

# Methodology for flocculant selection in fibre–cement manufacture

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## Abstract

In the Hatschek process used to produce fibre–cement products, it is necessary to use a suitable flocculant when asbestos is substituted by pulp fibres. The right selection of flocculant is crucial in the industrial process due to its effects on mineral fines retention, dewatering and formation and, as a consequence, on the overall efficiency of the machine. This paper presents a two-step methodology for flocculant selection in the fibre–cement manufacture. The first step is based on the study of the flocculation processes and the flock properties, using a focused beam reflectance measurement (FBRM). This technique allows the study of flock size, flock stability and flock resistance to shear forces, reflocculation tendency and reversibility of the flocks, as well as the optimal flocculant dosage for each particular fibre–cement suspension. The second step uses a drainage vacuum tester to study retention and dewatering. The two techniques give important and complementary informations that allow a proper selection of the best flocculant for the fibre–cement manufacture.

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## 1. Introduction

Over the last few years, many research studies related to the substitution of asbestos for others raw materials have been published [1,2]. These mainly focus on natural cellulose fibres (from wood or non-wood raw materials) [3–12] and synthetic fibres [1,2,13,14], alone or as a mixture. Out of all these sources the softwood unbleached Kraft fibres are the most widely used due to their strength characteristics, the high availability and the price.

Asbestos is a naturally occurring fibrous silicate and the fibre's size together with its chemical structure, make asbestos very compatible with cement. However, the different chemical composition and hygroscopic character of pulp fibres make the compatibility between cellulose

fibres and cement much more complex and, therefore, new aspects are needed to be considered. In the Hatschek process, the behaviour of these fibres is different and therefore, a suitable flocculant is needed when using pulp fibres. The right selection of flocculant is crucial in the industrial process due to its effect on mineral fines retention, dewatering and formation and, as a consequence, on the overall efficiency of the machine. Most work in this field has been carried out at mill sites and no public information is available in the literature. This paper addresses this issue, providing a methodology for optimal flocculant selection.

In the literature, the main methods to monitor the flocculation process of the fibre suspension are described. These methods are mainly based on electrokinetic properties like colloidal titration, cationic demand and zeta potential [15]. All these methods are difficult to apply to fibre–cement suspensions because of the high abrasion power of the solids, the colour of the suspensions and the high solid concentration in the

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suspensions. An indirect method for studying flocculation is based upon sedimentation tests after flocculant dosage has been added to the suspension. However, this method gives limited information about the flocculation process in the fibre–cement suspensions.

A different approach is based on monitoring the size distribution of the particles in the suspensions before and after adding the flocculant dosage. The change in both the average size and the size distribution of the particles in the suspension represents the flocculation process independently of the mechanism that takes place. The authors were pioneers in proposing this technique for the paper industry in 1996 [16–19]. This method allows to follow the flocculation process in real time, giving detailed important information such as type of flock, flock strength, the kinetics of the flocculation process, reversibility of flocks, influences of process conditions on flocculation like shear forces and temperature. Therefore, this method is appropriate to monitor the flocculation processes of a fibre–cement suspension, and this is the methodology developed in this paper.

On the other hand, in the literature several laboratory test procedures are described to predict the performance of chemicals for enhancing retention or dewatering for the manufacturing process [20]. For the fibre–cement suspensions, the most suitable method is the vacuum drainage tester (VDT). This allows to dewater the suspensions using a machine botting cloth, in order to simulate the retention on the vat, or a machine felt, in order to simulate the dewatering process once the different layers have been formed into the final sheet on the Hatschek machine. Consequently, it is also possible to determine the final water content of the sheet [21–23].

## 2. Materials and method

The methodology is a two-step process based on measuring flocculation, retention and drainage. First a FBRM probe is used to monitor the flocculation process and then, a vacuum drainage tester is used to study the retention and the dewatering processes.

The methodology to monitor flocculation is based on using a focused beam reflectance measurement system FBRM M500LF manufactured by Lasentec. The focused beam reflectance measurement offers the possibility of particle characterization in a wide concentration range. The FBRM instrument operates by scanning a

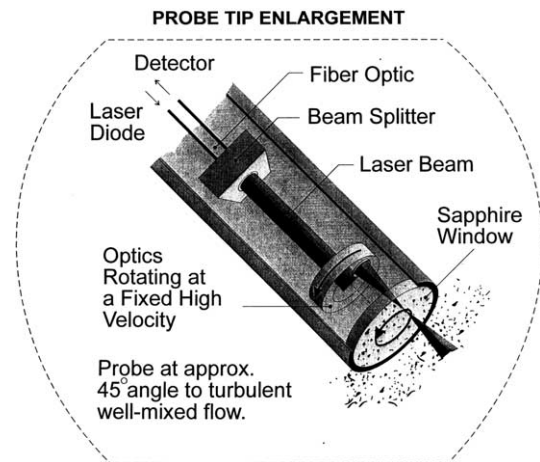


Fig. 1. FBRM probe.

highly focused laser beam, at a fixed speed, across the particles in the suspension and measuring the time duration of the reflected light from these particles (Fig. 1). The temporal duration of the reflection from each particle or flock multiplied by the velocity of the scanning laser, which is known, results in a characteristic measurement of the particle known as the chord length (Fig. 2). Thousands of chord length measurements are collected per second, producing a histogram in which the number of the observed counts is sorted in several chord length bins over the range of 0.2–2000  $\mu\text{m}$ . From the data, total counts, counts in specific size regions (population), mean chord length, and other statistical parameters can be easily calculated. The evolution of these various statistics under varying process conditions allows us to interpret the evolution of the flocculation process [24].

The second step in the methodology consists in retention and drainage studies. The equipment used for measuring retention and drainage was a vacuum drainage tester (VDT) supplied by Nalco. Fig. 3 shows a scheme of the lab device, it has two jars separated by a barrier: the upper jar is used to keep the fibre–cement suspensions stirred until the addition of the flocculant dosage. After the necessary contact time the barrier is removed and the suspension is drained to the second jar in which a filter is located. In this case two types of filters were used: a botting cloth (18 mesh) and a machine felt (mixed felt with a permeability to the air of 120 c.f.s.) in order to simulate the dewatering in the vat and in the upper part of the Hatschek machine. In this device

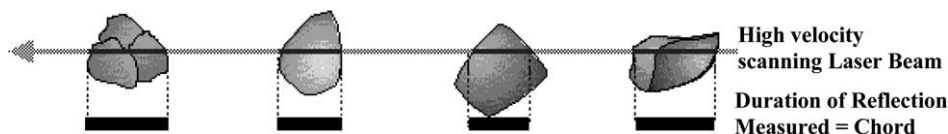


Fig. 2. Examples of particle chord length measurements.

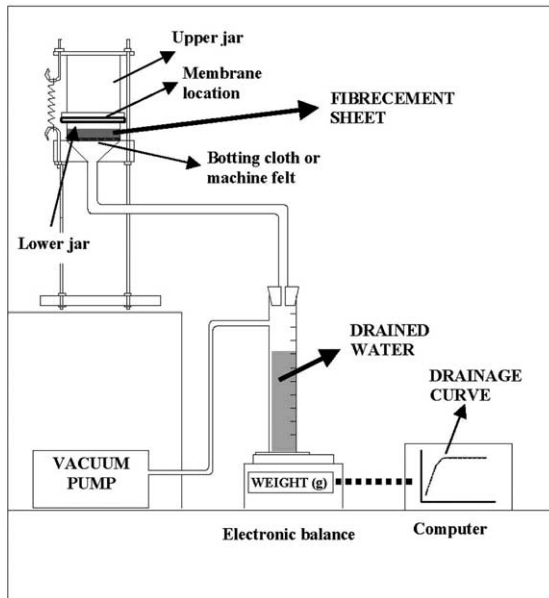


Fig. 3. Schematic view of the vacuum drainage tester.

the suspension is drained under a certain vacuum (150 mmHg) through the filter and a computerized balance records the mass of drained water over time. The drainage curve is analysed in order to obtain the drainage rate for the different flocculants.

The composition of the fibre–cement suspension was chosen to behave in a similar way as in the industrial situation with 50% cement (standard II/A-V 42,5N, clinker content 88%, with a specific surface of 330–350 m<sup>2</sup>/kg), 10% unbleached refined softwood Kraft pulp (kappa number 25 and refine degree 35° SR), 39% silica (size distribution 88.7% smaller than 1 µm and 99.8% smaller than 10 µm; specific surface 350–380 m<sup>2</sup>/kg) and 1% pigment. The suspension has a concentration of 75 g/L. This composition, valid for autoclaved products, has been used as an example to show the possibilities of the developed methodology. However, different types of raw materials and compositions could be tested with this methodology in order to cover any formulation valid for any technology (air cured or autoclaved products) or product (flat or corrugated sheets).

Several anionic polyacrylamides (A-PAM) were chosen as flocculants, with different charge and molecular weights from different suppliers. In total, fourteen A-PAMs were selected and they are referenced in this paper as A–N. Fig. 4 shows the two main characteristics of these A-PAM (molecular weight and anionic charge).

The experimental conditions were the same for each flocculant tested. A 500 mL fibre–cement suspension at 30 °C was used to monitor flocculation with the FBRM probe. The process was studied continuously in real time, while adding, at regular intervals, 22 different flocculant additions of 15 ppm each until a total dosage of

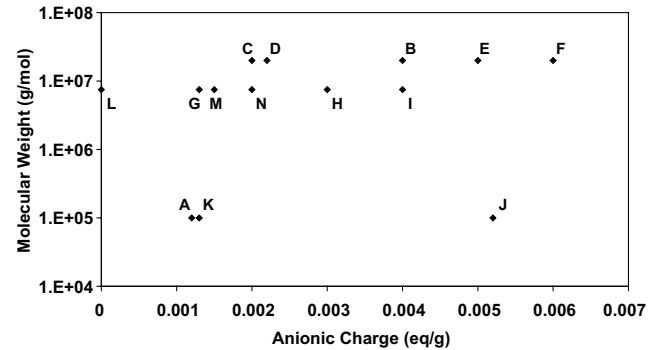


Fig. 4. Characteristics of tested A-PAM.

330 ppm was reached. The results were processed by the software and analysed for each flocculant.

To carry out the drainage and retention tests, a 500 mL volume of fibre–cement suspension was used. After flocculant addition (100 ppm) the dewatering process started and the water was collected in a graduate cylinder and then the evolution of weight versus time was recorded. The slope of the curve gives the dewatering rate for each flocculant. Finally, the retention and the water content in the cake were determined by gravimetric measurements.

### 3. Results

As an example, the results obtained from monitoring the flocculation process for each flocculant are shown in Fig. 5, in which the evolution of the average chord distribution of the particles in the fibre–cement suspensions, flocculated with the A-PAM (D, in this case), versus time is plotted. The profile of these curves provides the following singular point values:

- Initial value: corresponds to the measure before any flocculant addition.
- Maximum size of formed flocks: gives information about the maximum flock size obtained.
- Maximum size of broken flocks: is the maximum size value reached after the flocks have been broken after each flocculant addition.

In Fig. 5 is also possible to study the maximum variation of the formed flock or the maximum variation of the broken flocks, these two parameters give information related to the flocks stability and, as consequence, on flocculant performance for a given suspension.

As shown in Fig. 6, for each of these singular points obtained from the curve, it is possible to calculate the distribution chord length. After analysing the curves it can be observed that when the flocculant is added the average size increases because of the flocculation

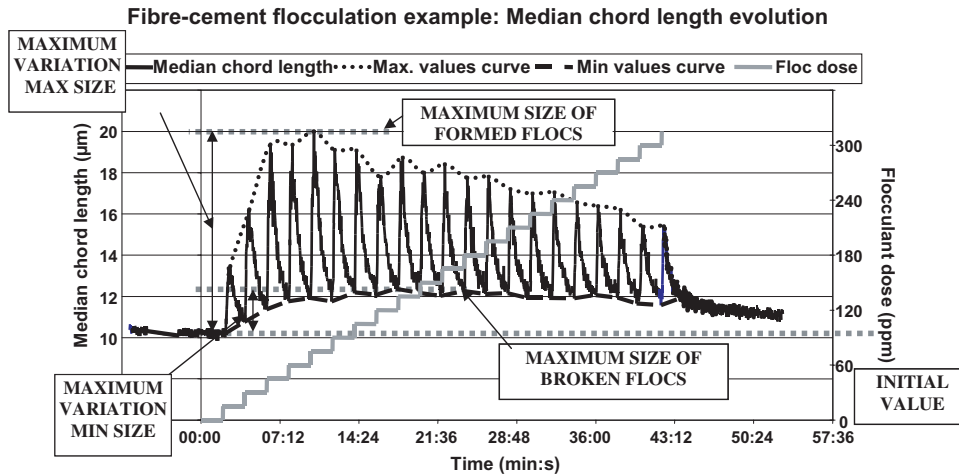


Fig. 5. Evolution of a fibre-cement suspension: median chord distribution versus time for different flocculants dosages (A-PAM, D).

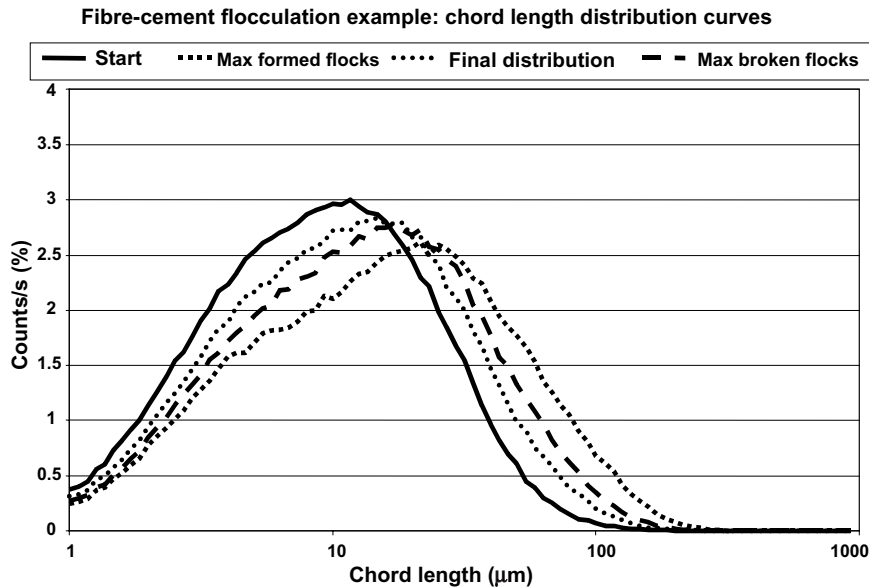


Fig. 6. Singular points in the flocculation process of a fibre-cement suspension.

process. After a certain time, the average size decreases until a final value is reached. This phenomenon occurs because the equilibrium between the flocculation, deflocculation and reflocculation processes has been reached. This gives an indication about the flocks' stability. Furthermore, this could be studied for a selected range of sizes, as illustrated in Fig. 7, which shows the evolution of four different range sizes.

In order to compare the behaviour of the different flocculants Table 1 presents a summary of important values obtained from the different curves of each flocculant. In principle, what is desirable is to obtain a high flock size in order to guarantee a good retention and this can be studied through the maximum variation of the formed flocks reached. It is also important to obtain the low

numbers for the total counts of the smaller particles (like between 6 and 12  $\mu\text{m}$ ) because these particles will be more difficult to retain. Therefore, a high number of counts of these particles will indicate that they will accumulate in the process. If the flocculant interacts with the smaller particles they will increase their size being more easy to retain. However, as well as the big flocks size, the stability of the flock is also crucial due to the fact that if the flocks are not stable they could be destroyed in the vat by the shear forces. This can be measured by studying the maximum variation of the minimum flock size for the average chord size (Fig. 5) as well as for the total counts in the specific size range (Fig. 7). Table 1 gives the results of the maximum variation for two different ranges: 6–12  $\mu\text{m}$  and for greater than 232  $\mu\text{m}$ .

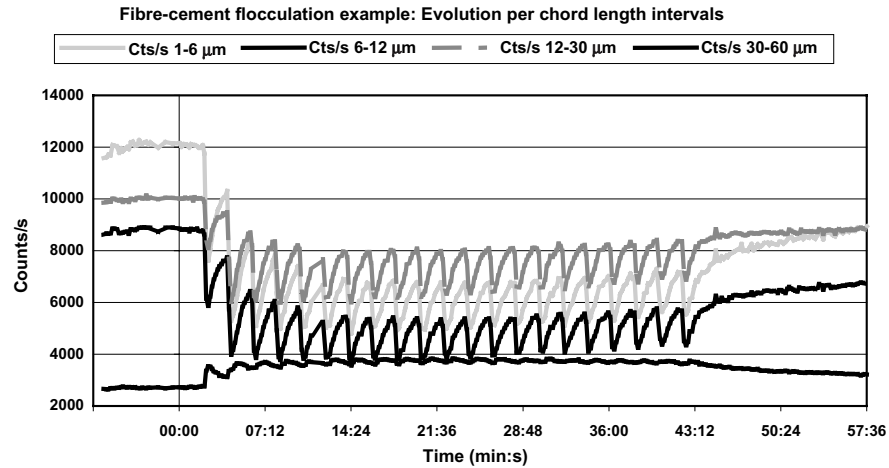


Fig. 7. Example of flocculation monitoring in different size ranges.

Table 1  
FBRM results for different flocculants

Flocculant	Maximum variation in formed flocks size ( $\mu\text{m}$ )	Maximum variation in broken flocks size ( $\mu\text{m}$ )	Maximum variation in 6–12 $\mu\text{m}$ max curve (cts/s)	Maximum variation in 6–12 $\mu\text{m}$ min curve (cts/s)	Maximum variation in >232 $\mu\text{m}$ max curve (cts/s)	Maximum variation in >232 $\mu\text{m}$ min curve (cts/s)
A	4.7	1.8	-3626	-1479	7.6	5.1
B	8.6	2.3	-5151	-1508	28.5	-1.3
C	7.9	2.4	-5615	-2217	25.2	-1.1
D	8.9	4.8	-7703	-5120	35.8	5.1
E	8.1	1.9	-5303	-995	21.4	-2.3
F	7.6	2.8	-6500	-2956	23.7	0.2
G	6.8	3.2	-4720	-2586	14.0	3.9
H	10.2	1.6	-6494	-1434	41.8	0.5
I	7.4	2.0	-5154	-1611	10.3	-0.6
J	5.6	2.4	-3541	-1279	5.8	-0.6
K	5.2	1.8	-4871	-1927	8.1	0.6
L	5.9	3.1	-6766	-4537	29.4	8.2
M	10.8	3.0	-8070	-3688	53.2	1.9
N	6.2	3.5	-6668	-4348	49.4	6.7

Table 1 shows that the flocculants that produce the smaller flocks are A, G, I, J, K, L and N (for all this flocculants the maximum variation in formed flocks size is lower than 7.5  $\mu\text{m}$ ). These flocculants will have poor retention behaviour. This can be further studied in the second step when retention is determined for each flocculant. At industrial scale, a low retention means a fines accumulation in the system and consequently associated problems in the production line. This will be particular important in formulations with high percentage of fines particles like, for example, in the production of flat product using air cured technology.

Flocks with low stability are also undesirable because they will break down in the vat and the retention will decrease. Flocculants A, B, C, E, H, I, J and K induce flocks of low stability and therefore, they are not suitable for the fibre-cement industry. This can be observed in Table 1, studying the maximum variation in broken

flocks size, which, for these flocculants, is low (less than 2.5  $\mu\text{m}$ ).

The only flocculants that interact with the suspensions producing the desirable type of flocks are D, F and M. This can be further analysis considering the values obtained for the different sizes intervals. For example, they correspond to the higher values of maximum variation in 6–12  $\mu\text{m}$  (Table 1).

This first step in the methodology allows us to exclude flocculants with low interaction with the particles in the suspension under study. Furthermore, the methodology allows to distinguish between different flocculants even in the case that the molecular weight and anionic charge are very similar. For example, flocculants G, M and N has similar characteristics however the behaviour is very different. This could be due to the different flocculant composition (e.g. number and types of branches, functional group for anionicity, viscosity and

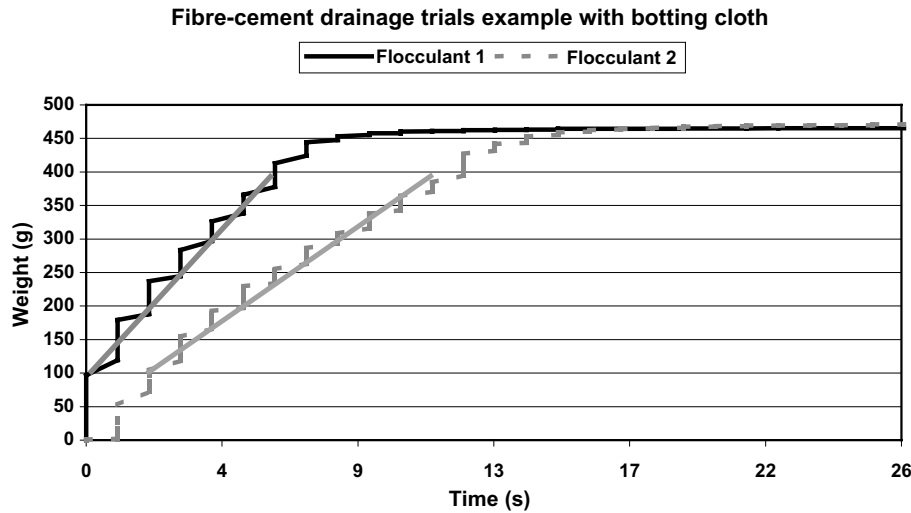


Fig. 8. Drainage curves obtained with the VDT.

present of other compounds in the formulation) and this information is very difficult to know as in many occasions the suppliers do not provide enough data.

The effects of flocculant on retention and drainage were obtained from the results of the VDT in the second step. Combining the two-steps it will be possible to determine the best flocculant for each specific fibre–cement suspension. The dewatering process, as weight evolution versus time, is represented in Fig. 8. From the slope, it is possible to calculate the dewatering rate. The retention values (using as the filter a botting cloth), the final water content (using as the filter a felt) and the slopes of the drainage curves (in both cases) for the different flocculants are summarized in Table 2.

With some flocculants the retention value is too low. As a consequence, if they are used at industrial scale, a high mineral fines accumulation into the water system is expected. These flocculants are therefore not suitable for fibre–cement production. The best values were obtained

using flocculants B, C, D or M (retention values higher than 96% in Table 2).

Looking at the dewatering rate, as the slope of the curve increases the machine speed can increase and hence the production rate. Lower dewatering rates lead to lower machine speeds and lower productivity. Therefore, these flocculants are unsuitable for use on an industrial scale. The best values were obtained using flocculants B, C and D (higher values of drainage rates in Table 2 for both cases, felt and botting cloth).

Finally, the final water content in the sheet should have intermediate values. There are two important problems for the operator in the fibre–cement production, known informally as elephant skin appearance and delaminating. These problems can be controlled through a proper flocculant selection. Usually elephant skin appearance is visible when the green sheet has a high humidity, this problem is more frequent and important in suspensions with a high amount of fine particles

Table 2  
Retention, drainage and final sheet water content for different flocculants

Flocculant	Water retention (%)	Solids retention (%)	Drainage rate with felt (g/s)	Drainage rate with botting cloth (g/s)
A	45.05	95.00	16.85	35.46
B	45.35	96.80	17.82	43.17
C	45.50	96.10	19.56	52.66
D	47.00	96.40	19.33	48.61
E	45.59	94.53	18.01	36.10
F	47.57	94.00	15.62	40.84
G	44.37	95.20	15.08	34.60
H	45.06	95.60	14.89	45.80
I	45.55	94.80	15.08	38.60
J	46.63	93.80	14.12	41.32
K	45.14	94.80	17.71	40.05
L	49.64	93.20	10.18	19.09
M	45.45	97.50	14.35	34.71
N	48.74	94.93	17.59	31.24

(like silica fume or kaolin) as is the case of the composition used to produce corrugate sheets using air cured technology. By the contrary, if the humidity is very low, then delamination problems will appear, being this problem more important and usual for suspensions with low amount of fine particles as, for example, in the production of flat product using autoclave curing technology. Analysing the data flocculants A, B, C, D, E, H, I, J, K and M would be suitable (Table 2).

Combining the results, the flocculant D could be selected as most suitable for the fibre-suspension analysed. In the case when machine speed is not a bottleneck, flocculant M could also be considered as an alternative.

#### 4. Conclusions

A valuable methodology has been developed to study flocculant behaviour in a fibre-cement industry, giving the opportunity to explore new and attractive interactions to improve final product properties and machine runnability.

Although in this work a fibre-cement suspension has been used representing the most complex situation, it is also possible and could be simpler to study individual raw materials in order to select the best one before using them in the process, e.g. different types of cements, pulps, pigments or any other minerals used as raw materials.

Once the methodology has been proved at laboratory scale the next step will be its validation at a mill site. It is planned to use an online FBRM probe as a sensor to follow flocculation in an industrial Hatschek machine.

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