providing an opportunity to optimize the operation of the press filter, allowing as many adjustments as necessary. Currently, this is not possible because the samples take 8 hours to process, close to the shift time for the same operations team. Statistical tests were done and the results indicate that the monthly averages are equal for both methods under varying conditions. This difference is due to only the normal oscillation of the process. The infrared

balance can be used with a 95 % confidence level. This is a great result for Hydro Alunorte, considering there are 7 press filters in operation.

5. References

1. Jianhua Zhao, Frank Meissener. Experimental investigation of moisture properties of historic building material with hydrophobization treatment, *11th Nordic Symposium on Building Physics,* NSB2017*,* (2017), 11-14.
2. S. R. de T. Valentini, M. F. P. Castro, F. H. M. Almeida, Determination of moisture of corn using micro-wave device, *Food Science and Technology.* Vol. 18, (1998), 237-240.
3. C. R. Muzzio, N. G. Dini, L. D. Simionato, Determination of moisture content in lyophilized mannitol through intact glass vials using NIR micro-spectrometers, *Brazilian Journal of Pharmaceutical Sciences* Vol.47 n.2 (2011).
4. G. E. P. Box, W. G. Hunter, J. S. Hunter, Statistics for Experimenters: An Introduction to Design, Data Analysis and model building. *John Wiley and Sons*. ISBN 0-471-09315-7 (1978)
5. W. W. Hines, D. C. Montgomery, D. M. Goldsman, C. M. Borror, Probability and Statistics in Engineering. 4th Edition. *John Wiley and Sons* ISBN 85-216-1474-8 (2006)
6. Minitab StatGuide Help Summary 18. Keywords Searching: Paired-t test. Acess in Nov/17.