BULK DENSITY MEASUREMENT OF REFRACTORY RAW MATERIALS – FASTER AND BETTER WITH A SPIN DRYER

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ABSTRACT

In order to determine the bulk density of refractory raw materials, the so-called water method following the Archimedes principle is normally used. This is where the effect of water displacement on the weight of the sample is used to determine the bulk volume of the sample grains. During this test procedure, the surface of water infiltrated sample grains must be dried with a wet towel. Experience shows, that this drying step is the main root cause for variation in reproducibility of results and even repeatability of tests. A new spin dryer (centrifuge) was developed and introduced to automate this surface drying step, and is now included as a new method in ISO 8840:2021. The paper discusses the improvement of measurement with the new approach and industrial experiences from two big industrial players in the raw material business.

Keywords: Bulk density, centrifuge, drying, infiltration

INTRODUCTION

The bulk density is an important property for refractory raw materials and the apparent (open) porosity is also an essential part of the specification. It is measured frequently as in-process control in refractory aggregate production. During tabular production, Almatis performs about 23,000 bulk density measurements per year over its five sites in the USA, The Netherlands, India, China, and Japan.

The different standard methods for determination of bulk density are briefly and comprehensively described by Lorenzoni [1] and will not be discussed in detail here. Popular methods such as DIN-EN 993-18 [2] and ASTM C357-07 [3] include sample volume determination by weighing the immersed sample in water following the Archimedes principle with buoyancy related to the volume of the immersed body. An important part of that procedure is weighing the water - infiltrated sample without excess water on the surface of the sample. This would lead to grain bulk density appearing lower. However, undesired removal of water from open pores at the grain surface would give apparently higher bulk density.

Therefore a wet cotton towel is wrung and then used to manually blot the grains to dry the surface until the wet sheen on the grains has disappeared (figure 1). It is clear that this procedure adds variability to the results because of slight differences between operators and their subjective judgements. This has been proven by multiple round robin tests both internally and externally at Almatis and RHI Magnesita. A bulk density variation of +/- 0.02g/cm³ is commonly accepted for tabular alumina or other refractory aggregate grain density measurements.

RHI Magnesita has developed a new method for this critical step in grain bulk density measurement. This replaces the manual and subjective method by the use of a centrifuge (spin dryer), to give an automated and more reproducible process [1]. It has been included in ISO 8840:2021 [4]. Almatis, at first, tested the centrifuge and then purchased six machines from RHI Magnesita for use in all worldwide tabular alumina sinter plants. The experiences gained during qualification and implementation of the centrifuge method in Almatis are described as follows.

Fig. 1: Surface drying of immersed tabular alumina grains by blotting with wet towel (cotton cloth).

CENTRIFUGE METHOD

Centrifuge for surface drying

Fig. 2 shows the centrifuge with five baskets for carrying the immersed, water-infiltrated grain samples. If less than five samples are to be tested, dummies should be used in the other baskets to ensure balance during the spinning process. The rotation speed and time can be adjusted based on calibration experiments depending on the requirements of specific materials. However, Almatis found the setting of 680 RPM for 90 secs as recommended by RHI Magnesita was also suitable for tabular alumina. This figure was defined by RHI Magnesita after an extended series of tests over a broad range of raw materials but with a particular focus on magnesia.

For each bulk density measurement Almatis uses three 90 g tabular alumina samples, crushed and sized to 5 - 8 mm. This is coarser than the figure in ISO 8840:2021 where 2 – 5.6 mm or 3 – 4 mm is recommended for the centrifuge method. Tabular alumina is a very hard material and lab-crushing of the 19 mm diameter sintered balls down to 3 – 4 mm for bulk density testing would be inappropriately cumbersome for the high number of samples in daily processing in the plants. The high homogeneity of tabular alumina as a granulated and sintered product allows that deviation from the norm.

Fig. 3 shows the bulk density of tabular alumina samples comparing surface drying with the towel with the centrifuge method. The correlation found in this and multiple other tests was very good and triggered the decision to equip all tabular alumina sintering plants within Almatis with centrifuges for bulk density measurement. With regard to nomenclature some clarification is needed here. Historically, bulk density of tabular alumina was always referred to as bulk specific gravity (BSG) which strictly speaking is a unitless

figure relating the gravity to that of water with 1 g/cm³. Nevertheless, BSG was and is used with g/cm³ as unit in data sheets and all technical communication.
Meanwhile the centrifuge method was qualified and implemented as a replacement of the towel method for drying in Almatis sinter plants.

Fig. 2: Centrifuge (spin dryer) built at the RHI Magnesita Technology Center Leoben, Austria.

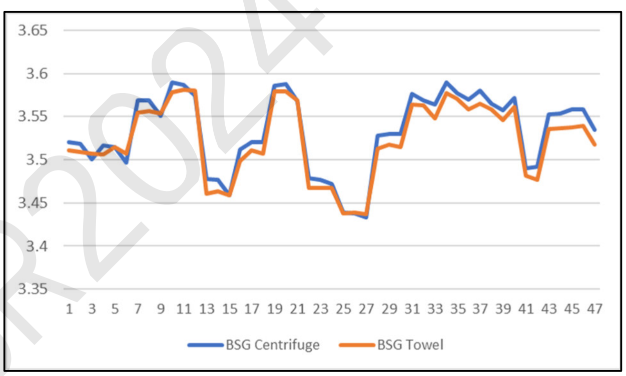


Fig. 3: Bulk Specific Gravity (BSG) of tabular alumina - centrifuge vs. towel drying.

Centrifuge for grain infiltration

The centrifuge provides another interesting opportunity for improving the bulk density measurement of raw material aggregates. RHI Magnesita has successfully tested grain infiltration with the centrifuge. In the standard methods, the sample is infiltrated with water either by the vacuum method [2] or by boiling in water [3]. Both procedures require a minimum of 45 minutes to achieve the required infiltration of the grains. At Almatis, boiling the sample in water was the common way for infiltration. When the dry sample is put into a plastic container with water and then into the centrifuge basket (figure 4), the spinning can be used to infiltrate the sample grains. Here, rotation speed and time have to be increased when compared to the surface drying step later in the procedure. For Almatis tabular alumina samples, 880 RPM for 220 seconds was established as the standard setting for achieving stable infiltration results. This is significantly faster than the boiling method.
With this new approach, using the centrifuge for infiltration and drying the surface, results from bulk density measurement are already available after about 15 minutes. This compares to about 90 minutes using the old approach. It also means that the lab analysts can perform the testing in one operation instead of having interruptions, waiting for the infiltration to be finished.

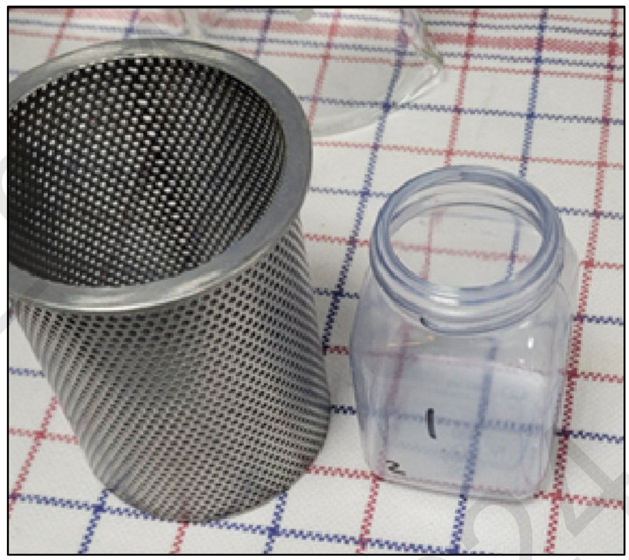


Fig. 4: Basket with plastic container for infiltration of tabular grains by centrifuge.

Results from new method with tabular alumina

Test series were started in different locations to compare results using the new method (centrifuge for infiltration and surface drying) with the old method (boiling in water and surface drying with wet towel). Fig. 5 shows good correlation of BSG between both methods from a test series of 48 samples and six different analysts at Almatis Rotterdam. There were only slight differences in apparent porosity (AP), and water absorption (WA). Fig. 6 shows box plots from the same comparison at Almatis Benton, AR with 30 samples and 4 different analysts. In addition, correlation for BSG is good here, and differences in AP and WA are small.
Based on these encouraging results, a global Gage R&R study was performed in the five tabular alumina sintering plants. 29 unique different samples covering a range of bulk density and including samples with bulk density clearly below specification limit of tabular alumina were tested in the five different quality laboratories by 21 different operators with the classical (boiling and towel drying) and the new method (centrifuge spinning for infiltration and drying). Fig. 7 to 9 show measurement results from the Gage R&R test for the specified tabular alumina properties bulk density (BSG), apparent

porosity (AP), and water absorption (WA) with both methods at the different sites. In general, variation is higher for very low bulk density samples with values clearly out of the specification range for tabular alumina (min. 3.50 g/cm³). For the other samples, results are well aligned.

Fig. 10 shows the result from the Total Gage R&R test as % Study Variance. The general rules to determine the capability of the system are: <10% acceptable; 10-30% marginal; >30% unacceptable. With total Gage R&R contribution between 14 and 24%, the variance is within acceptable limits due to tool type and application. In general, the variance for the centrifuge method is a bit lower than for the towel method. Based on the positive results from the Gage R&R test the new method has become the standard within Almatis.

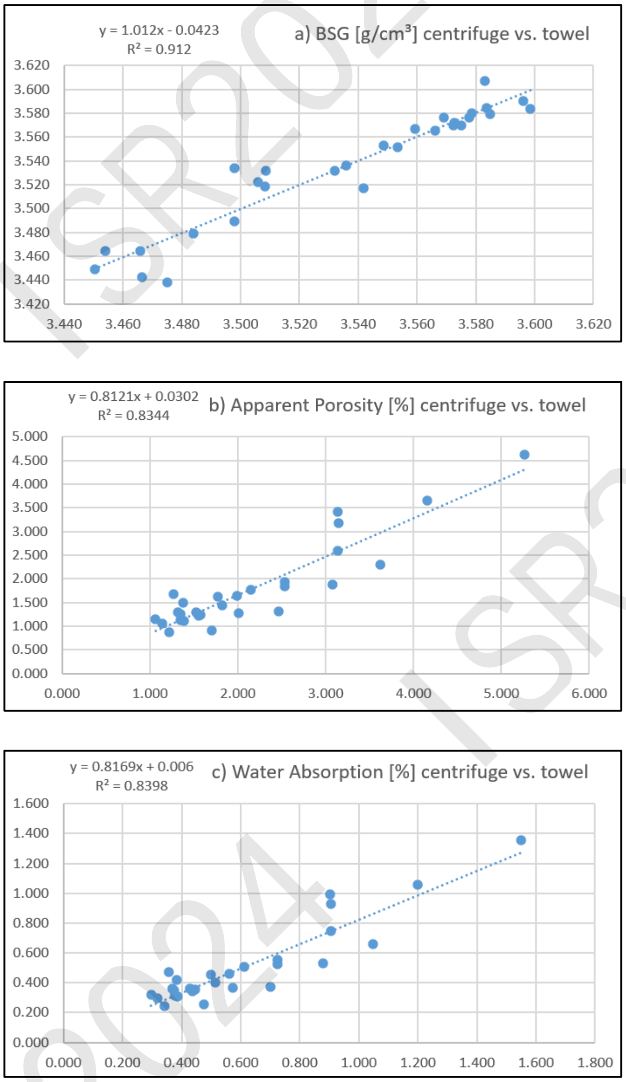


Fig. 5: Correlation of BSG, AP, and WA - centrifuge vs. towel method at Almatis Rotterdam, The Netherlands.

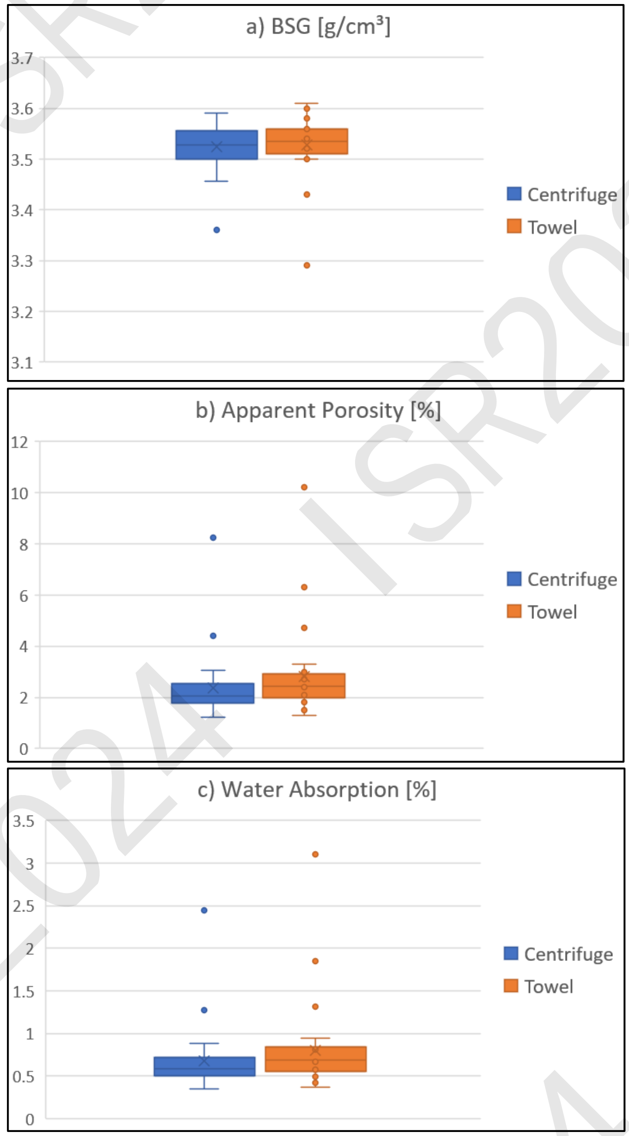
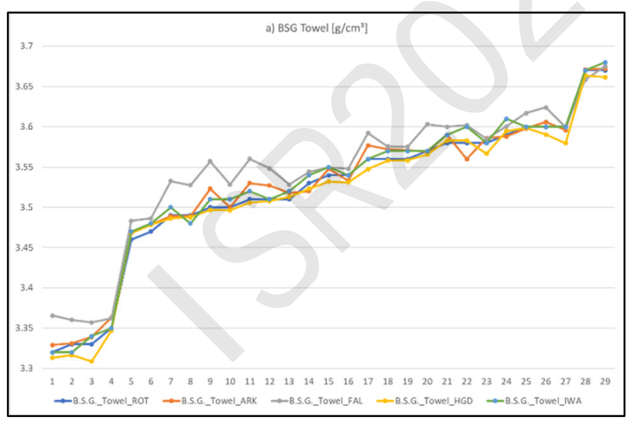


Fig. 6: Box plot of BSG, AP, and WA results Almatis Benton, USA.



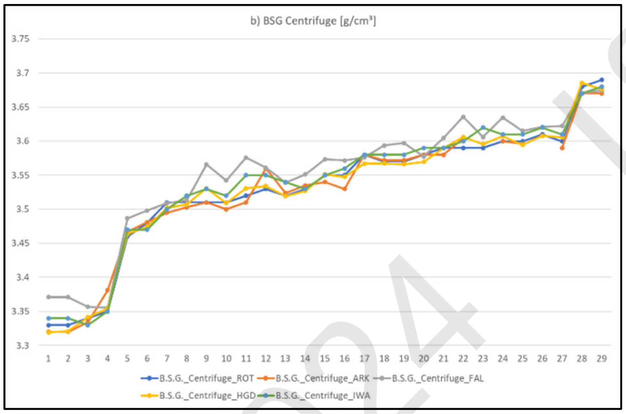
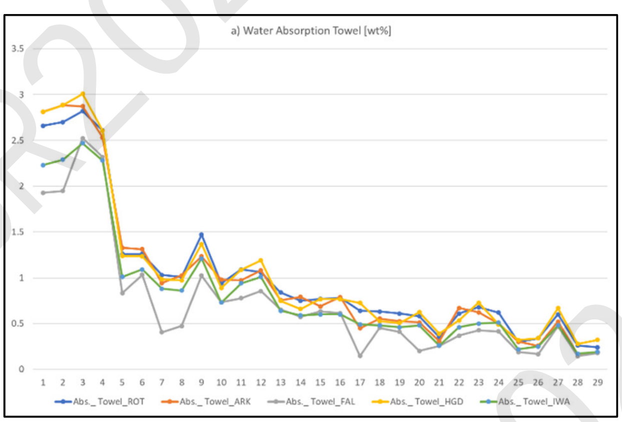


Fig. 7: Bulk density towel (a) and centrifuge (b) results from Gage R&R.

Fig. 8: Apparent porosity towel (a) and centrifuge (b) results from Gage R&R.



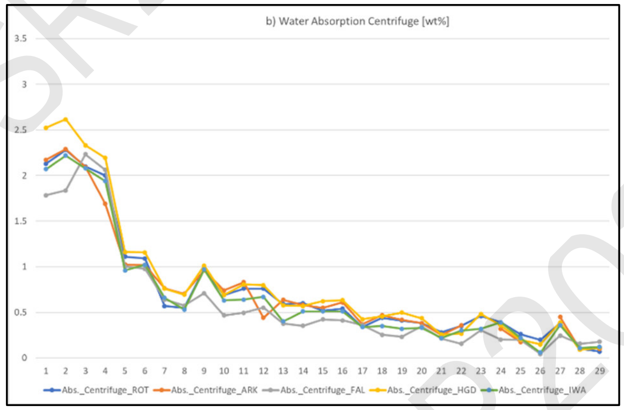


Fig. 9: Water absorption towel (a) and centrifuge (b) results from Gage R&R.

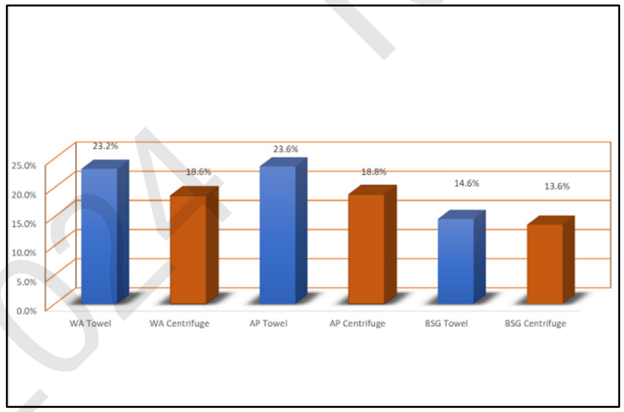


Fig. 10: % Study Variance results of Total Gage R&R. General rules to determine the capability of the system: <10% acceptable; 10-30% marginal; >30% unacceptable.

CONCLUSION

The centrifuge enables automation of the surface drying step in bulk density measurement based on the water method. It can help to reduce human element variation in lab work. This will improve repeatability and reproducibility in bulk density measurement in the laboratory and between different laboratories. The extension of centrifuge usage for the infiltration of the grains replacing the vacuum or boiling method leads to significantly reduced times for the entire measurement. The new approach was established as the future standard within Almatis.

REFERENCES

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[2] DIN EN 993-18 - 2002 Methods of test for dense shaped refractory products - Part 18: Determination of bulk density of granular materials by the water method with vacuum

[3] ASTM C357-07 Standard test method for bulk density of granular refractory materials

[4] ISO 8840:2021 Refractory materials – Determination of bulk density of granular materials (grain density)