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3. There is no fixed format for a paper, but authors are asked to note the following:
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   - Provide a Conclusions section toward the end of the paper.
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FILTRATION PERFORMANCE TEST – EXPERIMENTAL RESULTS AND CASE STUDIES

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This paper describes some of the features of new equipment for laboratory scale filtration testing. Descriptions are provided for the Filtration Performance Test, which facilitates the automated acquisition of constant pressure filtration data, and Filtration Performance Software which facilitates data analysis, including the automated calculation of characterising parameters such as specific cake resistance and filter cake compressibility. Exemplar experimental data are presented to show equipment and analysis performance in relation to repeatability measurements, filtration cell diameter, particle diameter and particle shape. The use of the approach to aid process scale equipment specification is also considered and some examples are provided.

INTRODUCTION

The Filtration Performance Test is a tool for precisely analysing how a suspension behaves during filtration over a wide range of experimental conditions to ensure the best filtration possible; experimental variables include filtration pressure, deliquoring pressure and the chosen filter media. Achieving such precision has required several years of development including numerical simulation, trial comparisons, ergonomic studies with a designer and user feedback. The result is a product that is patented all over the world. The aim of the Filtration Performance Test is to, in just a few minutes, assess product filterability in order to:

- increase the productivity of an industrial process
- anticipate the variability of a valuable product
- amortise more quickly the investment on equipment.

With reference to Figure 1, a simple and secure agitated tank was specially designed and its protection ensured by an anti-projection receptor. The human machine interface (HMI) fitted to the FPTLab has been designed to be intuitive, all the trial information is traceable and libraries of products can be created. Filtration tests can follow quickly one after another, with or without deliquoring, the only requirement being to connect-click a new filtration cell. The HMI screen allows:

- all traceability parameters to be entered
- live plotting of $t/V = f(V)$
- determination of filtration resistance, including a colour coding system for filterability
- calculation of the correlation coefficient
- immediate production of a filtration report
- data back-up from trials.

Post processing via the specially developed Filtration Performance Software allows:

- import of trial data
- production of personalized filtration reports
- cake compressibility determination
- objective and comparative analysis
- standardised filterability report.

GENERAL ASPECTS OF THE FILTRATION PERFORMANCE TEST LAB (FPTLAB)

The FPTLab (Figure 1) allows the quick measurement of filtration parameters such as cake specific resistance and compressibility. Its operation is based on the integrated form of Darcy's law at constant pressure which shows the relationship between filtration time and volume of filtrate collected according to equation (1):

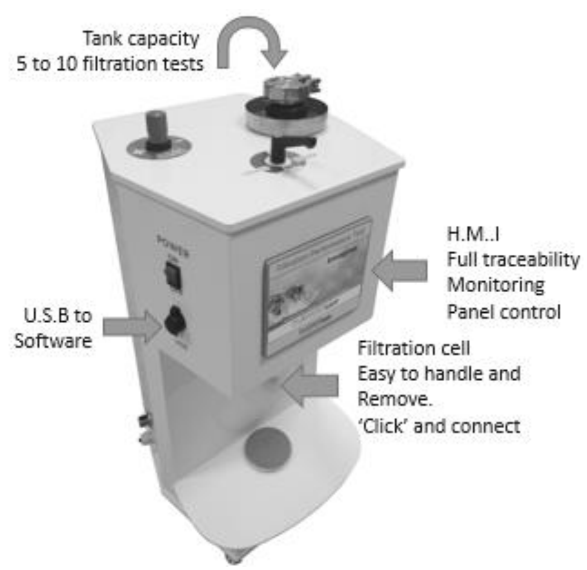

Figure 1: Labelled photograph of the FPTLab.

$$t = \frac{\mu \alpha_{av} W}{2S^2 \Delta P} V^2 + \frac{\mu R_s}{S \Delta P} V \qquad (1)$$

where t is the filtration time, μ is fluid viscosity, α_{av} is average specific cake resistance, W is effective solids concentration, ΔP is filtration pressure, S is filter area, V is cumulative volume of filtrate and R_s is filter medium resistance. Division by the volume of filtrate collected gives the link between t/V and V:

$$\frac{t}{V} = \frac{\mu \alpha_{av} W}{2S^2 \Delta P} V + \frac{\mu R_s}{S \Delta P} \qquad (2)$$

Equation (2) is the theoretical basis of the FPTLab. The suspension, under constant pressure in the tank, is conveyed to the filtration cell which is equipped with a suitable filter medium. The solids are stopped and the filtrate is collected in a beaker beneath the cell. The mass of filtrate collected as a function of time is recorded. By using the liquid density the inbuilt calculator determines the volume of filtrate collected as a function of time and then plots the ratio t/V as a function of V. A linear regression is performed upon the wanted part of the curve which can be shown on the display screen. The slope of the t/V vs. V plot allows the calculation of specific cake resistance according to:

$$\alpha_{av} = \frac{2S^2 \Delta P}{\mu W}.\text{slope} \qquad (3)$$

The HMI integrated into the FPTLab gives an immediate result for the measured specific cake resistance and the correlation coefficient associated with the linear regression of t/V vs. V. The measurement of specific cake resistance at different filtration pressures allows calculation of the compressibility coefficient from a linear regression of ln(ΔP) vs. ln(α) where the slope (n) is the compressibility coefficient and given by:

$$n = \frac{\ln(\Delta P) - \ln(\Delta P_0)}{\ln(\alpha) - \ln(\alpha_0)} \qquad (4)$$

Terms with the subscript ‘0’ are reference values that are usually taken at a pressure of 1 bar. However, this can vary according to the experiments performed. The Filtration Performance Test acquires values of n thanks to the automated handling of measurements at different pressures. For the reprocessing of data collected, FlowerSEP has developed Filtration Performance Software which extracts data from Filtration Performance Test trials (see Figure 2). The Filtration Performance Software also allows the production of standardized reports, and gives a direct, colour coded, assessment of suspension filterability (see Table 1).

REPEATABILITY MEASUREMENTS FOR SPECIFIC CAKE RESISTANCE

This section presents exemplar repeatability tests for the FPTLab with 6 µm diameter calcium carbonate spheres dispersed in water. Here, five different trials were carried out at an applied pressure of 3 bar using a 3 cm diameter filtration cell. The trials were compared in terms of calculated specific cake resistance and a statistical analysis was performed. Figure 3 shows that the results are within the 95% confidence interval (shown in grey highlight) and hence the measurement of specific cake resistance is considered to be repeatable.

INFLUENCE OF CELL DIAMETER ON SPECIFIC CAKE RESISTANCE

This section presents a study of the influence of filter

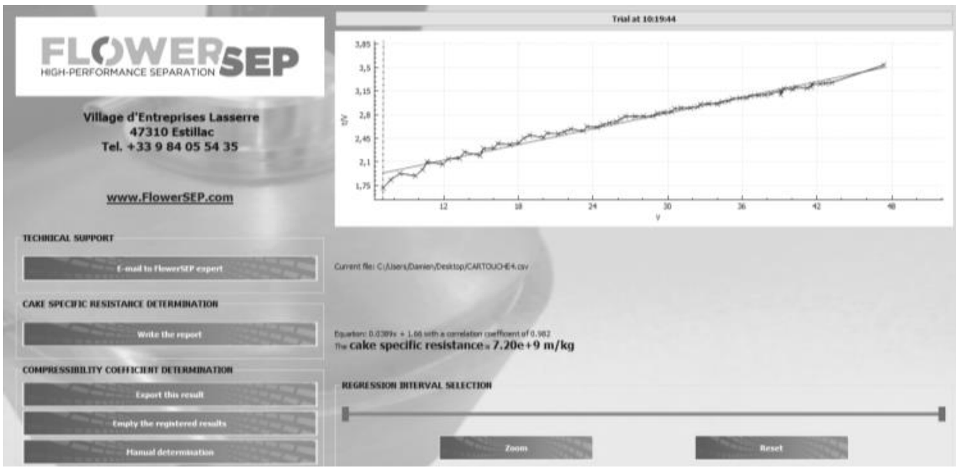

Figure 2: Example screenshot from the Filtration Performance Software.

Colour coding of the Filtration Performance Test	Associated average specific cake resistance (m kg^{-1})	Filterability
EASILY FILTERABLE	$\alpha_{av} < 10^9$	Very low resistance, under validation by the compressibility coefficient
SHOULD BE FILTERABLE	$10^9 < \alpha_{av} < 10^{10}$	Low resistance, under validation by the compressibility coefficient
SPECIAL PRECAUTIONS NEEDED	$10^{10} < \alpha_{av} < 10^{11}$	High resistance, study carefully the compressibility coefficient
NEEDS TO BE CAREFULLY STUDIED	$\alpha_{av} > 10^{11}$	Very resistant, study very carefully the compressibility coefficient

Table 1: Classification of suspension filterability.

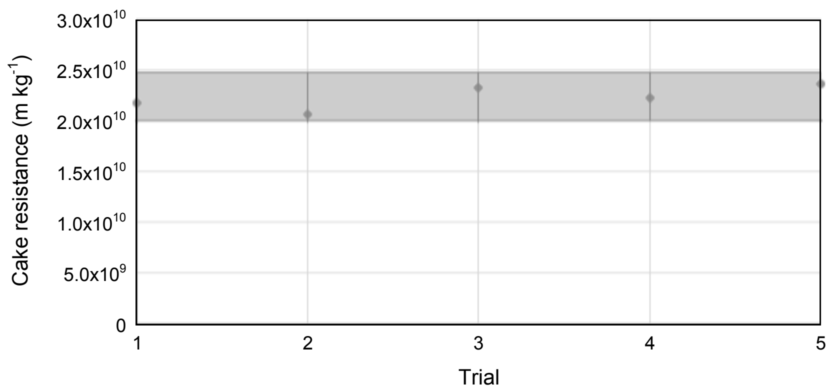

Figure 3: Exemplar repeatability measurement distribution for the Filtration Performance Test.

cell diameter on specific cake resistance. The trials were again carried out on the FPTLab equipment with calcium carbonate spheres. In each case five different trials were performed at an applied pressure of 3 bar using two different cells of 1 cm and 3 cm diameter, respectively. Statistical tests with a 95% confidence interval were done on the calculated specific cake resistances and these showed that the mean values for both cells are not significantly different (see Table 2). Hence, wall effects can be neglected and it is concluded that measurements with the 1 cm cell are representative of those with the 3 cm cell, and the reduction of diameter is validated.

INFLUENCE OF PARTICLE DIAMETER ON SPECIFIC RESISTANCE & COMPRESSIBILITY

This section presents measurements for the influence of particle diameter on specific cake resistance and compressibility coefficient. The trials were again carried out using the FPTLab, in this case with 20 μm and a 50 μm calibrated spheres of poly methyl methacrylate (PMMA, see Figure 4) dispersed in water. For each particle size three different trials were performed at 1, 3 and 5 bar applied pressure using the 1 cm diameter filtration cell. The results are presented in Table 3.

As expected, the specific cake resistance increased significantly with a reduction in mean particle diameter. At each pressure the specific cake resistance was *ca.* 7 times higher for a suspension containing 20 μm particles compared to one containing 50 μm particles. This result is broadly in accordance with the rule of thumb that cake resistance is inversely proportional to particle size (x) squared (i.e. $\alpha_{av} \propto 1/x^2$). The cake compressibility didn't seem to be significantly affected by the difference in particle diameter which could be reasonably expected for monodisperse particles of the same shape in suspension.

	Cell diameter		Colour coding of the Filtration Performance Test
Trial No.	3 cm	1 cm	
1	2.18x10^{10}	2.33x10^{10}	SPECIAL PRECAUTIONS NEEDED
2	2.06x10^{10}	2.28x10^{10}	SPECIAL PRECAUTIONS NEEDED
3	2.32x10^{10}	2.27x10^{10}	SPECIAL PRECAUTIONS NEEDED
4	2.22x10^{10}	2.30x10^{10}	SPECIAL PRECAUTIONS NEEDED
5	2.36x10^{10}	2.20x10^{10}	SPECIAL PRECAUTIONS NEEDED
Mean	2.23x10^{10}	2.28x10^{10}	SPECIAL PRECAUTIONS NEEDED
Standard deviation	1.19x10^{9}	4.83x10^{8}	

Table 2: Influence of filter cell diameter on specific cake resistance (in m kg^{-1}).

Applied pressure (bar)	Particle diameter		Colour coding of the Filtration Performance Test	
	20 µm	50 µm	20 µm	50 µm
1	5.70x10^{9}	8.00x10^{8}	SHOULD BE FILTERABLE	EASILY FILTERABLE
3	8.10x10^{9}	1.30x10^{9}	SHOULD BE FILTERABLE	SHOULD BE FILTERABLE
5	1.00x10^{10}	1.40x10^{9}	SPECIAL PRECAUTIONS NEEDED	SHOULD BE FILTERABLE
Compressibility coefficient (*n*)	0.34	0.38		

Table 3: Effect of pressure and particle size in the feed suspension on specific cake resistance (in m kg^{-1}).

INFLUENCE OF PARTICLE SHAPE ON SPECIFIC RESISTANCE & COMPRESSIBILITY

This section illustrates the effects of particle shape on specific cake resistance and compressibility. Two types of particles in aqueous suspension were used in the FPTLab, namely 20 µm diameter PMMA spheres (Figure 4) and 20 µm diameter calcium carbonate needles (Figure 5). For each shape, three different trials at 1, 3 and 5 bar applied pressure were performed with a 1 cm diameter filtration cell. Table 4 shows the results.

The specific cake resistance values for each particle shape were in the same order of magnitude, but increased at higher filtration pressure which is a typical indicator of cake compressibility. The compressibility coefficient (*n*) is highly dependent on the particle dissymmetry and *n* was *ca.* three times higher for the needle shape particles.

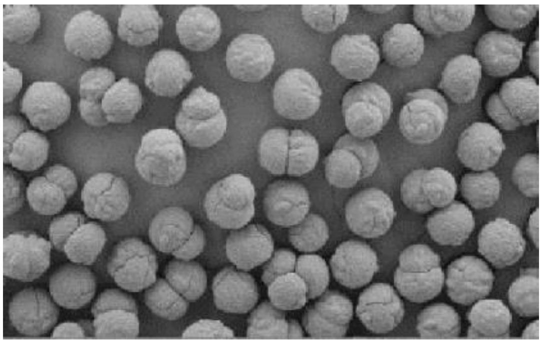

Figure 4: SEM image of PMMA spheres.

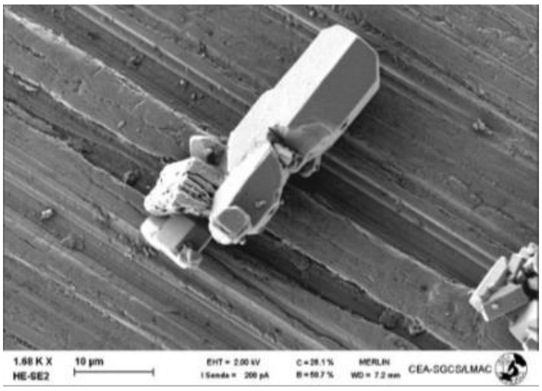

Figure 5: SEM image of synthesized $CaCO_3$ needles.

FURTHER USES OF THE FILTRATION PERFORMANCE TEST

In addition to the range of tests described above, the results from the Filtration Performance Test, e.g. specific cake resistance and compressibility coefficient measurements, can be used to aid the choice of solid-liquid separation device at the process scale. Figure 6 is an exemplar chart which indicates how the data obtained from the FPTLab is related to the typical requirements of some filter and centrifuge types; the Laminar Flow CS is a centrifugal separator that was jointly developed between FlowerSEP and the Atomic Energy Committee.

The data obtained from the Filtration Performance Test can also be used to help optimize the filtration and deliquoring pressures for a given suspension. Such optimization potentially allows industry to improve their productivity with a dryness defined at the end of the process. The Filtration Performance Test together with a specially designed calculation code is an important tool to help reach this target. The exemplar surface plot in Figure 7 represents, for a given dryness, the separation time for a range of filtration and deliquoring (drainage) pressures. Using such a surface plot allows an optimum to be identified for a chosen pair of variables. The Filtration Performance Test can be used to generate data to feed into a calculation code that optimizes the filtration and deliquoring pressures for a particular suspension.

APPLICATION OF THE PROCESS DESCRIBED

By way of example, the process described in the previous sections has been applied to the separation of cerium oxalate from aqueous suspension. Cerium oxalate is a crystalline solid that is slightly soluble in water and has the chemical formula $Ce_2(C_2O_4)_3$.

Table 5 shows the specific cake resistance and compressibility coefficient for cerium oxalate as measured using the FPTLab and Filtration Performance Test. For a continuous process, as the solids are the valuable phase, the technological choice for pressures lower than 3 bar would be vacuum filtration. This is because the cake specific resistance is not high. If the process

Applied pressure (bar)	Particle shape		Colour coding of the Filtration Performance Test	
	Spheres	Needles	Spheres	Needles
1	5.70×10^9	1.45×10^9	SHOULD BE FILTERABLE	SHOULD BE FILTERABLE
3	8.10×10^9	3.83×10^9	SHOULD BE FILTERABLE	SHOULD BE FILTERABLE
5	1.00×10^{10}	6.47×10^9	SPECIAL PRECAUTIONS NEEDED	SHOULD BE FILTERABLE
Compressibility coefficient (*n*)	0.34	0.93		

Table 4: Effect of particle shape on specific cake resistance (in m kg^{-1}) and cake compressibility (*n* value).

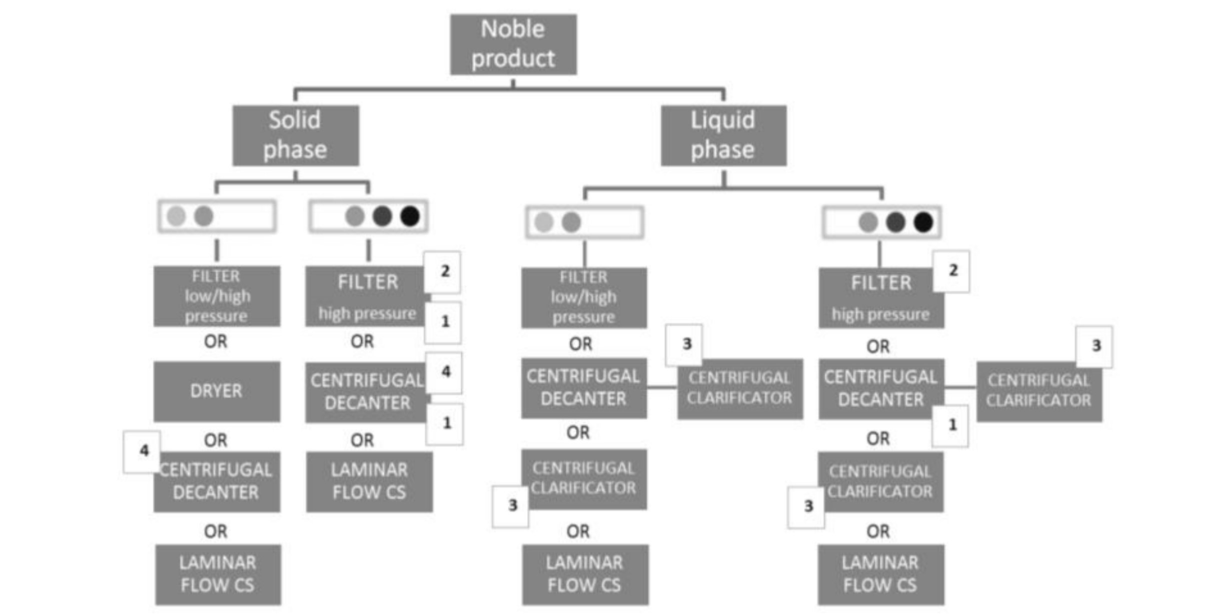

Figure 6: Chart showing the link between FPTLab results and the choice of an industrial device for solid-liquid separation. Key: 1 = possible adjustment of chemical conditions; 2 = possible addition of filtration additives; 3 = loss of noble liquid; 4 = loss of noble solid.

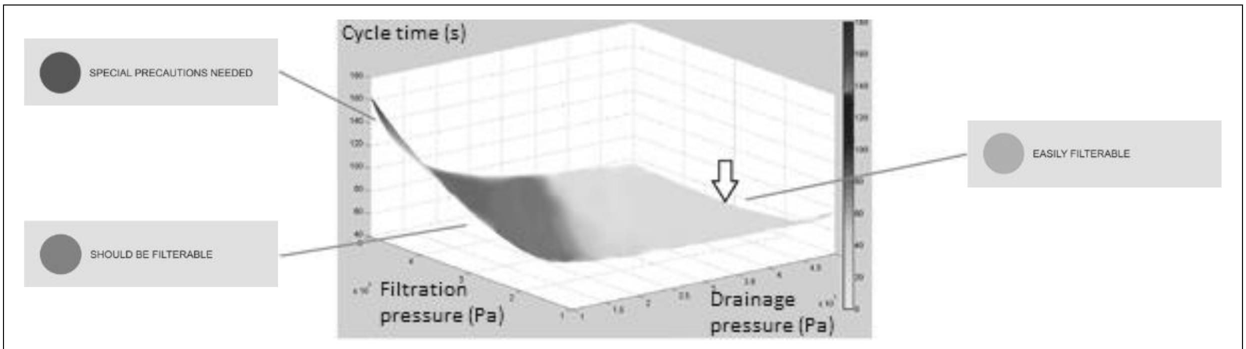

Figure 7: An example surface plot showing the relationship between filtration pressure, drainage pressure and filter cycle time.

Applied pressure (bar)	Specific cake resistance (m kg^{-1})	Colour coding of the Filtration Performance Test
1	6.20×10^{9}	SHOULD BE FILTERABLE
3	9.61×10^{9}	SHOULD BE FILTERABLE
5	1.33×10^{10}	SPECIAL PRECAUTIONS NEEDED
Compressibility coefficient (*n*)	0.46	

Table 5: Specific cake resistance and compressibility coefficient values for cerium oxalate.

must work at a higher pressure, for instance due to flow rate constraints, the technological choice would be or a spin dryer, or pressure filtration.

CONCLUSIONS

Filtration Performance Test repeatability and the use of a 1 cm diameter cell were validated by means of a range of experiments with the FPTLab. Different measurement possibilities were shown. The FPTLab is a new device that allows a quick and reliable measurement of specific cake resistance and cake compressibility coefficient. Knowing these parameters can guide a choice of industrial equipment or predict the impact of product variability on the whole process. Use of the FPTLab also allows the prediction of bottlenecks in an industrial process. For research and development units, it allows an optimization of operating pressures for the solid-liquid separation step.

NOMENCLATURE

d	cell diameter (m)
n	compressibility coefficient (-)
ΔP	applied filtration pressure (Pa)
ΔP_0	reference applied filtration pressure (Pa)
R_s	filter medium resistance (m^{-1})
S	filter medium area, = $\pi d^2/4$ (m^2)
t	time (s)
V	cumulative volume of collected filtrate (m^3)
W	effective solids concentration (kg m^{-3})
x	particle size (m)
α_{av}	average specific cake resistance (m kg^{-1})
α_0	reference specific cake resistance (m kg^{-1})
μ	fluid viscosity (Pa s)