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Effect of fibre morphology on flocculation of fibre–cement suspensions

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ABSTRACT

The objective of the present research was to evaluate the effect of fibre morphology (e.g., length, width, fibrillation, broken ends, content of fines and number of fibres per gram) on flocculation and drainage properties of fibre–cement suspensions and on physical properties of the fibre–cement composites. Mechanical refining was used to change the morphological properties of Eucalyptus and Pinus pulps. Results show that the mechanical refining increased the size of the formed flocs and decreased the concentration of free small particles (with dimensions between 1 and 20 µm) as a consequence of the increased fibrillation and content of fines, which increased the capacity of the fibres to capture the mineral particles. High levels of refining were necessary for Pinus pulp to obtain cement retention values similar to those obtained by unrefined Eucalyptus pulp. This is due to the higher number of fibres per gram in Eucalyptus pulp than in Pinus pulp. Pulp refining improved the packing of the particles and, although decreased the drainage rate, it contributed to a less porous structure, which improved the microstructure of the composite.

1. Introduction

The different chemical composition and hygroscopic character of cellulose fibres make the compatibility between fibres and cement particles highly complex [1,2]. In the Hatschek process for fibre–cement fabrication, the behaviour of cellulose fibres depends on their morphological properties. Changes in the source of fibres may entail changes in the industrial process due to its effect on mineral fines retention, suspension dewatering, sheet formation and, as consequence, on the overall efficiency of the machine.

In general, softwood fibre (mainly *Pinus radiata*) has replaced asbestos fibre as the reinforcing element in commercial autoclaved cement products. In some countries, this fibre has a reasonably high participation in the production cost due to its market price. Thus, considerable research effort has been put into the study of fibres from fast growing wood as cheaper alternatives for the fibre supply [3]. In tropical countries Eucalyptus is a fast grown wood species with good fibre qualities and cheaper market price in comparison to softwood options. Although Eucalyptus pulp has been widely employed in the paper industry throughout the world, there is limited information in the scientific literature concerning its use as reinforcement in fibre–cement. In the future, due to the growing demand of fibres, it will be interesting for the industry to rely much more on currently less demanded short fibres species, e.g., hardwood species, for their products. However, the optimum pulp freeness of Eucalyptus fibres and its effect on flocculation and drainage of the fibre–cement suspension are unknown and no systematic studies have dealt with the effect of fibre morphology on fibre–cement flocculation. To promote its use, it is essential to study those fibre properties which provide optimum performance in fibre–cement manufacture. Additionally, there is an increasing interest to gain a more in-depth knowledge of the different kinds of fibres available for the reinforcement of composites.

Therefore, the objective of the present research was to evaluate the effect of different fibre morphologies (e.g., length, width, fibrillation, broken ends, content of fines and number of fibres per gram) on natural aggregation, drainage properties, particles retention of fibre–cement suspensions and physical properties of the composites produced.

2. Materials

2.1. Pulp and fibre properties

Conventional bleached and unbleached Eucalyptus and Pinus kraft pulps were used in the experiments. Kappa number of the pulps was determined following the SCAN C 1:77 [4] Standard. The total residual lignin content (TRLC) was evaluated using the kappa number according to: TRLC = (kappa number)/6.546, as described by Laine et al. [5]. The wood extractives of the pulps were determined following the Tappi T 204 cm-97 [6] Standard. The mean viscosity of the pulps was determined in cuproethylendiamine diluted solution [7]. The total residual lignin content (TRLC), the wood extractives content of the pulps and the mean viscosity are presented in Fig. 1.

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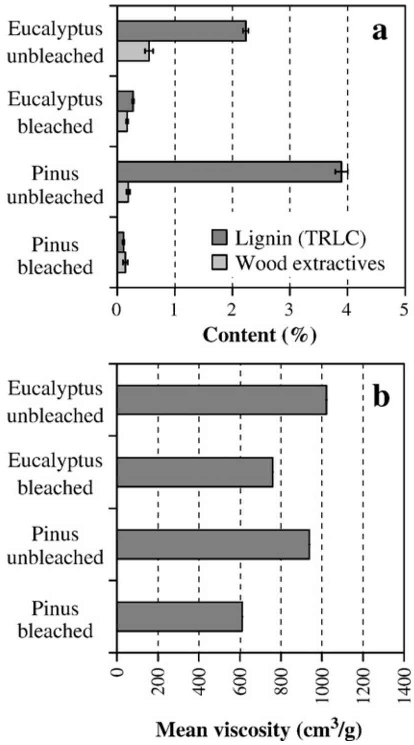

Fig. 1. a) Total residual lignin content (TRLC) and content of wood extractives in the unrefined pulps; b) Mean viscosity of the unrefined pulps.

Pulps were disintegrated in distilled water (3000 revolutions) before refining, or before using them in the fibre–cement suspensions.

2.2. Fibre–cement suspensions

Fibre–cement suspensions, with concentration of 50 g/L were prepared at 28 °C using the following constituents (percentage by dry mass): 10.0% of pulp (at the different refining intensities), 77.2% ordinary Portland cement (OPC) and 12.8% ground calcium carbonate (GCC).

3. Experimental

3.1. Refining of the cellulose pulp

Pulps with fibre concentration of 100 g/L were refined in a standard PFI lab refiner following the procedures described by Tappi T 248 sp-00 [8] Standard. Pulps were refined from around CSF 700 mL to CSF 70 mL. The CSF values were determined after each refining level following the Tappi T 227 om-99 [9] Standard. The Canadian Standard Freeness test (CSF) is a widely recognized standard measure of the drainage properties of the pulp suspensions [10] and it relates well to the initial drainage rate of the wet pulp pad during the dewatering process. Low freeness (CSF values less than 300 mL) is indicative of high degrees of external fibrillation and/or shortage of the fibres, leading to long drainage periods during the test.

3.2. Pulp characterization (morphological properties)

Fibres were analyzed by a Pulptec™ MFA-500 Morphology Fibre and Shive Analyser — MorFiTrac. This equipment consists basically of a charge-coupled device (CCD) camera that captures the images of the fibre/water suspension and records them for further analysis with a software that carries out the measurements and statistical corrections. Fibres diluted in water (at around 25 °C) flow into a measuring cell between two sapphire glass plates. The flow gap, 1.5 mm × 30 mm, is larger than the usual capillary systems (0.5 mm diameter). This large cell allows analyzing rather big elements while avoiding a long preparation of the pulp sample. The non-polarized light beams across the glasses and projects the images of the flowing fibres via a zoom lens on the CCD camera, located in the back side. The images are crowded with fines and fibres. The software discriminates between fibres and fines through size criteria (length and width). A fine element was considered as any detected object present in the pulp with dimensions lower than those of fibres, i.e., length under 200 µm or width under 5 µm. The length values are calculated from the framework of the fibre following the course of each segment. It is consequently the real developed length of the fibre. The length weighted in length (L_{wl}) is given by Eq. (1).

$$L_{\mathrm{wl}} = \sum_{i=1}^{n} (L_i \cdot L_i) \Big/ \sum_{i=1}^{n} L_i \qquad (1)$$

Where n is the number of fibres and L_i is the length of each fibre ($1<i<n$).

3.3. Natural aggregation of fibre–cement without flocculant

Dimensions and concentration of particles in the suspension were obtained by using a focused beam reflectance measurement system FBRM M500LF, Lasentec®, manufactured by Mettler Toledo, Seattle, USA. The FBRM instrument operates by scanning a laser beam highly focused in a focal point that moves with a circular trajectory at a fixed speed, across the particles in the suspension measuring the time duration of the reflected light from these particles [11–13]. The FBRM provides the chord length distribution of the particles in the suspension in real time, in a wide concentration range. This distribution depends on the shape, size and concentration of particles. Each measured chord is denominated “count”. In a typical trial, the probe was immersed in 400 mL of fibre–cement suspension stirred at 800 rpm during 720 s. Then, stirring rate was reduced to 400 rpm to encourage the natural aggregation of particles and fibres. The median chord size and the number of counts per second were obtained after 180 s of stirring the suspension at 400 rpm, because at this time the values of these statistics were more stable. This indicates that equilibrium between the aggregation of particles and fibres and the floc breakage had been reached and only cement hardening was taking place. The median of the chord size distribution gives information about the particles' size, being the total number of counts per second a function of the concentration of particles in the suspension [14].

3.4. Vacuum drainage tester (VDT)

The retention and drainage studies were performed with a vacuum drainage tester (VDT) previously described by Negro et al. [2]. This equipment consists basically of two jars separated by a barrier (usually a latex membrane): the upper jar is used to keep the fibre–cement suspensions stirred until homogenization. After the necessary stirring time, the barrier is removed and the suspension is drained to the second jar in which a filter is located. In this case a botting cloth (18 mesh) was used in order to simulate the dewatering in the vat of the Hatschek machine. The suspension is drained under a certain vacuum (around 0.09 MPa) through the filter and a computerized balance records the mass of drained water over time. The drainage curve is analyzed in order to obtain the drainage rate for the different fibre–cement suspensions studied (pulps at several refining degrees).

A 500 mL volume of 50 g/L fibre–cement suspension at the temperature of around 28 °C was used. Fifteen minutes after homogenization (stirring at 800 rpm and 400 rpm), the dewatering process started and the evolution of the filtrate weight was recorded in a personal computer. The slope of this curve results in the drainage rate. Finally, the solids retention and the water content in the cake were determined by gravimetric measurements.

3.5. Composite preparation and physical characterization

Cement based composites were prepared using unbleached Eucalyptus or Pinus pulps. The cement based composites were moulded in plates measuring 200 mm × 200 mm and around 6 mm thick. They were prepared at laboratory scale using a slurry vacuum dewatering followed by the pressing technique described in details by Savastano Jr. et al. [3].

Apparent porosity and bulk density values were obtained from the average of six specimens for each mix design, following the procedures specified by the ASTM C 948-81 [15] Standards.

4. Results and discussion

4.1. Effect of refining on pulp freeness

Minimal differences on CSF values were noticed between bleached and unbleached Eucalyptus pulps (Fig. 2). Differences in the refining curves (CSF value versus PFI revolutions used in the refining procedure) for bleached and unbleached Pinus pulps are related to the greater viscosity loss promoted by the bleaching process (Fig. 1b). Bleaching extracted compounds from the fibre structure, e.g., residual middle lamella, making the fibrils from the primary cell wall more sensitive and exposed to the mechanical treatment provided by refining.

4.2. Effect of refining on fibre length distribution and content of fines

The fibre length distribution of the unbleached Eucalyptus and Pinus pulps is presented in Fig. 3. It is observed that 70% and 99% of the fibres in the unrefined Eucalyptus pulp are shorter than 1 mm and 2 mm, respectively. Refining did not change severely this distribution which indicates that, at the experimental conditions, refining did not cut Eucalyptus fibres severely. Around 10% and 30% of the unrefined Pinus fibres are lower than 1 mm and 2 mm, respectively. Refining of the Pinus pulp changes notably the fibres length distribution, increasing the content of fibres lower than 1 mm and 2 mm to 30% and 60%, respectively. This increase is due to the shortening of fibres and consequent fibre debris.

The effect of refining on the different morphological properties is presented in Fig. 4. The shortening of the Pinus fibres occurs even in the first levels of refining (Fig. 4a). The fibre shortening, presented in Fig. 4a, produces small particles (lower than 200 µm) commonly called fines, which also contain band-like materials from both primary wall and secondary wall layers of the fibres [16]. The content of fines in Pinus pulp increased substantially with refining (Fig. 4b). Fines do not contribute significantly to fibre–cement strength but act more as filler [17] and, in most cases, influence negatively the drainage of the suspension.

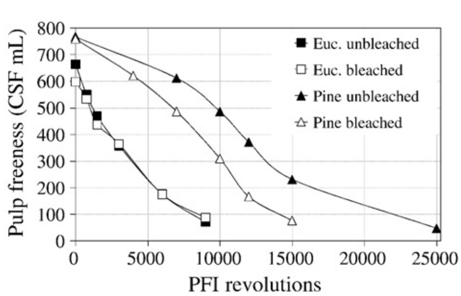

Fig. 2. Effect of refining on pulps' freeness.

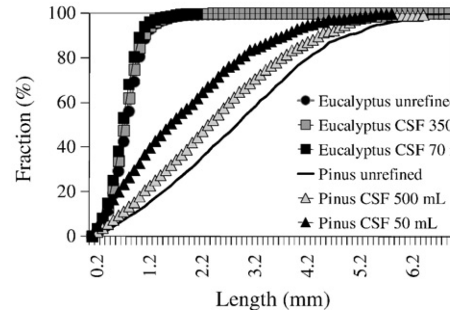

Fig. 3. Accumulated length distribution of the pulp fibres.

Fig. 4b shows that the fines contents in the Eucalyptus pulps vary from 25% in the unrefined pulp to around 30% in the refined pulps. Nevertheless, in the Pinus pulps the contents of fines after severe refining come close to 60%. Fig. 5 depicts the fines length distribution of the unrefined Eucalyptus and Pinus pulps. In the case of Pinus pulp, around 60% of the fines is smaller than 40 µm, while in the Eucalyptus pulp this content is around 40%.

4.3. Effect of fibre morphologies on natural aggregation

Beghello and Eklund [18] demonstrated how fibre concentration and fibre length distribution, among other variables, affected the state of dispersion of papermaking fibres. Also, aggregation and floc properties of fibre–cement suspensions should depend on the morphological properties of the fibres. Therefore, the correlations between the different morphological properties and the properties of the flocs were analyzed.

Fig. 6 shows the effect of refining on the behaviour of fibre–cement suspension. As it was expected, in general, pulp refining improved the agglomeration of the cement suspensions. Therefore, refining decreased the number of counts per second between 1 µm and 20 µm, representing free small particles (Fig. 6) and increased the median chord size (Fig. 4).

Bleached Pinus pulps provided to the fibre–cement suspension a higher median chord size (Fig. 4) and lower number of counts (Fig. 7) than unbleached Pinus pulps with similar morphological properties. This indicates that bleaching increases the interactions of fibres with cement. Bleaching chemically degrades the external layer of fibres, which contains wood compounds that could affect cement hydration and coagulation. Furthermore, the degradation of this layer makes the fibre easier to be modified by refining action, increasing its effect on fibrillation. The effect of bleaching was not observed in the size of the flocs or agglomerates in the Eucalyptus suspensions because of the higher number of fibres in the Eucalyptus pulp in relation to Pinus pulp for the same mass of fibres.

Refining increased the average width of Eucalyptus fibres (Fig. 4c) and the median chord size of fibre–cement suspension, indicating an increase of the aggregation. The increase of fibre width by the outer cell wall swelling and internal fibrillation after refining [19,20] should lead to an increase of fibre volumetric concentration. Therefore, the increase of the yield stress of suspension is expected, i.e., the force needed to break the fibre network, at a given mass concentration [21]. In the case of Pinus fibres, the average width of the fibres is around two times higher than in Eucalyptus and it decreased with the refining, as a consequence of the removal of the primary and even the secondary cell wall. No linear correlation was observed for the fibre width with the median chord size and counts of chords per second in the Pinus fibre–cement suspensions (Fig. 4c).

One of the effects of interest of refining on cellulosic fibre structure is the fibrillation of the fibres surface as a result of mechanical action [22]. Microfibrils are detected fibrils attached to the fibre and were expressed as percentage in length of microfibrils. Broken ends are

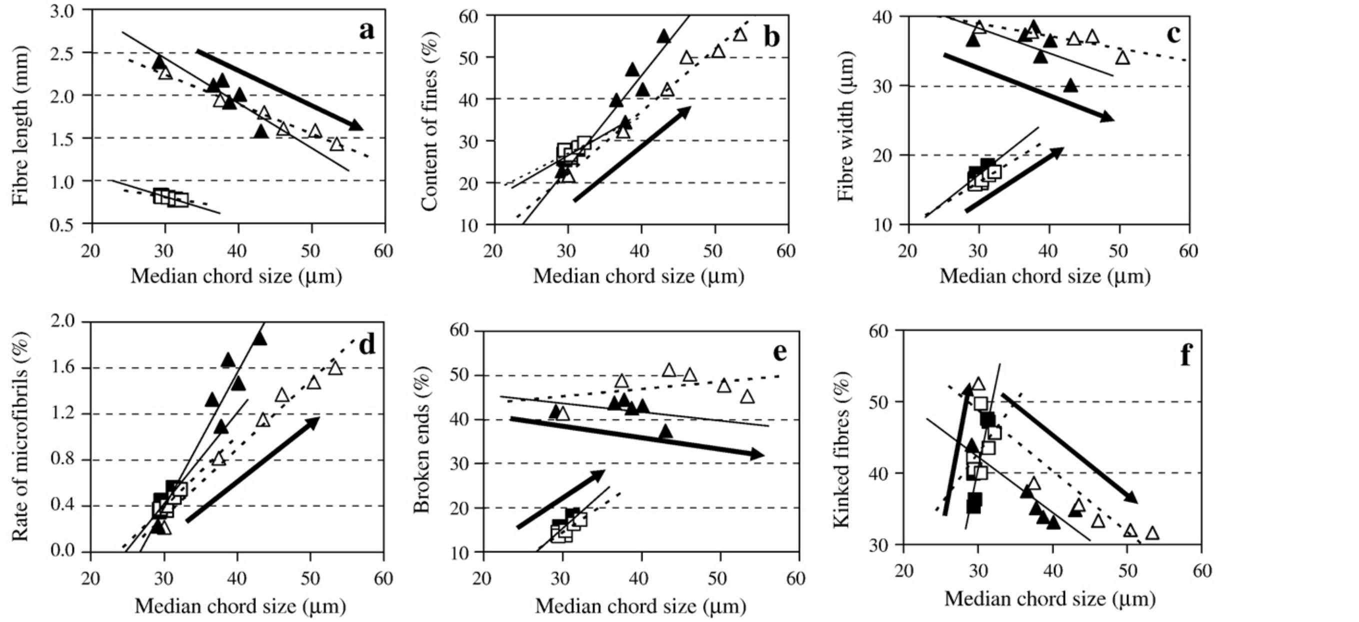

Fig. 4. Fibre length weighted in length (a); content of fines (b); fibre average width (c); amount of microfibrils (d); content of broken ends (e) and content of kinked fibres (f), in relation to median chord size of the fibre–cement suspensions. Legend: ■ and □ represent Eucalyptus unbleached and bleached pulps respectively; ▲ and Δ represent unbleached and bleached Pinus pulps respectively. Arrows indicate the increasing of refining.

detected as fibres having fibrils on their ends. Both microfibrils and broken ends increase the surface area of fibres, which turns them more reactive with the particles present in the suspensions. As observed in Fig. 4d, fibrillation increased with refining. The fibrillated fibres contribute to the formation of a net inside the mixture with the consequent retention of the cement particles during the stirring and drainage processes. Swelling also contributes to increase the interaction of fibres with other particles. Consequently, the median chord size and counts per second correlate linearly with the amount of microfibrils for both Eucalyptus and Pinus pulps (Figs. 4d and 7c, respectively). Broken ends correlated linearly with median chord size (Fig. 4e) for Eucalyptus pulp suspensions, but not for the Pinus fibre–cement suspensions, because high refining cuts fibres, thus decreasing again the percentage of broken ends. However, the cutting of Pinus fibres and the increase in fines contents contribute to increase the surface area available to interact with minerals. As a result, the median chord size increased (Fig. 4a) and the number of counts decreased (Fig. 7a) when the length weighted by length decreased. The percentage of fines in Pinus pulps showed good linear correlation with the median chord size and with the number of counts per second in the fibre–cement suspensions prepared with Pinus fibres (Figs. 4b and 7d, respectively), while no good correlation was found in the Eucalyptus pulp suspensions.

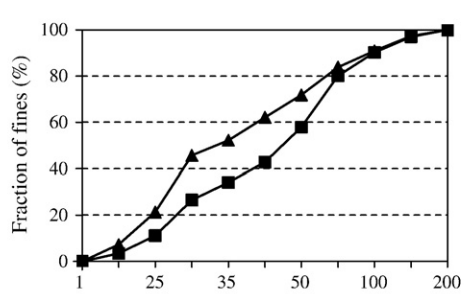

Fig. 5. Accumulated length distribution of the fines in the unrefined pulps. Legend: ■ and ▲ represent unbleached Eucalyptus and Pinus pulps, respectively.

Kinks are abrupt angle changes within short distance of the heart line of the fibre. Kinks are generally considered as the first favourable point to fibre rupture. According to Page and Tydeman [23] the major effect during refining is the straightening-out of both the long and short-range kinks, crimps and curls that are created in fibres during pulping and bleaching. In papermaking, removal of these kinks and curls in the fibres greatly improves the stress distribution in the sheet [23]. In the present research only refining of Pinus pulp decreased kinked fibres and showed a good linear correlation with the median chord size (Fig. 4f). In the case of Eucalyptus pulp, refining increased the percentage of kinked fibres (Fig. 4f) and, therefore, the risk of fibre rupture, because of the inclusion of new points of tension.

4.4. Effect of natural aggregation on retention and drainage of the fibre–cement suspensions

Fig. 8a presents the effects of natural aggregation in the drainage rate with botting cloth performed in VDT. As expected, drainage rate of fibre–cement suspension decreased with pulp refining. The higher content of fines in the highly refined pulps fills up the empty spaces during the formation of the cake, which makes difficult the dewatering process and, consequently, increases the water retention

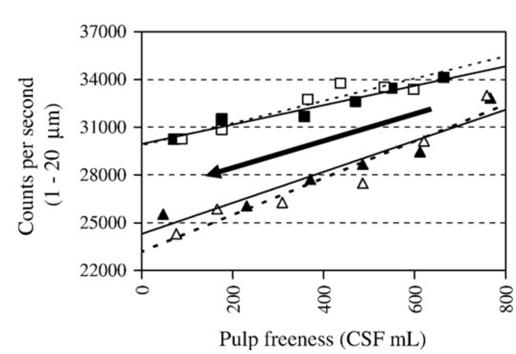

Fig. 6. Counts of chords per second in the range between 1 and 20 μm in relation to pulp freeness (refining level). Legend: ■ and □ represent Eucalyptus unbleached and bleached pulps respectively; ▲ and Δ represent unbleached and bleached Pinus pulps respectively. Arrow indicates the increasing of refining.

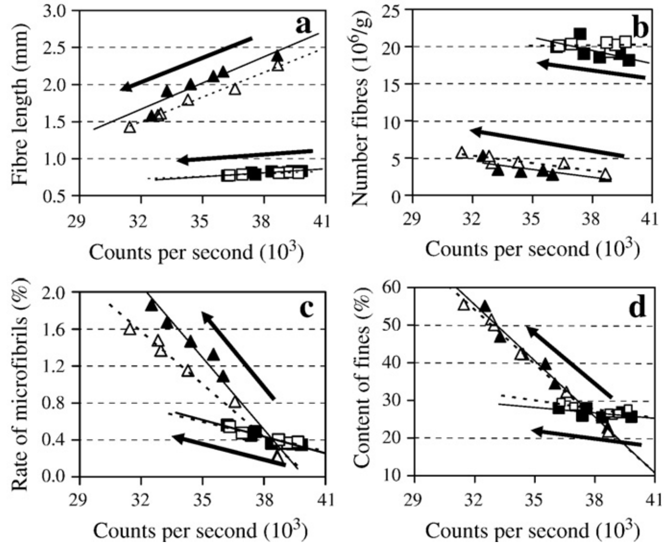

Fig. 7. Length weighted in length (a); number of fibres per gram (b); amount of microfibrils (c) and content of fines (d) as a function of counts of chords per second in the fibre–cement suspensions. Legend: ■ and □ represent Eucalyptus unbleached and bleached pulps respectively; ▲ and Δ represent unbleached and bleached Pinus pulps respectively. Arrows indicate the increasing of refining.

in the cake after dewatering (Fig. 8b). Furthermore, the higher rate of fibrillated fibres in refined pulps leads to the improvement in the cement particles retention (Fig. 8c). Pulp refining improved the packing of the particles favouring a less porous structure of the flocs, which also contributes to the decrease of the drainage rate.

As it was expected, it is possible to improve solids retention by Pinus pulp decreasing its pulp freeness, e.g., increasing refining (Fig. 8c). In the present experimental conditions, high levels of refining were necessary for Pinus pulp to obtain solids retention values similar to that obtained by unrefined Eucalyptus pulp (Fig. 8c). In the cases which correspond to the highest values of solids retention, the content of fines is twice and the rate of microfibrils is fivefold higher in Pinus pulp than in Eucalyptus pulp.

Higher solids retention (around 90%) in suspensions with unrefined Eucalyptus pulp is due to the higher number of fibres per gram (four times higher) in relation to Pinus pulp (Fig. 7b), which provide them with a higher capacity to capture the cement particles. The higher number of fibres per gram in Eucalyptus pulp did not prejudice the drainage rate of the fibre–cement suspensions when compared to refined Pinus pulps (Fig. 8a).

4.5. Effect of natural aggregation on physical properties of the fibre–cement composites

Fig. 9 shows the physical properties of composites reinforced with Eucalyptus and Pinus pulps at different refining levels as a function of the median chord size of the fibre–cement suspensions. As previously reported by Negro et al. [13], the measurements in the FBRM were highly influenced by the structure of the flocs. The structure of the flocs achieved by the refined pulps was detected by the probe as higher median chord size values and lower counts per second. This occurred due to the fact that refining improved the packing of the particles and it favoured a less porous structure, as shown by the decrease in apparent porosity with the increasing median chord size (Fig. 9a). Consequently, the higher the median chord size in the suspensions the higher the bulk density of the composites (Fig. 9b). In the case of Pinus fibre–cement composites, the apparent porosity was significantly decreased even in the first level of refining, due to the sudden increase of the median chord size (from 29 μm to 36 μm) of the fibre–cement suspensions (Fig. 9a). As previously presented,

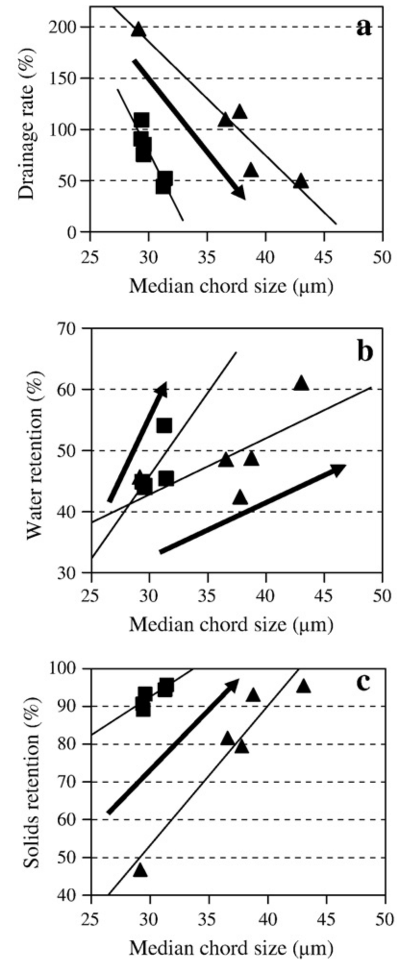

Fig. 8. Drainage rate of the cake (a); water retention in the cake (b) and solids retention (c) as a function of median chord size of the fibre–cement suspensions. Legend: ■ and ▲ represent unbleached Eucalyptus and Pinus pulps, respectively. Arrows indicate the increasing of refining.

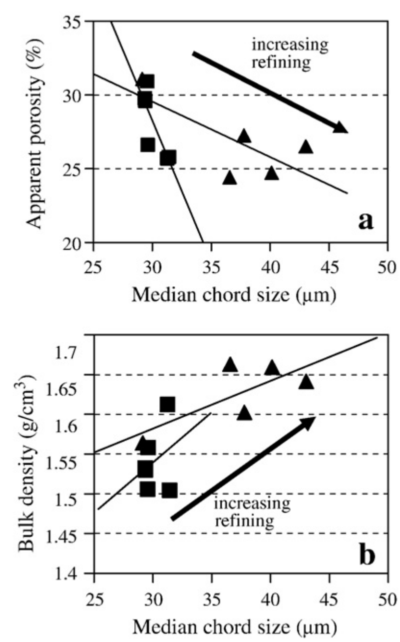

Fig. 9. Apparent porosity (a) and bulk density (b) of the fibre–cement composites as a function of the median chord size of the fibre–cement suspensions. Legend: ■ and ▲ represent unbleached Eucalyptus and Pinus pulps, respectively.

shortening (from 2.4 mm to 2.2 mm), amount of microfibrils (from 0.2% to 1.1%), broken ends (from 42 to 45%) and fines content (from 23 to 35%) of the Pinus fibres also increased in the first level of refining.

5. Conclusion

Mechanical refining was used to change the fibre morphology (e.g., length, width, fibrillation and content of fines) of the pulps. Fibre length distribution of the Pinus pulp was more affected by refining than Eucalyptus pulp. Higher shortening, fibrillation and fines generation were observed in the refined Pinus pulps. Fibrillation, and fines generation in refined pulps increased the size of the formed flocs in the fibre–cement suspensions and decreased the concentration of the free small particles (between 1 μm and 20 μm), which increased the capacity of the fibres to capture the mineral particles, increasing solids retention. High levels of refining were necessary for Pinus pulp to obtain cement retention values similar to those obtained by unrefined Eucalyptus pulp. Higher cement retention in suspensions with unrefined Eucalyptus pulp is due to the higher number of fibres per gram (four times higher) in relation to Pinus pulp, which provide them with a higher capacity to capture the cement particles. Pulp refining improved the packing of the particles favouring a less porous structure of the flocs that decreased the drainage rate. Denser flocs led to lower apparent porosity in the composites and, consequently, to increased bulk density, which improved the microstructure of the composite.

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