

PERMEABILITY MEASUREMENTS - NEW CONTRIBUTION TO FLOCCULANTS SELECTION IN NON-ASBESTOS FIBRE CEMENT HATSCHEK PROCESS

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ABSTRACT

Research has been done both to study and to evaluate the stability and interaction of different flocculants within the slurry in the fibre cement Hatschek process but so far, these studies have been restricted to the vat environment and no correlation could be made to final products. In tropical countries, seepage in corrugated sheets can occur as consequence of improper flocculants selection that influences the characteristics of flocs formed during filtration in vats and processing stability. Proper selection of flocculants not only helps to solve this problem but contributes to build up a suitable microstructure which is responsible for the final properties of the composite. In this paper, a new technique of measuring permeability is presented as a tool to characterize and quantify permeability properties in fibre cement composites. It has been shown to be mainly useful at industrial scale to characterize and select the performance of flocculants with different molecular weight and ionic charges.

KEYWORDS:

Fibre-cement, Permeability, Flocculants, Processing

INTRODUCTION

In the non-asbestos fibre-cement processing by Hatschek machine, flocculants are necessary to help cellulose and synthetic fibres to retain fine particles. Flocculants are long chain polymers with ionic charges that promote agglomeration of the cementitious materials and fibres present in the slurry. These agglomerates are larger than the particulate and fibres themselves and are more easily retained on the sieve during the filtration step inside the vats. The mechanism of flocculation and properties and requirements were well described in the literature (Cooke, 2010).

In case of inappropriate selection of flocculants, low output, slow setting, low initial strength, high porosity can result. This also results in large amount of circulating fines which if kept in the water circuit result in risks for the process.

On the other hand, when flocculants are used in right dosage with suitable molecular size and charge, a better microstructure can be built and as result, higher strength, lower seepages and better freeze-thaw resistance are obtained.

For this reason flocculants play important role in the Hatschek process not only increasing the retention rate of cementitious and fillers particles on the laminar sheet formed by cellulose and synthetic fibres on the rotating sieve but also retaining water in the right amount to increase laminar bonding and green sheet plasticity to facilitate the corrugation without causing cracks.

Previous works related to flocculants for Hatschek process have been presented focusing on filtration process (Cooke, 2002), flocks formation (Negro et al., 2005) and flocculants selection (Negro et al, 2006; Cooke, 2010).

The objective of this article, in addition to those studies, is to present a new methodology that can be used in selection and characterization of obtained products based on permeation analysis as consequence of flocculants and process parameters. Permeability is an important issue directly related to performance of corrugated sheets.

This technique determines permeability measurements based on the Forchheimer's equation which is used to characterize the permeability of porous media (Innocentini et al., 1999a).

BACKGROUND

In 1856, Darcy proposed the following equation to characterize the passage of an incompressible fluid with low flow rate and having viscosity μ through a porous media with thickness (L):

$$\frac{\Delta P}{L} = \frac{\mu}{k_1} v_s$$

Where v_s is the face velocity of the fluid (the volumetric flow rate divided by the face area of the sample exposed to flow) and k_1 is the viscous or Darcy's constant which is related only to the porous structure considering only the friction effects which causes the drop of pressure drop.

In the 1900's, Reynolds and Forchheimer working with granular structures realized that Darcy's law as a linear correlation between pressure drop and flow rate could not explain behaviour at higher fluid flow rates. They observed a variation of kinetic energy of the incompressible fluid passing through a porous structure. Based on this observation, the following parabolic equation was proposed:

$$\frac{\Delta P}{L} = \frac{\mu}{k_1} v_s + \frac{\rho}{k_2} v_s^2$$

Where ρ is the density of the fluid and k_2 is the inertial or non-Darcynian constant which is also related to porous structure.

In the Forchheimer's equation, $\mu v_s/k_1$ represents the viscous interaction between fluid and solid and $\rho v_s^2/k_2$ represents the energy losses caused by changes in flow direction and by contractions and expansions of the flow path. Higher tortuosities affect both viscous and inertial effects which will result in higher fluid pressure drop.

Equipment used in this work

The apparatus used in this work is showed in the Figure 1. The procedure to trace the permeability curve is obtained by measuring the flow rate of gas or liquid through the sample at different pressures using a soap bubble meter or rotameter (Innocentini et al, 1999b).

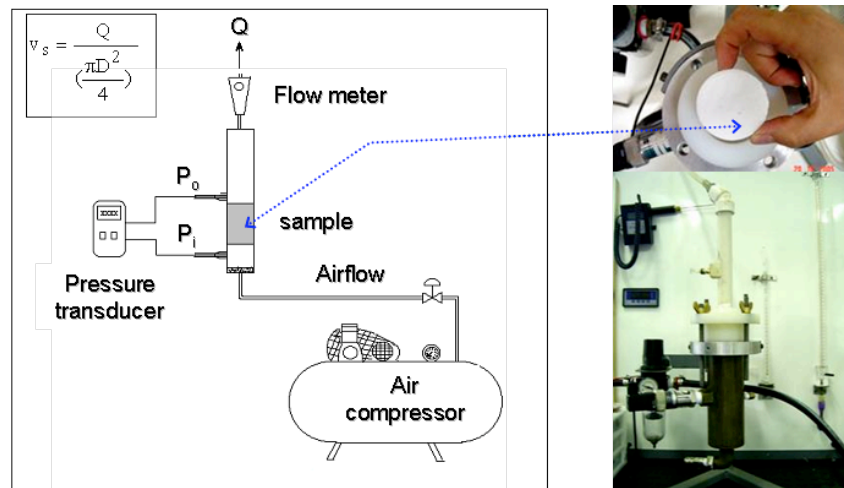


Figure 1 – Schematic permeability apparatus (Innocentini et al., 2001)

EXPERIMENTAL PROCEDURE FOR SAMPLES

The objective of this work was to present a method to evaluate the influence of flocculants available in the market on the permeability of products. Some commercial products were already used in industrial scale on Hatschek machine and were previously validated in laboratory using Leite sieve method (Leite in Cooke et al, 2010).

By this experiment, we would like to analyse the influence of flocculants on the sequence:

- Flocculation
- Filtration
- Packing
- Microstructure

Commercially, flocculants are characterized by averaged molecular weight (Mw) and ionic charge (IC). Here, we will also use the correlation of these two characteristics (Cooke, 2010) and will define here as Potential Flocculation Index (PFI) which is obtained by the following relationship:

$$PFI = M_w * IC$$

Six different compositions of anionic flocculants characterized were tested differing according to molecular weights and ionic charges using similar dilution in the preparation. Dosages were adjusted according to process response. All of them are random copolymers based on anionic polyacrylamide.

Table 1 – Flocculants used in the trial

Sample	PF Index
FE 57A	360
FE 57B	800
FE 57C	400
FE 57D	320
FE 57E	240
FC H04	660

Running trials were performed in industrial scale using in similar dosages according to their response in the process. During trials it was confirmed that not only the molecular weight and ionic charge can explain differences to the expected response of the process (Cooke, 2010) but other parameters like crosslink bonding and distribution of molecular weight may also be relevant.

Along the transversal direction of green sheet, samples (Figure 2) were collected before corrugation step and kept in plastic film until 21 days curing age when samples was dried and prepared for permeability measurements. Silicone based sealant was used to seal the edge of each sample to guarantee that pressure drops occurred in the right pathway.

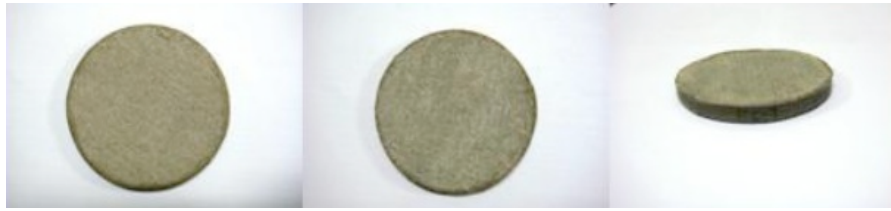


Figure 2 – Samples used in permeability measurements.

For each sample, permeability measurements were performed based on the stationary flow method collecting 20 data set of entry and output pressure through sample and air superficial velocity (v_s).

Data were analysed using the least square fitting method to Fochheimer's equation (Figure 3). Darcian and non-Darcian constants could be calculated for each sample.

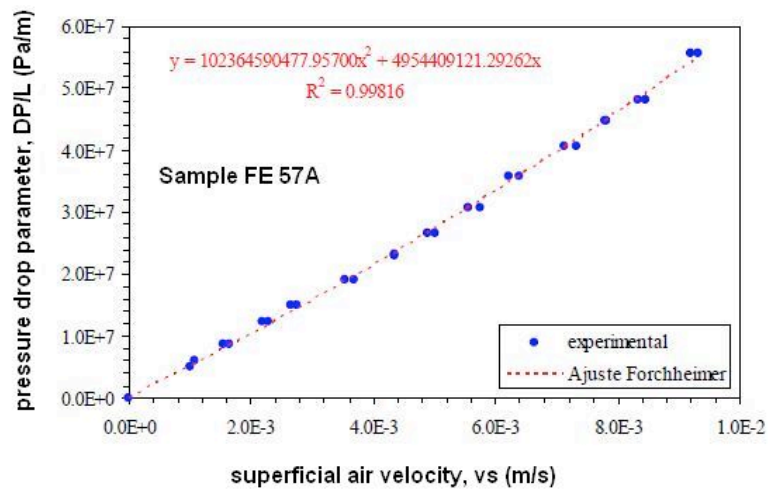


Figure 3 - Example of experimental curve with fitting of Fochheimer's equation to calculate permeability constants k_1 and k_2 .

DISCUSSION OF THE RESULTS

Table 2 presents the results of permeability characterization using the Fochheimer equation determined for each set of sample.

For the reference, pressures drops for water permeability were estimated using the Fochheimer's equation for a face velocity of 0,6mm/s.

Table 2 – Permeability data characterization of samples using Fochheimer equation.

Sample	PF Index	Darcynian Constant, k_1 (m ²)	Non-Darcynian Constant, k_2 (m)	Water Permeability (Σv in atm) for $V = 0,6\text{mm/s}$	Water Absorption (%)
FE 57A	360	2,43E-15	7,35E-12	4,3	27,0
FE 57B	800	1,38E-14	5,25E-11	0,8	28,3
FE 57C	400	2,09E-15	2,82E-12	6,6	24,1
FE 57D	320	1,30E-15	1,14E-12	6,6	24,3
FE 57E	240	7,51E-16	3,67E-13	11,8	24,5
FC H04	660	6,64E-15	2,11E-10	1,1	27,3

Note: FE 57D used a mixture of flocculants FE 57A and FE 57E, in ratio 1:1.

By analyzing the data of PF Index and water permeability, a direct relationship can be observed showing that high PF Index (product of high molecular weight and ionic charge) results in products with high porosity.

In Figure 4, the plot of constants k_1 and k_2 for all samples show where they are located in comparison to different porous materials.

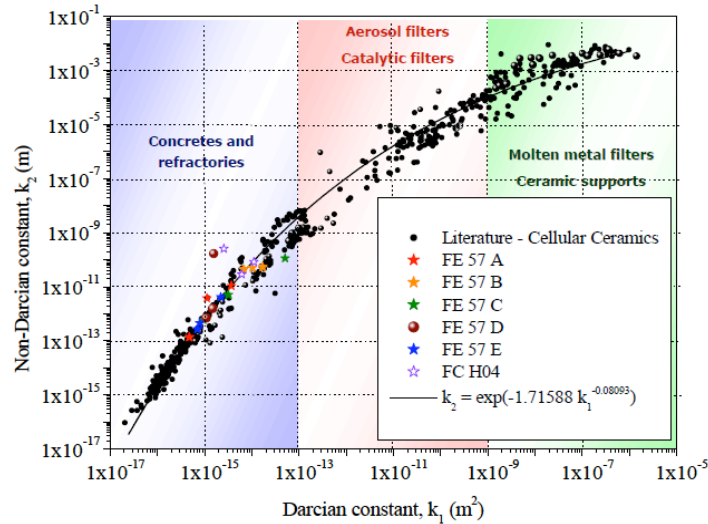


Figure 4–Location of fibre cement composites in comparison to other porous materials (data from Innocentini et al, 2010)

Figure 5 presents the plot of both constants of samples. Flocculants with high PF Index like 800 and 660 showed higher permeability characteristics.

On the other hand, samples produced with flocculants with low PF Index, as 360, 400 and 240 showed low permeability characteristics. It is interesting to note that a mixture 1:1 of two flocculants 240 and 360 resulted in products with permeability characteristics in between both flocculants (PF Index \sim 320). These may represent a more open distribution of molecular weight or ionic charge.

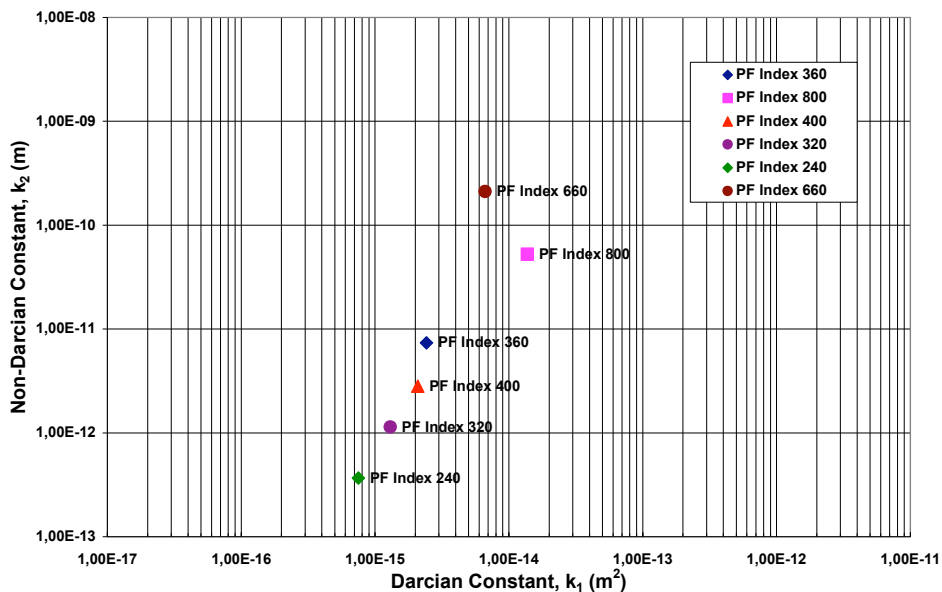


Figure 5 – the plot of constants k_1 and k_2 of samples with different flocculants.

Further trials using those flocculants were also subjected to the permeability methodology. In table 3, the averaged permeability was calculated for all trials using the same flocculants besides raw materials variation. PFI 660 was the reference to former flocculants used in the process. In figure 5, relative permeability and PFI show an excellent correlation to flocculants demonstrating to be a very useful tool for flocculants selection. These results emphasize the influence of flocculants on the microstructure of fibre-cement composites.

Table 3 – Averaged water permeability and relative permeability for different trials using the same flocculants.

PFI	Averaged Water Permeability (Σv in atm) for $V = 0,6\text{mm/s}$	Relative Permeability
320	6,61	4,48
400	6,59	4,46
360	6,06	4,11
440	3,49	2,37
600	2,63	1,78
660	1,48	1,00
800	0,75	0,51

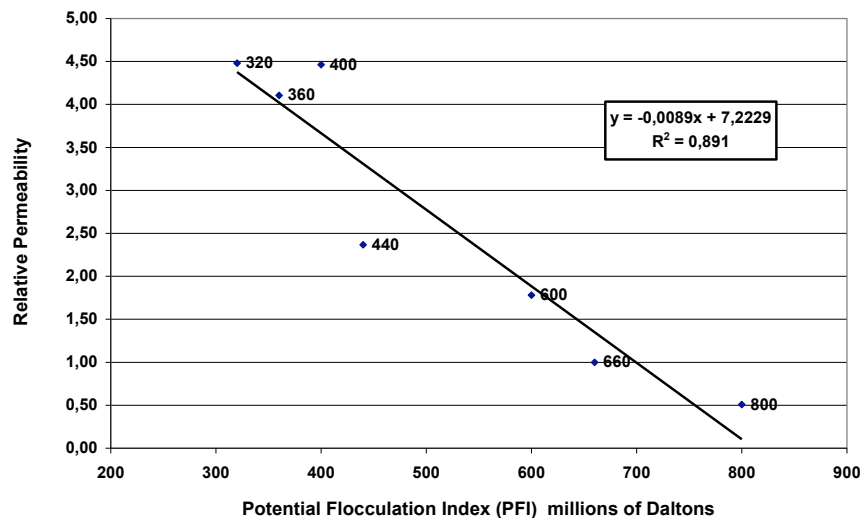


Figure 5 – Correlation of PFI and relative permeability

CONCLUSION

The influence of different flocculants on the microstructure of fibre cement products was analysed based on permeability characteristics.

Flocculants characteristics related to molecular weight and ionic charge control the flocculation and stability of flocks, filtration process and microstructure formation. Those steps contribute to build up of the microstructure of final products.

The technique for measuring permeability using Forchheimer's equation was found to be an interesting method for selection flocculants involving influences of all steps in the Hatschek processing industrial scale.

Further studies will be developed based on this technique to access microstructure build up in green sheets to evaluate process parameters like flocculants dosage, drying vacuum and forming pressure, in addition to raw materials characteristics.

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REFERENCES

- Cooke, A.M., Akers, S.A.S., Leite, J.V. and Winter, R. 2010. “Selection and performance of flocculants for Hatschek made air cured fibre cement.” *In*” Proceedings of 2010 International Inorganic Bonded Cementitious Composite”. Aalborg, Denmark.
- Innocentini, M.D.M. and Pandolfelli, V.C. 1999a. “Issues concerning the evaluation of permeability in refractory concretes with Darcy and Forchheimer equations”. *Cerâmica* 45, 292-293, Publication of Associação Brasileira de Cerâmica.
- Innocentini, M. D. M.; Salvini, V. R. ; Pardo, A. R. F. ; Pandolfelli, V. C. 1999b . “How Accurate is Darcy's Law for Refractories.” *American Ceramic Society Bulletin, USA*, v. 78,(11), 64-68.
- Innocentini, M. D. M., Caldato, R. 2010. Evaluation permeability of Saint-Gobain Fibre cement corrugated sheets. Internal report.
- Leite, J.D. “Method for evaluation of flocculants”. Ashland Hercules Water Technologies Latin America.
- Negro, C; Blanco, A; Fuente, Elena; Sánchez, L.M. and Tijero, J. 2005. “Influence of flocculant molecular weight and anionic charge of flocculation behaviour and n the manufacture of fibre cement composites by Hatschek process.” *Cement and Concrete Research*, 35, 2095-2103.
- Negro, C; Blanco, A; San Pío, I. and Tijero, J. 2006. “Methodology for flocculant selection in fibre-cement manufacture.” *Cement & Concrete Composite*, 28, 90-96.