CHARACTERISATION OF AGRICULTURAL RESIDUES USED AS A SOURCE OF FIBRES FOR FIBRE-CEMENT PRODUCTION

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ABSTRACT

Nowadays, certain components of non-wood annual plants such as corn stalk and industrial hemp core are considered waste materials or used in low value applications; both by-products have a very low cost. On the other hand, given the large quantities of these materials generated worldwide and their renewable character, it is reasonable to explore new routes for their exploitation. The aim of this paper is to study the potential of both corn stalk *(Zea Mays L.)* and industrial hemp core *(Cannabis Sativa L.)* fibres as a renewable source of cellulose fibres in the production of fibre-cement. For each source of fibres, a number of chemical cooking treatments were studied. The morphological properties of the fibres were determined using a scanning electron microscope and a fibre and pulp morphological analyser. Pulp refining was carried out in a PFI mill to improve the characteristics of the fibres. In the case of corn fibres, different degrees of refining were applied. The fibre flocculation process was investigated using several polyacrylamides. The process was studied by monitoring the chord size distribution in real time by means of a focused beam reflectance measurement probe.

The results indicated both pulps can be used for the production of fibre-cement, having the two types of pulp morphological similarities with the pine fibres currently used.

Through the flocculation process it was concluded the floc size depends on the length of the fibres.

Keywords: *Cannabis Sativa L.*; *Zea Mays L*; natural fibres; fibre-cement; morphological characterisation

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1. INTRODUCTION

In recent years, there has been a growing interest in the use of natural fibres in composite materials. This is the case of the fibre-cement industry, especially after the use of asbestos as reinforcing fibres was forbidden due to the health problems. Since then, new technologies for the production of fibre-cement using cellulose and synthetic materials as reinforcement fibres have been developed. Among the potential substitutes for asbestos, four types of fibres are distinguished by their importance, namely steel fibres, glass fibres, synthetic fibres and natural fibres.

Fibre-reinforced cement with steel fibres. They are materials characterised for their high resistance, which increases with the fibre length. However, an excessive length may lead to the formation of kinks in the fibres, reducing the mechanical properties of the resulting material. Steel fibres improve significantly the mechanical characteristics of mortars and concrete, in terms of impact strength and toughness. Tensile strength, flexural strength, fatigue strength and ability to resist cracking and spalling are also increased (Qi-sheng et al., 2008; Song and Hwang, 2004; Teng et al., 2008).

Fibre-reinforced cement with glass fibres. The main characteristic of glass fibre-reinforced cement composites is the durability of their fibres in the alkaline environment of cement. The long term properties of glass fibre-reinforced cement composites are the reduction in strength and ductility with time. The poor durability of glass fibre-reinforced cement is generally attributed to: (a) degradation of the fibres by hydroxyl ions from the hydrating cement matrix, (b) precipitation of hydration products, especially calcium hydroxide and, (c) densification of the

matrix at the interface (Bentur and Mindess, 1990; Marikunte et al., 1997; Purnell et al., 2000).

Fibre-reinforced cement with synthetic fibres. The mechanical properties of materials from synthetic fibre-reinforced cement depend largely on the properties of the individual fibres. Several types of fibres have been tested as reinforcement, among which acrylic, aramid, carbon, nylon, polyester, polyethylene and polypropylene fibres. The improvement in the properties achieved through synthetic fibre-reinforce cements varies, for example, carbon fibres have been used as fillers in cement matrix composites to improve both mechanical and electrical behaviours as well as the electromechanical and electromagnetic behaviours. They can be used in cement together with steel fibres, as the addition of short carbon fibres to steel fibre-reinforced mortar increases the fracture toughness of the interfacial zone between steel fibres and the cement matrix (Guan et al., 2006; Jingyao and Chung, 2001; Laukaitis et al., 2009; Song et al., 2005; Wang et al., 2008;).

Fibre-reinforced cement with natural fibres. In recent years, considerable efforts have been made to develop natural fibre-reinforced cement composites for affordable infrastructures: from plants, minerals and/or animals. However, the long-term durability of natural fibre-reinforced composites may be limited due to their high permeability and lack of resistance to crack growth, particularly vegetable with fibres obtained from agricultural by-products (Angelini et al., 2000; Savastano et al., 2009). Vegetal fibres as bagasse from sugar cane, bamboo and coconut fibres may be used as an alternative material due to their biodegradability, low specific mass, low cost and availability (Guimarãesa et al., 2009).

Detailed knowledge of the fibre morphology is necessary to evaluate its potential application and the product quality (Bartl et al., 2004; Oksanen et al., 2000).

On the other hand, flocculation processes are of great importance in the actual manufacture of fibre-cement, because they affect the formation of plaque, the retention of solids and the drainage of the suspension (Fuente et al., 2010). The flocculants used are generally anionic polyacrylamides with high molecular weight and low-medium electrical charge that induce flocculation by bridges (Jarabo et al., 2010a; Negro et al., 2005; Negro et al., 2006a).

In this paper, fibres from agricultural residues, specifically *Zea Mays L.* and *Cannabis Sativa L.*, have been characterised morphologically to evaluate their potential application as alternative sources of cellulose as reinforcement fibre in fibre-cement manufacturing. For this application the most important properties are morphological characteristics (length, width and presence of microfibrils) and the content of fine particles. These properties have been compared with those of pine fibres commonly used in fibre-cement manufacture (Silva et al., 2008).

Moreover, the refining of these fibres has been also studied due to its effects on internal and external fibrillation, fines formation, fibre shortening and fibre curling or straightening, which favours the bonding with the cement particles (Gil et al., 2009; Sain and Fortier, 2002; Vidal et al., 1999).

The select the most efficient pulp suspension, the flocculation and floc properties of the different pulp suspensions with anionic and cationic polyacrylamides were studied by a focused beam reflectance measurement technique (FBRM), whose evolution enables to monitor flocculation.

2. MATERIALS AND METHODS

2.1. Materials

The raw materials used in this study were corn stems *(Zea Mays L.)* and industrial hemp straws *(Cannabis Sativa L.).* As reference, unbleached pine kraft pulp refined at 35 °SR was used. Tables 1 and 2 show the cooking conditions applied to obtain both the hemp and corn pulps by an Organosolv process using ethanolamine and ethanol as solvents, respectively (Akgul and Tozluoglu, 2010; Anatoly and Pereira, 2005; Pan et al., 2006).

An anionic polyacrylamide (APAM) with a high molecular weight of $7.4 \cdot 10^6$ g/mol and a cationic polyacrylamide (CPAM) with a medium molecular weight of $5 \cdot 10^6$ g/mol commonly used in the industrial Hatcheck process were used. They were dissolved in distilled water to prepare solutions with a concentration of 1.5 g/L.

2.2. Methods

2.2.1. Characterisation by a Scanning Electron Microscopy

The morphological characterisation of the fibres was carried out by a scanning electron microscope (SEM), JEOL, mod. JM-6400. Each sample was placed on a cylindrical slide and placed in a vacuum oven for 24 hours to be dried. After drying the sample was coated with gold. Then, it was introduced in the SEM and was visualised with a magnification of 100, 500 and 1500 (Li et al., 2009).

2.2.2. Characterisation by a Morphological Analyser

The morphological characterisation of fibres was performed using a fibre and pulp morphological analyser, Morfi, V7.9.13.E (Techpap, France). The parameter limits to measure these fibres were set as follows: length between 100 and 6000 µm and for the width, between 5 and 75 µm. The morphological performed through the equipment is based on characterisation an image analysis system, consisting of a

diode that emits unpolarised light. Imaging is performed until the equipment counts 5000 fibres, which is the optimum value for subsequent statistical analysis using a computer software. The final results are obtained both numerically and graphically. The samples were analysed using a specific program to determine different parameters of the fibres and pulps (Jarabo et al., 2010b; Moral et al., 2010).

The samples for morphological characterisation were prepared by adding 1 g of dry corn or hemp to 600 mL of water and homogenising in an ENJO-model 692 lab disintegrator.

The characterisation was done in duplicate. The average values of the two measurements are shown. Although a large number of parameters are measured by this equipment, the parameters selected for the comparison of the different raw materials were the length and the width of fibres, percentage of microfibrils and fines number.

2.2.3. Characterisation by Focused Beam Reflectance Measurement

The flocculation of the different pulps prepared was studied using a commercial FBRM-M500L, S400 IP 14/206 manufactured by Mettler Toledo, Seattle, USA. The FBRM device measures the chord length distribution in real time over a wide range of solid concentrations in the suspension. A laser beam is projected through the probe and focused on the focal point by means of a rotating lens. The focal point describes a circular path on a 20 μm plain outside the surface of the probe, immersed into the suspension, at high rotation speed (2000 rpm). Every time a particle in suspension crosses the circular line circumscribed by the focal point, light is reflected from the surface of the particle and it reaches the detector. A computer calculates the chord length of the particle on the basis of the reflectance

time and focal point speed. Each measurement provides a particle chord length distribution representative of the size and shape of the population of particles in the suspension. The flocculation process of the fibres and fillers in the manufacture of cement, allows the real-time monitoring of all processes involving a change in particle size. The addition of flocculant allows observing the changes that occur in the solution during the flocculation process (Blanco et al., 2002; Negro et al., 2006b).

In a typical trial, the probe was immersed in 400 mL of fibre suspension, prepared with $Ca(OH)_2$ saturated water and stirred at 800 rpm. After 6 min., the stirring intensity was reduced to 400 rpm and maintained for 2 minutes, after which 100 ppm of PAM were added to induce the floc. The evolution of the flocs was subsequently studied at 400 rpm for 4 min.

2.2.4. Refining process

The refining process of corn stalks fibres was carried out under controlled conditions, using the PFI mill (ISO 5264/2-2003). The industrial hemp core was not refined due to its high hardness. The refining of corn residue pulp was performed in three stages:

Disintegration of the pulp by means of a lab disintegrator ENJO model 692 according to ISO 5263 and ISO/DP 625: 30 g of dried pulp were disintegrated in 2 L of water to obtain a final pulp consistency of 1.5%. It was programmed to disintegrate at 9000 revolutions per minute.

Pulp refining was performed by a PFI mill at 10% consistency according to ISO 5264/2. The revolutions number applied in each corn pulp is summarised in Table 3.

The degree of pulps refining was measured using a Canadian Standard Freeness (CSF), according to ISO 5267/2.

3. RESULTS AND DISCUSSION

3.1. Morphological characterisation

Each fibre sample was characterized by duplicate. The variability of the morphological parameters (RSD) was determined by Equation [1] (Spiegel, 1993).

$$RSD\ (\%) = [(X_i - X_{average})/\ X_{average}] \cdot 100 \qquad [1]$$

Where, *RSD* is the variability of the parameter (%); X_i represents the measured value; and $X_{average}$ represents the mean value of the two measurements. Length and width measurements showed less than 0.5% of variability and microfibrils and fines number measurements around 2%.

3.1.1. Hemp core

The morphological characterisation results of hemp core and pine pulps, obtained by Morfi are shown in Table 4.

The length and the width of hemp fibres are very similar to each other, but a slight decrease in the value of these parameters was observed when cooking conditions were harsher. The most important factors affecting these parameters are temperature and time since the ethanolamine concentration is constant (Pulp A and B) and the morphological parameters will change (Table 1 and 4). Hemp fibres were shorter than pine fibres in all cases. However, the value of hemp fibres width was similar to pine fibres when cooking conditions were milder.

The quantity of microfibrils in the processed hemp samples increased when cooking conditions were harsher. This increase in the percentage of microfibrils improves the cross linking with other fibres, and consequently, the mechanical properties of

the final material. The microfibrils percentage of the fibres of pulp D (harsher cooking conditions) became similar to that in pine fibres.

In the case of fines number, the most influencing factor is the concentration used in the cooking process. Pulps A and B were obtained using the same ethanolamine concentration. It was observed that the number of fines was practically the same in both cases. An increase in the concentration of ethanolamine implied a reduction in the fines number. The number of fines in pine pulp was very high compared to hemp pulps. A higher number of fines improves retention in the formation of fibre-cement but reduces the resistance (Fuente et al., 2010).

Fig. 1 shows photographs of hemp fibres, obtained by SEM at different cooking conditions shown in Table 1. It was observed that the degree of deterioration in the fibres structure was greater at more aggressive cooking conditions. The deterioration of the fibres can be observed in the breaking of the structure due to the holes produced in the fibre and they are visible only in pulps C and D, pulps obtained by more aggressive cooking conditions.

3.1.2. Corn stalk

The morphological characterisation results from corn pulps are shown in Table 5. In this case, when cooking conditions were harsher, the length of corn fibres was larger but the percentage of microfibrils decreased. These results are contrary to those expected and may be due to the union of microfibrils, which produces agglomerated fibres which are consequently longer.

The width of corn fibres, as in the case of hemp fibres, was narrower when cooking conditions were harsher. The width of corn fibres for pulp 4 was similar to pine fibres when cooking conditions were harsher.

In the case of fines number, the main factor affecting was observed to be the temperature. When cooking temperature was lower, the fines number of corn pulps 1 and 2 was lower. When cooking temperature was increased in corn pulps 3 and 4, the fines number increased. Comparing pulps cooked at the same temperature, pulps 1 and 2 (165 ºC), and pulps 3 and 4 (185º C) the fines number decreased when the concentration of ethanolamine and cooking time were increased. Fines number in corn pulps was much higher than in hemp pulps. The value of fines number of corn pulp 1 was similar in value to that in pine pulp.

Fig. 2 shows photographs of corn fibres obtained by image SEM. Visually the degree of deterioration of corn fibres was more difficult to identify in the case of corn pulps than in the case of hemp pulps due to the cooking conditions.

3.1.3. Effect of refining on the morphological properties of corn pulp

As mentioned above, hemp pulps could not be mechanically refined due to their hardness. Then, only the effect of refining could be studied in the case of corn pulps. The effect of refining measured in the Canadian standard freeness (CSF), expressed as volume in mL, and the morphological characteristics of corn fibres are shown in Fig. 3. A high CSF value (volume in mL) indicates a low degree of refining.

As expected, the length and width of the fibres decreased when the degree of refining was increased. This may be due to the wear experienced by fibres in the refining process, leading to an increase in the fines number and percentage of microfibrils. This increase in percentage of microfibrils enhances the cross linking of fibres, forming a more stable network for the formation of fibre-cement.

Fig. 4 shows SEM images of corn fibres, obtained before and after refining the pulp. It can be observed that the fibres are broken when they are refined (low quantity of volume mL). This confirms that the length and width of the fibres are decreasing, thereby the number of fines increased.

When the concentration of ethanol was increased, the degree of refining achieved in the pulps was higher (using less revolutions, see Table 3), and therefore they had a lower CSF (volume in mL).

For the pulps obtained at high concentrations of ethanol and high cooking temperature (pulps 3 and 4), the refining of fibres with the PFI required less energy to achieve the same refining achieved in pulp 1 and 2.

Thus, it is better to use pulps obtained at aggressive cooking conditions to obtain a more refined pulp (less volume in mL) using a lower amount of energy in the refining process.

As shown in Fig. 3.c, pulp refining produces an external fibrillation of the cells, what allows flexibility and the formation of bridges with other fibres.

3.2. FBRM characterisation

3.2.1. Flocculation trials using hemp core

Fig. 5 shows the flocculation monitored for each hemp pulp with and without flocculant. Mean chord size (μm) is represented versus time. It is observed that the addition of flocculant led to an increase in the mean chord size.

In general, fibres are themselves flocculated mechanically, so it was difficult to induced flocculation and increase the mean chord size (Jarabo et al., 2010a). The APAM shows better results than the CPAM in all the pulps.

In Fig. 6, the comparison between the different hemp pulps and the pine pulp using APAM is shown. It can be observed that there may be two factors that influencing the fibres mean chord size:

Fibres length. When the lengths of the fibres are longer the mean chord size is higher and more flocculation is produced, as it was the case of pulp A, (which has the low cooking conditions). The mean chord size decreased each time the fibre length decreased as well.

Microfibrils. When the quantity of microfibrils increases the mean chord of the floc decreases and the flocculation is lower.

Then, it can be concluded that the decrease of fibre length and the increase in the percentage of microfibrils lead to a decrease in floc size, what means that flocculation takes place to a lower extent.

Comparing the floc size of hemp fibres with pine fibres it can be observed that the latter present larger sizes due to the length of pine fibres, 1130 µm, compared to that of hemp fibres, the larger of which was found to be pulp A, with a length of 509 µm. Despite this difference with pine fibre, there are similarities between the two pulp types. For example, the width of pulp A fibres and the quantity of microfibrils of pulp D are comparable to those of pine fibres. In view of these results, the two above mentioned hemp fibres were selected to study their potential as reinforcement in fibre-cement sheets.

3.2.2. Flocculation trials of corn stalk

Fig. 7 shows the graphs for each corn pulp with and without flocculant.

In these pulps, unlike hemp fibres, flocculation was less noticeable when the flocculant was added. The corn fibres are flocculated mechanically too, so it is

difficult to induce flocculation (Jarabo et al., 2010a), but there was a small increase in the floc size when APAM is added instead of CPAM.

In this case, the factor influencing the mean chord size was mostly the length and number microfibrils. When the fibres length increased and the microfibrils decreased, the mean chord size increased and, therefore, the size of the floc was greater.

With corn fibres, unlike with hemp, the harsher the cooking conditions employed, the greater the length of the fibres and lower the percentage of microfibrils were.

Fig. 8 shows that pulps 1 and 3 reach a similar floc size despite starting from different fibre lengths. Pulps 1 and 3 have smaller floc size than pulp 2. It is concluded, in addition to fibre length, another factor that affecting the floc size of the corn pulp is the "cooking time" of the raw material.

Comparing the mean chord size of corn fibres with pine fibres, it was found that pine fibre flocs had larger size due to the length of pine fibres, 1130 µm, compared with corn fibres. Nevertheless, in this case the difference with the corn pulp 4 (734 µm) is smaller than in the case of hemp fibres (509 µm). Pulp 4 also presented fibre widths and number of fines similar to those of pine pulps. Based on those characteristics pulp 4 was selected to be studied as further a reinforcement material in fibre-cement sheets.

3.2.3. Flocculation trials of corn stalk refining

Fig. 9 and 10 show the results of the flocculation tests carried out with refined corn pulps 1 and 3 respectively.

An important factor regarding the flocculation is the length of the fibres which is directly influenced, as shown in fig. 3, by the process of refining.

As expected, Fig. 9 shows that pulps obtained at less aggressive cooking conditions (pulp 1) need more energy to be refined process, whereas Fig. 10 shows that the pulp obtained at more aggressive cooking conditions (pulp 4) need less energy in the refining. This factor is very important from the environmental point of view.

4. CONCLUSIONS

The selection of suitable cooking conditions to obtain hemp and corn pulps is important for the morphological characteristics of the fibres, and, therefore, for their application in the fibre-cement manufacture.

In the studied conditions, corn fibres are longer and wider than those of hemp. Both types of fibres have similar microfibrils contents, but corn fibres show higher contents of fine elements.

When cooking conditions are aggressive, hemp fibres are shorter, narrower, have more microfibrils percentage and lower number of fines. Hemp fibres and pine fibres have the same width and percentage of microfibrils when cooking conditions are milder.

When cooking conditions are more aggressive, corn fibres are longer, narrower and have a lower microfibrils percentage. Corn fibres and pine fibres have the same width and similar length and fines number when cooking conditions are harsher. The number of fines does not follow a clear trend.

Refining produces defibrillation of corn fibres. Therefore a homogeneous mixture with the appropriate level of defibrillated fibres can be obtained, in suspensions of fibre-cement without affecting retention.

Natural fibres commonly need to be sized to a specific dimension for a particular application. Floc size depends on the length of fibres and, in the specific case of corn fibres, it depends on the cooking conditions.

From these results, it may be concluded that the two types of pulps studied can be used as reinforcement fibres for the manufacture of fibre-cement. At certain cooking conditions, the fibres from corn pulps and from hemp pulps can be made so as to have similarities with the pine fibres often used in this industry.

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Fig. 1. Hemp images obtained by SEM with an increase of 1500. Fibres from: a) pulp A, b) pulp B; c) pulp C and d) fibre from pulp D.

Fig. 2. Corn image by SEM with magnification 1500. Fibres from: a) Pulp 1; b) pulp 2; c) pulp 3 and d) pulp 4.

Fig. 3. Effect of refining on morphological characteristics of corn fibres. a) length, b) width, c) microfibrils and d) fines number.

Fig. 4. Corn image by SEM with magnification 1500. Pulp 1: 40% ethanol-60 min-165ºC. a) Before refining, b) increased 800 and c) increase 1500 after refining.

Fig. 5. Mean chord size with and without flocculant in fibres of hemp. a) Pulp A, b) Pulp B, c) Pulp C and f) Pulp D.

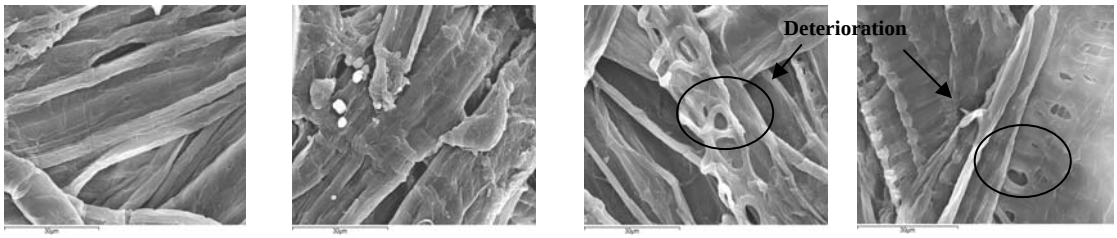
Fig. 6. Mean chord size with anionic polyacrylamide in fibres of hemp and pine.

Fig. 7. Mean chord size with and without flocculant in fibres of corn. a) Pulp 1, b) Pulp 2, c) Pulp 3 y d) Pulp 4.

Fig. 8. Mean chord size with anionic polyacrylamide in fibres of corn and pine.

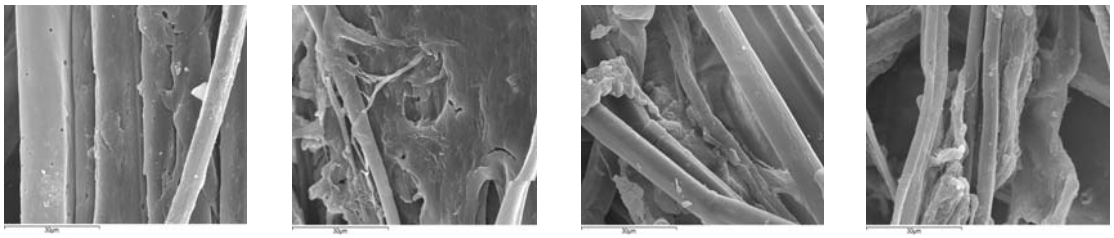
Fig. 9. Mean chord size with anionic polyacrylamide in refined corn pulp 1.

Fig. 10. Mean chord size with anionic polyacrylamide in refined corn pulp 3.



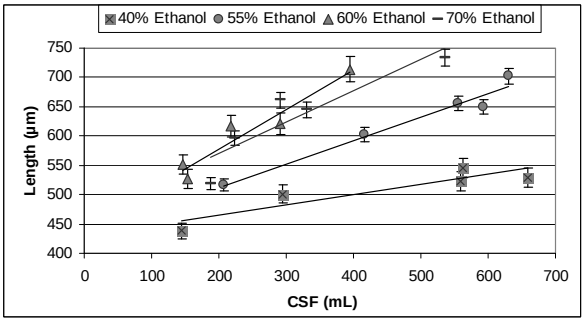
a) b) c) d)

Fig. 1. Hemp images obtained by SEM with an increase of 1500.
Fibres from: a) pulp A, b) pulp B; c) pulp C and d) fibre from pulp D.

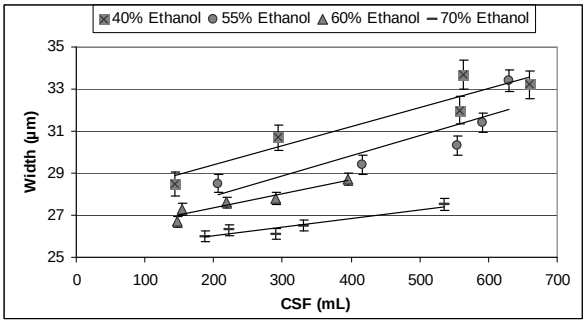


a) b) c) d)

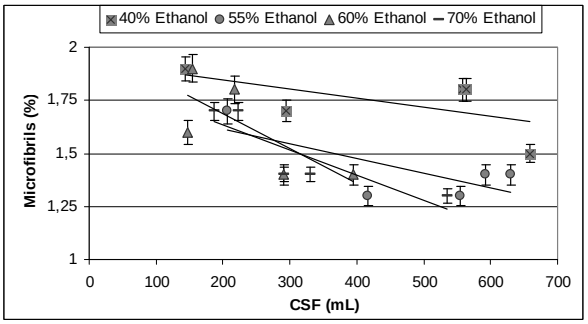
Fig. 2. Corn image by SEM with magnification 1500.
Fibres from: a) Pulp 1; b) pulp 2; c) pulp 3 and d) pulp 4.



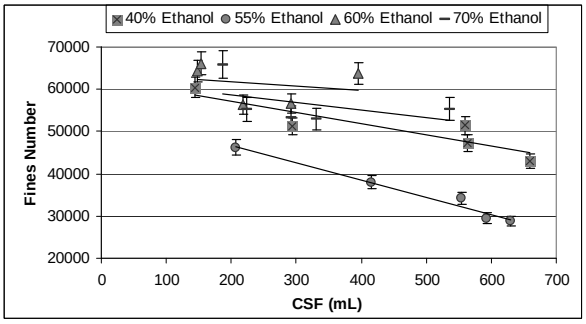
a)



b)

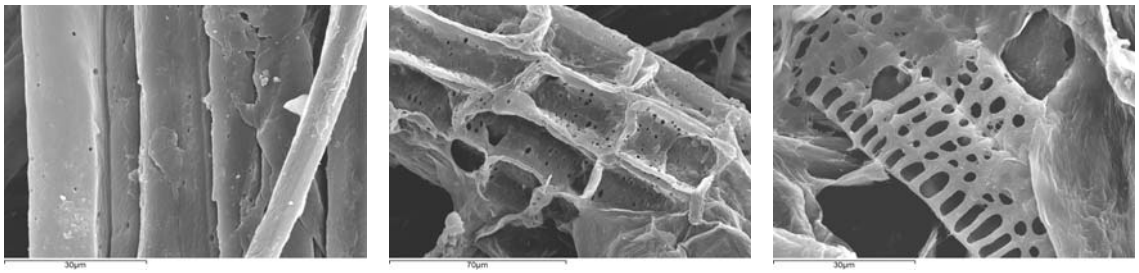


c)



d)

Fig. 3. Effect of refining on morphological characteristics of corn fibres. a) length, b) width, c) microfibrils and d) fines number.



a) **b)** **c)**

Fig. 4. Corn image by SEM with magnification 1500. Pulp 1: 40% ethanol-60 min- 165ºC. a) Before refining, b) increased 800 and c) increase 1500 after refining.

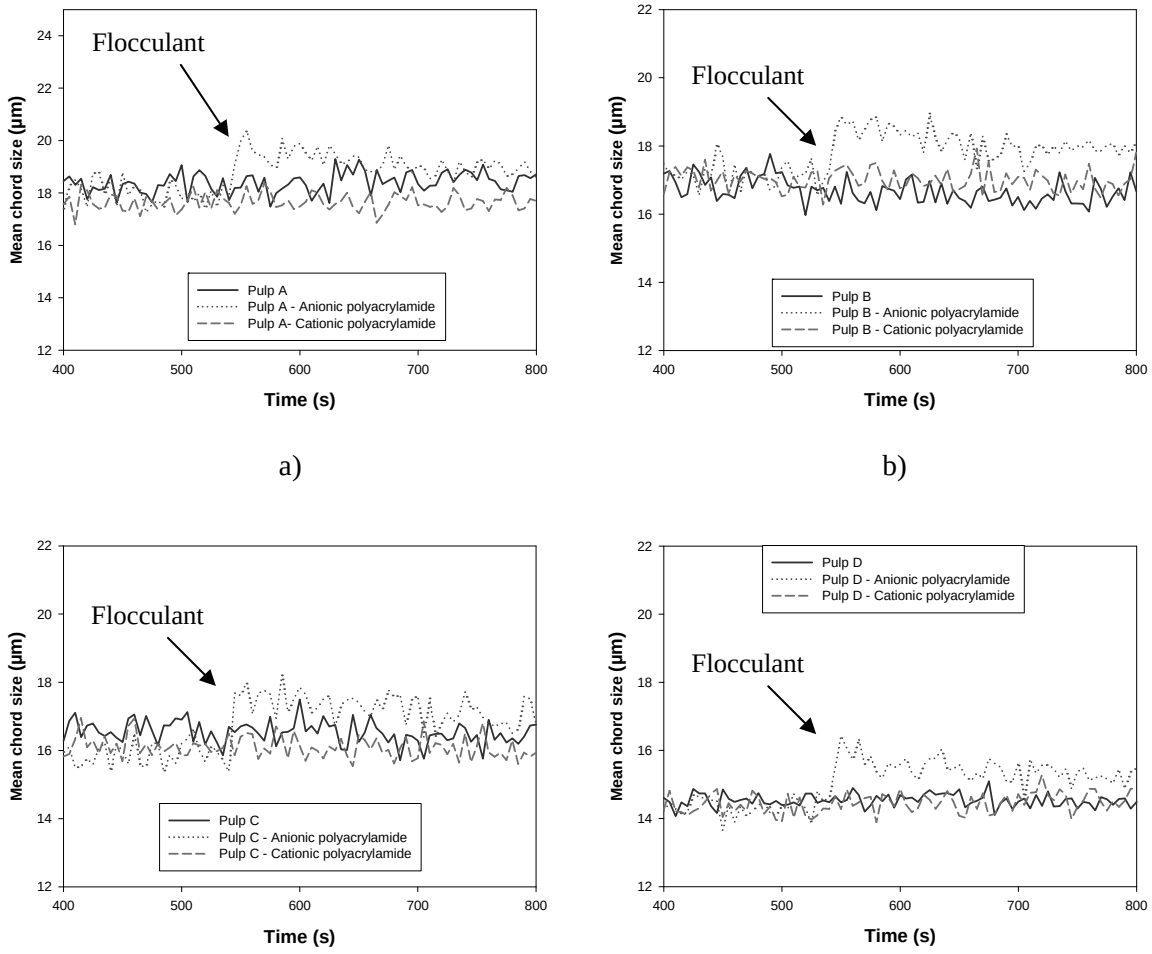


Fig. 5. Mean chord size with and without flocculant in fibres of hemp. a) Pulp A, b) Pulp B, c) Pulp C and f) Pulp D.

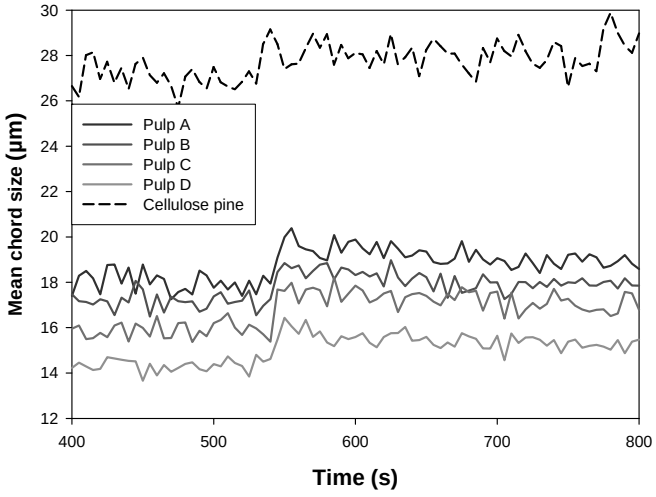
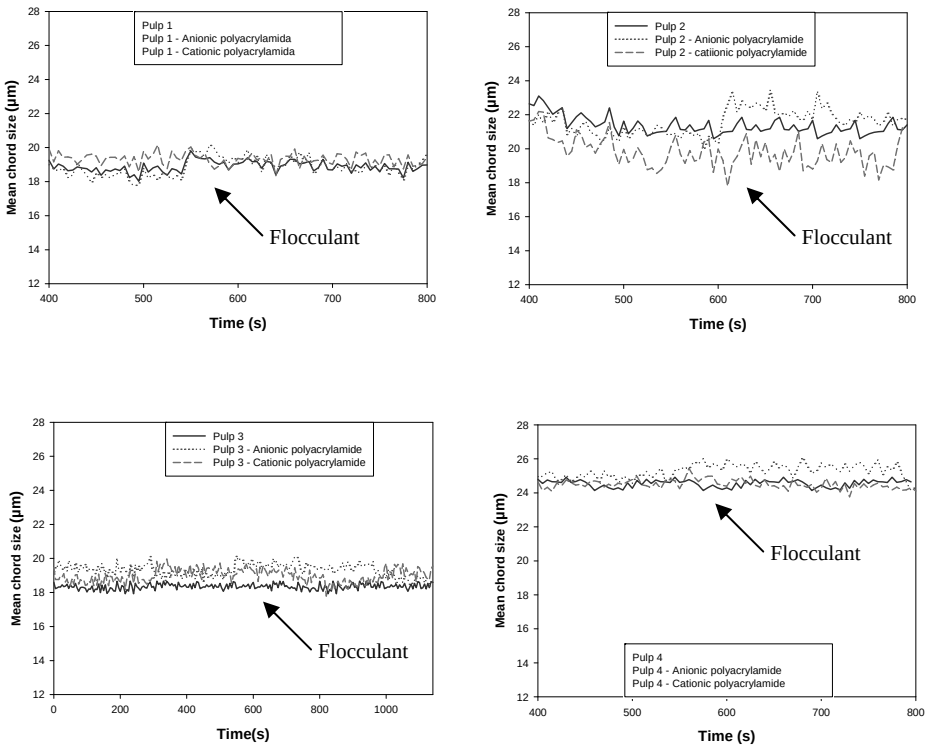


Fig. 6. Mean chord size with anionic polyacrylamide in fibres of hemp and pine.



a)

c) **d)**

Fig. 7. Mean chord size with and without flocculant in fibres of corn. a) Pulp 1, b) Pulp 2, c) Pulp 3 y d) Pulp 4.

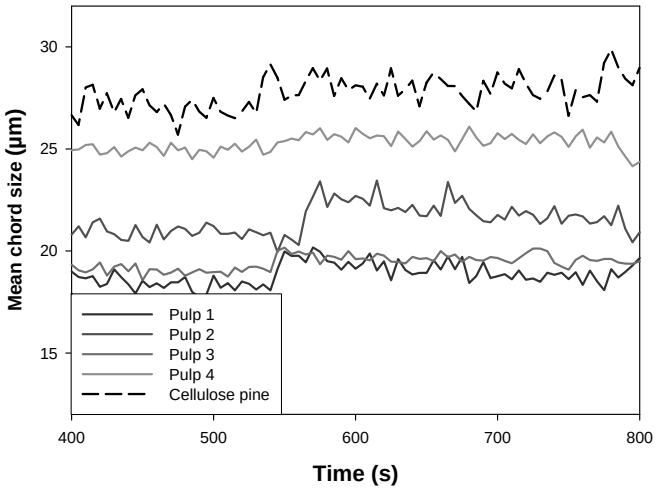


Fig. 8. Mean chord size with anionic polyacrylamide in fibres of corn and pine.

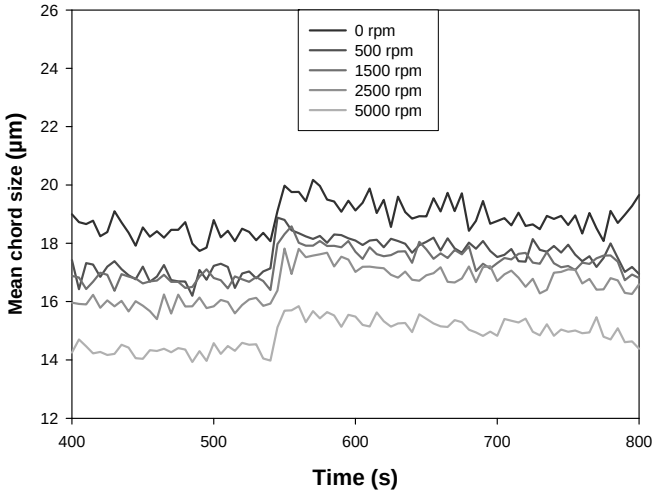


Fig. 9. Mean chord size with anionic polyacrylamide in refined corn pulp 1.

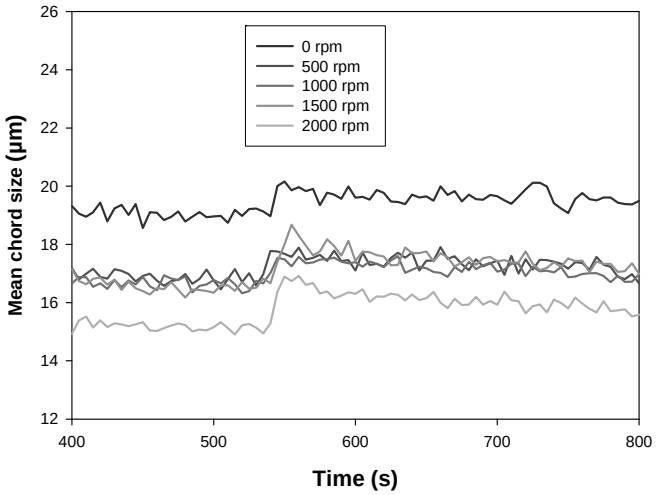


Fig. 10. Mean chord size with anionic polyacrylamide in refined corn pulp 3.

Table 1. Cooking conditions of hemp using ethanolamine.

	Ethanolamine (%)	Time (min)	Temperature (ºC)
Pulp A	40	30	155
Pulp B	40	60	170
Pulp C	50	60	170
Pulp D	60	90	185

Table 2. Cooking conditions of corn using ethanol.

	Ethanol (%)	Time (min)	Temperature (ºC)
Pulp 1	40	60	165
Pulp 2	55	90	165
Pulp 3	60	60	185
Pulp 4	70	120	185

Table 3. Revolutions number of each corn pulp.

Pulp	Revolutions number
40% Ethanol	0-500-1500-2000-5000
55% Ethanol	0-500-1500-2000-5000
60% Ethanol	0-500-1000-1500-2000
70% Ethanol	0-250-500-1000-1500

Table 4. Morphological characterization of hemp pulps at different cooking conditions and pine pulp.

Pulp	**A**	**B**	**C**	**D**	**Pine**
Length weighted in length (µm)	509	500	497	483	1130
Average Width (µm)	23,4	21,5	21,5	21,1	25,5
Microfibrils (%)	1,2	1,3	1,4	1,6	1,6
Fines number	9324	9910	7973	7683	49192

Table 5. Morphological characterization of corn pulp at different cooking conditions.

Pulp	**1**	**2**	**3**	**4**	**Pine**
Length weighted in length (μm)	529	702	714	734	1130
Average Width(μm)	33,2	33,4	28,7	27,5	25,5
Microfibrils (%)	1,5	1,4	1,3	1,3	1,6
Fines number	42938	28766	63726	55299	49192