J. Palmer[1], D. Safonov[2], A. Häkkinen[3], B. Ekberg[4], A. Kraslawski[5]
Larox Corporation (1, 4), Saint-Petersburg State Mining Institute (2),Lappeenranta University of Technology (3, 5)
Jason.Palmer@larox.com; safonov.dmitry@mail.ru; Antti.Hakkinen@lut.fi

Trends in test filtration responding decrease of sample size of iron concentrates

As mining technology becomes more mature and with the increasing cost of pilot testing before developing a new mine, there has been a steady decline in the quantity of concentrate sample available to make process testwork. The suppliers of filtration equipment and research universities actively develop new equipment and testing methodologies with the objective of being able to determine the optimum filtration technology and the parameters for sizing full scale equipment with a minimum sample size. With smaller sample sizes the test scale diminishes from pilot scale to bench scale and the small equipment size has implications for scale-up. Redesigning the equipment to simulate full scale equipment rather than match filtration theory has led to some interesting developments. In addition comparisons between bench scale equipment and production units help identify the limitations of bench scale testing. The nature of iron concentrate filtration is such that it is unreliable to estimate filterability theoretically and Lappeenranta University of Technology (Finland, Lappeenranta) has been investigating different testing methodologies to be able to test the feasibility of filtration from samples as small as a few grams. While not optimizing the filtration technology these methodologies provide an adequate level of accuracy for scoping studies.

The amount of iron concentrate required to conduct a filtration test campaign is dependent upon the equipment size and the number of tests conducted. The smallest equipment size commonly used today requires a minimum of 2 kg for a vacuum filtration test and small piston type pressure filter test. At this scale filtration rate and cake properties can be established with reasonable accuracy. Vacuum filter tests are generally more accurate as a larger area can be used for the same cake weight. Vacuum filter tests are generally conducted using a Buchner funnel with an area of 0.01 m^2 to simulate horizontal belt filters or a dip test disc with an area of 0.01 m^2 to simulate disc or drum filters. Piston type pressure filters with an effective area of 0.005 m^2 to 0,0025 m^2 are used to simulate pressure filters. Pressure filtration at small scale suffers from edge leakage and as the unit size drops below 0.01 m^2 the results tend to be less repeatable and the edge leakages make dry blow and cake wash results less reliable. As iron industry has been producing smaller samples at later stages of development filtration equipment suppliers have worked to develop methodology to reduce the sample size while retaining the testing accuracy. The focus of these developments has been in three directions:

- Improved data collection
- Reduction in unit size
- Developing new test methodologies

Data collection

Good data collection is quite mature and test scale equipment generally has better measurement systems than some full scale process equipment. While described briefly here improving data collection was not considered to be an area of focus to help reduce sample sizes.

Slurry properties. Solids density, liquid density, viscosity, temperature, specific surface area and particle size are parameters that effect filtration performance and can all be accurately defined prior to filtration testing.

Cake properties. Weight, density, void fraction, moisture and thickness are required to quantify filtration performance and can all be accurately determined after filtration tests.

Time dependent variables. While physical properties can be easily measured some parameters such as; filtrate volume vary with time during filtration testing. Improved analysis and reduced testing can be achieved when these parameters are measured and electronically recorded. Time dependent variables include filtrate volume, cake thickness, drying air flow, vacuum (thinned air) flow, wash water, filtrate concentration and pressure.

Test equipment and scale

Test equipment design for filtration test work has produced many variations. Traditionally laboratory scale equipment has tended to be used for simulation of filtration theory whereas pilot scale equipment has been designed to simulate full scale production equipment performance. When reducing pilot scale equipment to reduce sample sizes the use of existing pilot scale equipment gave accurate filtration simulation at the expense of scale up.

Vacuum filtration. Laboratory scale vacuum filtration equipment does not suffer as many limitations as laboratory scale pressure filtration and accurate results can readily be achieved from small scale equipment. Filtration rate and cake moisture can be accurately be defined at almost any scale, however vacuum consumption and cake washing accuracy decreases rapidly with areas smaller than 0.01 m^2. This reduction in accuracy is due to leakages at the edge of the filtration area. These leakages vary with sample type and are not easily separated from the filtration performance. While laboratory scale equipment is accurate their full scale counterparts are less perfect and lab scale results generally require derating when used to size full scale equipment. Published research

work [5] relating leaf test data with either pilot scale or full scale operation, as presented in Fig. 1, suggest that an overestimation is obtained using the leaf test. These differences arise from deficiencies such as media blinding, agitation intensity and cake fall off, cake cracking, equipments leaks and consequent vacuum reduction and sedimentation in the filter bath. In a recent study [7] on the improvement of the sizing accuracy of Ceramec disc filters looked at the techniques required to improve reliability of testing and reduce the differences between lab and full scale equipment performance. The results shown in Fig. 2 show a good correlation on cake moisture. As with the results of Osborne [5] capacities of the test equipment are in many cases higher than that of the full scale equipment.

Pressure filtration. At pilot scale pressure filtration simulates production equipment well. Part of the solution lies in the combination of ancillary equipment used to facilitate the filtration. As unit size decreases pumps of a suitable scale are no longer an option. Lab scale equipment does not simulate pumping depending upon a variable volume and atmospheric slurry addition. Similar effects are seen in drying air and wash water where the supply is instantaneous. These limitation results in piston type lab equipment being ideal for filtration theoretical simulation but poor at full scale equipment simulation. As with vacuum filters the result is that lab scale test equipment tends to overestimate the achievable capacity and the results must be derated to account for full scale equipment performance imitations. These differences arise from deficiencies such as media blinding, the lag in introducing slurry to the filter and the time required to achieve constant pressure, manifold draining and steps that are required for cake handling, cake cracking and consequent increase in compressed air requirements.

As size diminishes full scale replication declines, but as size diminishes the accuracy of cake specific resistance improves, therefore it is reasonable to conclude that provided the methodology for derating to account for real world inefficiencies is well understood that very small filter equipment can be used to estimate filtration performance by measuring cake specific resistance. As any purely theoretical approach to date has had quite low levels of confidence this may improve early estimations of filterability.

Reduction of sample size by using the Blaine air permeability method

Background. This chapter introduces the main parts of an experimental study that was carried out in laboratory scale to find out if the information obtained by using a simple Blaine permeability meter could be utilized for predicting the filtration characteristics of powder samples including iron concentrate powder. The Blaine permeability meter is an instrument that is used routinely for example in the cement industry for measuring the specific surface area (i.e. the fineness) of cement powders. Currently this method is used as an effective tool both for laboratory work as well as for production control for a wide range of powders [2].

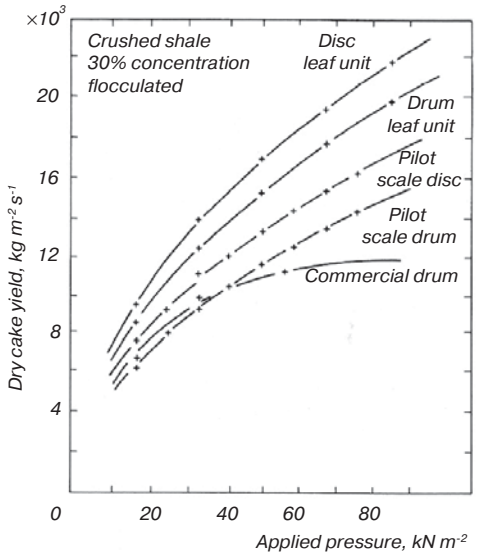

Fig. 1. Comparison curves for leaf, pilot scale and commercial scale rotary vacuum filters [5]

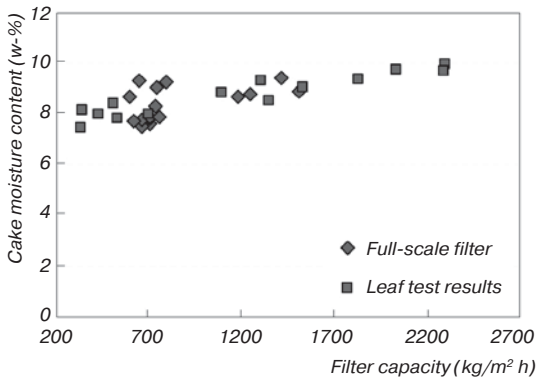

Fig. 2. Comparison between the results obtained from the leaf tests and the values measured from full-scale filters at process test application 1 [7]

The Blaine method is based on measuring the time that is required for a certain volume of air to pass through a constant layer of the investigated solid powder. The passage time can be used for estimating the specific surface area of the solids expressed as total surface area in square centimeters per gram of powder. These kinds of tests can be carried out with a cheap and easy-to-operate Blaine air-permeability apparatus and the results can be obtained fairly quickly and by using very small samples (less than 100 g of solids). Despite the obvious advantages, this method of material characterization has not been widely applied in the filtration industry. Potgieter and Strydom [6] have reported that the main weaknesses of the Blaine method are that it is normally not very accurate and it may become too unreliable in those cases where the surface areas of the samples are greater than 500 m^2/kg.

It is well-known that the filtration characteristics of any suspension are determined by the properties of both solids and the liquid. The most important characteristic of a filter cake from the theoretical point of view is the average specific filter cake resistance. Filter cake resistance determines not only the flow rate of filtrate through the cake during the

actual solid/liquid separation step but it also influences the flow rate of air trough the cake during the air blowing stage and the flow rate of wash liquid during the cake washing. Probably the most widely applied model for relating the flow rate of fluid through a filter cake with the properties of the particles making up the cake is the well-known Kozeny-Carman equation:

$$u = \frac{\varepsilon^3}{(1-\varepsilon)^2 K S_0^2} \cdot \frac{\Delta p}{\mu L}, \quad (1)$$

where u is the superficial velocity of the fluid, ε is the porosity of the cake, K is the Kozeny constant, S_0 is the specific surface area of particles ($m^2\ m^{-3}$), Δp is the applied pressure difference, μ is the dynamic viscosity of the fluid and L is the thickness of the cake. The Kozeny constant K depends on the structure of the cake and it is typically assumed to be 5.0 for randomly packed incompressible beds [3, 8]. Equation (1) can be further modified and written in terms of average specific cake resistance α_{av} to give:

$$\alpha_{av} = \frac{5\rho_s SSA^2(1-\varepsilon_{av})}{\varepsilon_{av}^3}, \quad (2)$$

ρ_s is the density of solids, SSA is the specific surface area of the particles (m^2kg^{-1}), α_{av} is the average specific cake resistance and ε_{av} is the average porosity of the filter cake. Equation (2) now clearly shows that the relationship between the average specific cake resistance and specific surface area should be quadratic if the cake porosity remains constant. It should be pointed out here that the use of equations (1) and (2) for actually predicting the values of u or α_{av} is strictly limited to incompressible filter cakes since in the case of compressible cakes, the applied pressure has a direct influence on the packing structure of the bed thus influencing also several other parameters in the equations [4].

Air permeability tests. The objective of the experiments carried out in this study was to investigate if obvious correlations existed between the specific surface areas measured with the Blaine method and the average specific resistances of the filter cakes formed in laboratory scale pressure filtration tests. The main characteristics of the eight different model powders compared in the tests are summarized in Table 1.

The laboratory devices utilized in this study included the Blaine meter for estimating the specific surface area of the dry powder samples and the pressure Nutsche filter for determination of the average specific resistances of the filter cakes. The Blaine air permeability apparatus and the accessories were products of Humboldt Mfg. Co. (USA) and all the measurements were performed according to the standard test method provided by the American Society for Testing and Materials (ASTM) [1]. The main parts of the equipment used for the air permeability measurements are presented in Fig. 3. The value measured by the permeability apparatus is the time t that a certain volume of air requires to pass through the packed bed. The measurement consists of two steps – preparation of the powder bed and performing of the air permeability test. An accurately weighed mass of the tested powder was first poured into the cell to make a packed bed with the desired porosity. The permeability cell containing the prepared packed bed was then attached to the manometer tube and the measurements were performed at least three times for each material. Each set included air permeability tests with five different packed beds having different porosities. The value that was measured during the tests was the time that was needed for the manometer liquid level to drop from the second mark from the top (mark *b* in Fig. 3) to the third mark from the top (mark *c* in Fig. 3). The specific surface areas for the different samples could then be determined from the measured times and bed porosities by using the calculation procedure introduced by ASTM [1].

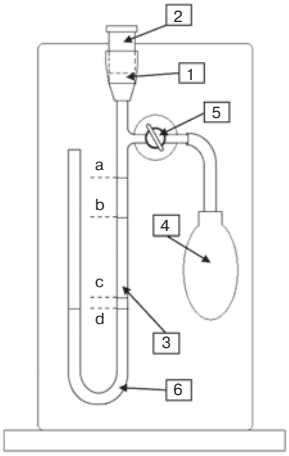

Fig. 3. Schematic view of the Blaine air permeability apparatus. Numbers and letters mean: 1 – permeability cell; 2 – plunger; 3 – U-tube manometer; 4 – rubber bulb; 5 – airtight glass valve; 6 – manometer liquid; a, b, c, d – marks put on U-tube manometer

Pressure filtration tests. The aqueous model suspensions which were made from each of the studied powders were used for the constant pressure filtration tests which were performed by using a laboratory scale Nutsche filter. A schematic view of the filtration system is shown in Fig. 4. The test suspensions for the filtration experiments were prepared from each of the model powders introduced in Table 1 by mixing a certain amount of those in water and homogenizing the mixtures properly. The concentration of solids in most test suspensions was approximately 12 vol-%. The tests were performed by taking a slurry

Table 1. Main characteristics of the powders compared in the tests

#	Material	Origin	Solid density (kg m^{-3})
1	Coal	From an industrial process	1554
2	Ground $CaCO_3$	Commercial bulk material	2593
3	Apatite	From an industrial process	3191
4	Magnetite (Fe_3O_4)	From an industrial process	4772
5	Fe concentrate	From an industrial process	3947
6	Zn concentrate	From an industrial process	4137
7	Cu concentrate	From an industrial process	3977
8	Pyrite	From an industrial process	4821

volume of about 300 ml from the mixing tank and pouring it into the filter chamber. To avoid quick settling of the solids the filter was pressurized immediately after the slurry had been introduced by using nitrogen at pressure of 2.0 bar which caused the filtration to start. The mass of the filtrate accumulated onto the scale was constantly recorded by the PC. At the end of each filtration test the cake thickness was measured and the cake was discharged from the chamber. The mass of the cake was weighed and the dried in an oven with a constant temperature of 105 °C. After 24 h the weight of the cake was measured again to determine the moisture content. The parameter that was used for describing the filtration characteristics of the examined suspensions was the average specific cake resistance, which was determined from the plots of t/V vs. V using the classical constant pressure filtration equation [9].

Results. The specific surface areas determined by using the Blaine air permeability method and the average specific filter cakes resistances at pressure difference of 2.0 bar for all of the studied powders are presented in Fig. 5. It can be noticed from the experimental data presented in Figure 5 that quite good correlations between the Blaine index and the average specific filter cake resistances were obtained for most samples. Fig. 5 shows a trendline that has been drawn by using a basic second-order equation and as can be observed, there is clearly a quadratic relationship between the specific surface areas and the average specific cake resistances (correlation coefficient $R2 = 0.967$). This is an expected result as was already shown earlier by equation 2. An overall conclusion that can be made according to the results presented in Fig. 5 is that the Blaine air permeability method was a suitable technique for estimating the filtration behaviour of the materials examined in this study. It should be pointed out here that most of the studied materials were fairly coarse and it would therefore be necessary to continue the experiments so that also finer powders would be included into the sample set. It may be assumed based on the existing literature [6] that there is probably a limiting particle size under which the Blaine method fails to provide reasonable results. Another property that has not been considered in this study at all is the compressibility of the filter cakes. It is reasonable to assume that this method is suitable only for incompressible or slightly compressible cakes since the accuracy of the air permeability method is probably not good enough to take into account the variations in the packing structure caused by changes in the applied filtration pressure. Despite these possible weaknesses, the Blaine air permeability method seems to be an efficient alternative for obtaining information about the filtration characteristics of powders in those cases where the quantity of sample is limited. The total amount of powder samples required by this technique is less than 100 g/sample.

The results of this study and those of previous studies demonstrate the limitations of small scale equipment in predicting full scale filter performance, however if these limitations are well understood very small scale equipment can be used to predict filtration performance with significantly better accuracy than a purely theoretical approach. As with any experimental campaign a systematic approach to planning the campaign will improve the reliability of the data and reduce the time and cost of conducting that experiment.

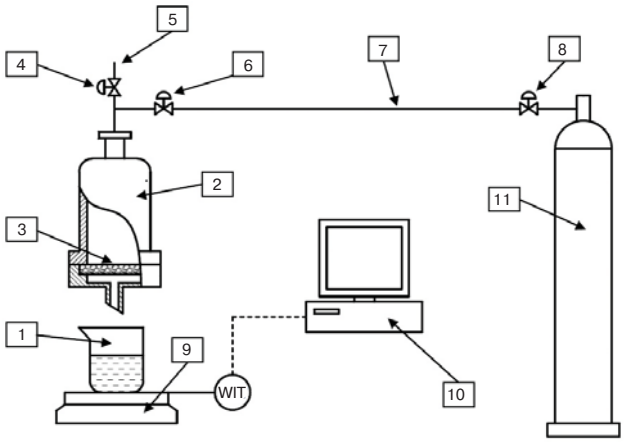

Fig. 4. A schematic view of the pressure filtration equipment. Numbers mean:
1 – glass container for filtrate; 2 – laboratory-scale Nutsche filter; 3 – replaceable filter medium; 4 – inlet pipe for slurry; 5 – outlet pipe for filtrate; 6 – gas valve; 7 – pipeline for nitrogen supply; 8 – valve for gas pressure adjustment; 9 – laboratory analytical scale; 10 – computer; 11 – gas bottle

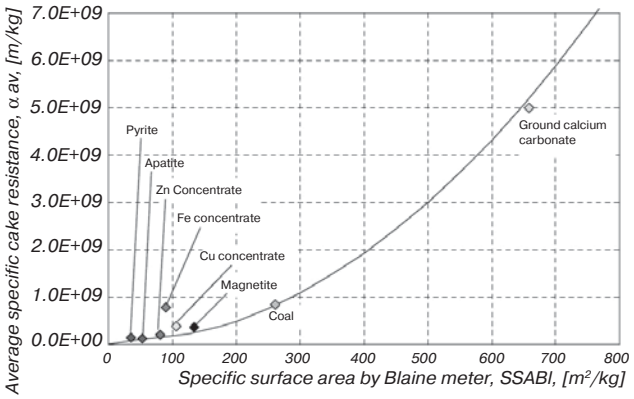

Fig. 5. Correlation between the specific surface areas measured by the Blaine meter and the average specific cake resistances for pressure difference of 2.0 bar

REFERENCES

1. ASTM, 2007. Standard test methods for fineness of hydraulic cement by air permeability apparatus (American Society for Testing and Materials: West Conshohocken).
2. *Chmelar J.* 2006. Size Reduction and Specification of Granular Petrol Coke With Respect to Chemical and Physical Properties, Doctoral thesis (Norwegian University of Science and Technology, Trondheim).
3. *Grace H. P.* 1953. Resistance and compressibility of filter cakes, Chemical Engineering Progress, 49(6): 303-318.
4. *Häkkinen A., Püllanen K., Reinikainen S., Louhi-Kultanen M., Nystrom L.,* 2008. Prediction of filtration characteristics by multivariate data analysis. Filtration, 8(2). p. 144-153.
5. *Osborne D. G.*, 1981. Vacuum Filtration – Part I, in Solid-Liquid Separation, 2nd Edition (ed: L Svarovsky), p 349 (Butterworth & Co Ltd: London).

6. *Potgieter J. H.* and *Strydom C. A.* 1996. An investigation into the correlation between different surface area determination techniques applied to various limestone-related compounds. Cement and Concrete Research, Vol. 26, No. 11, p.1613-1617.
7. *Savolainen M., Häkkinen A., Ekberg B., Hindström R.* and *Kallas J.* 2009. Development of testing procedure for ceramic disc filters, in Proceedings Physical Separation '09 (Minerals Engineering International: Falmouth).
8. *Tiller F. M.,* 1953. The role of porosity in filtration. Numerical methods for constant rate and constant pressure filtration based on Kozeny's law, Chemical Engineering Progress, 49(9): 467-479.
9. *Wakeman, R. J.* and *Tarleton, E. S.,* 2005. Solid/liquid separation: principles of industrial filtration, Elsevier, Oxford, UK.

A. V. Lychev , E. N. Vinogradov, I. V. Loginov
Lengipromez, Severstal Metallurgical Works
alychev@yandex.ru

On the blast losses in the blast furnace practice

Development of the ironmaking practice in the last decades is characterized by a steady improvement of the raw material and coke quality, by use of various coke-substituting materials injected into the blast-furnace hearth (coal dust, natural gas, fuel oil, etc.) with a high-temperature oxygen-enriched blast jet, as well as charged via the furnace top (nut coke, thermoanthracites). Introduction of the latest advanced technologies resulted in a substantial stabilization of the gas-dynamic and thermal conditions of the blast-furnace smelting process, and, as a consequence, in a considerable reduction of the specific coke rate per unit of iron made. The increased number of the practicable process control parameters, on the background of the further development of the computer technique in general, not only makes the monitoring process by no means more complicated, but what's more, allows to select the optimum combinations of all these factors for the particular conditions of each furnace.

The control and forecast systems for the thermal conditions of the smelting process, which use in their software algorithms the information pertaining to the top gas composition from the gas-analytical systems of high precision, do not have available the data on the quantity of hydrogen involved in the indirect iron reduction reactions ($V_{H_2O_i}$). These data are introduced into the system by means of calculations of the hydrogen imbalance in the furnace, and the above calculations are often based on incomplete, and what's more, inadequate information about the hydrogen content in some materials. Essential inaccuracies in calculation of $V_{H_2O_i}$ reduce drastically the effectiveness of operation of the automatic control system of the thermal conditions of the smelting process (ACS TC).

In this connection, creation of a reliable in operation additional autonomous channel providing the ACS with a reasonably fair forecast for the thermal state of the blast-furnace hearth, which shall be based on some other principles and not use the information on the top gas composition, seems to us to be a burning problem the solving of which will contribute to the further improvement of the blast-furnace process. We propose to accept the data about the volume of hot blast delivered into the blast furnace as a source of information for an alternative monitoring and control channel.

An attractive Alternative for determination of the rate of blowing into the furnace is its direct measurement. But to carry out the measurement of the hot blast flow rate (t_b = =1200÷1300 ^oC) is very complicated from the engineering point of view, and the cold blast flow measured by means of an orifice plate prior to entering of blast air into the hot-blast stoves differs considerably from the heated blast volume delivered into the blast-furnace hearth.

It has been noticed long ago already that the major blast losses occur at non-tight closed chimney valves of the Cowper stoves [1, 2], that is the consequence of their extreme operating conditions. The blow-through points at these valves, apart from a loosely fitting of the valve disk to its seat, are also the flanges connecting the valve cover with the valve case, the case with the hot-blast stove pipe connection and the valve case with its base, the seal failure of which is caused by an action on these flanges of variable forces resulting from the hot-blast stove wind operation or gas operation. During the gas period there is a thermal growth of the hot-blast stove casing by height, and due to this, the chimney valve, which is rigidly connected with the pipe connection of the hot-blast stove, moves in the vertical plane, as well. This results in extension of the bolts connecting the valve case and the base, and, as a consequence, the air leaks via the sealing cord between the valve case and seat occur [3]. The operating experience of the hot-blast stoves suggests that these losses make up 5÷20 % [4]. The losses are individual for each hot-blast stove unit and may vary in the process of their operation.

Before the breakdown of the former USSR the data on the cold blast losses at the enterprises had to be obtained in compliance with the existing at that time «Instructions for Determination of the B.F. Gas Yield and B.F. Blast Losses» elaborated by YuVEnergometallurgprom. But even at that time already not every enterprise followed strictly all the instructions. Thus, at the Makeevsky Metallurgical Works and «Zaporozhstal» Metallurgical Works the amount of air that was lost when passing through the duct from the blowing engines to the air-relief valve was determined as a difference between the indicated values of the flow meters at the blowing engines and of the flow meters upstream of the air-relief valve. And the hot blast losses were determined by the difference