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Factors Influencing the Drainage and Drying of Pulp

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Declaration

I, Richard Anthony Botha, hereby declare that this Master's dissertation is my own work. It is the intention that this Masters dissertation be submitted for the degree of Master of Engineering in Chemical Engineering at the University of Pretoria. This dissertation has not been submitted to any other university for any examination, degree or other form of qualification.

Signature of Candidate

Signed at Pretoria on this 30th day of October, 2015

Abstract

Thermal drying of a pulp sheet on a paper machine is one of the most energy-intensive operations in the paper-making process (Ghosh, 2011). The drying section of a paper machine removes less than 20% of the total water eliminated from the wet sheet, but is responsible for 78% of the energy consumption of the paper machine (Karlsson, 2009 & Biermann, 1996). Any enhancement of the dewatering of the paper sheet in the forming and pressing sections, thereby sending a sheet of lower moisture content to the drying section, would result in significant energy savings in the drying of the paper. Typically, the Canadian Standard Freeness (or freeness) and the Water Retention Value (WRV) of pulp are used as a measurement for the dewatering ability of pulp on the wire of the paper machine (Hubbe, 2007). However, neither of these measurements was designed to predict the water removed from the wet sheet (TAPPI T227, 1999, Scallan & Carles, 1972).

The factors that influence the dewatering of pulp on the forming fabric and in the pressing section of a paper machine have been the subject of many studies (Hubbe, 2007). The first objective of the current study was to compile a literature review to build an understanding of the dewatering of pulp on the paper machine. The effect of the properties of the fibres constituting the pulp on its dewatering ability, including the type of fibre, its length, pliability and extent of fibrillation were investigated in the literature. Furthermore, the type and location of water in and around the individual fibres was examined. Phenomena that occur during the drainage of water from the wet pulp sheet were also studied, including the inhibition of pulp drainage by mechanical refining as well as the potential for improvement of drainage by enzymatic treatment of the pulp.

The literature presented models that enabled visualisation and understanding of the phenomena that affect drainage. The choke-point hypothesis discusses how fines and other small particles in the pulp sheet may inhibit the drainage of water as these particles become lodged in channels that are available for the flow of water. The pliability of fibres, influenced by their coarseness and extent of internal fibrillation, may also inhibit the dewatering of the pulp sheet. Pliable fibres may conform to each other, resulting in a sealing effect that inhibits drainage. Mechanical and enzymatic refining may induce external and internal fibrillation of fibres, and the generation of fines in the pulp. Treating the pulp with an enzyme, specifically endoglucanase, may enhance the susceptibility of the fibres to development by mechanical refining.

In the current study, the dewatering of pulp was analysed at pilot scale in order to test which pulp measurements best predicted its forming and pressing performance. A hardwood pulp, composed of *Eucalyptus grandis*, was refined in a pilot refiner and yielded samples over a range of refining energies. The conventional dewatering measurements, freeness and WRV, were performed on the pulp samples and related to the dewatering performance of the handsheets produced therefrom. The formability (water removed during forming) and pressibility (water removed during pressing) of the handsheets were also determined, and compared to the freeness and WRV of the pulps. It was found that the formability of a handsheet was a much stronger predictor of its dewatering performance than either the freeness or WRV of the pulp. Formability was likely the best predictor of handsheet dewatering because it was a direct measurement of the dewatering performance of a sheet on the forming fabric, whereas freeness and WRV both measured the dewatering of a thicker pad of pulp.

The effect of the refining of pulp with an enzyme additive on its dewatering properties was also studied at pilot scale. An endoglucanase was incubated with a hardwood pulp, composed primarily of *Eucalyptus grandis*, and refined in a pilot refiner. The enzyme was added at four levels, including a control pulp with no enzyme. The dewatering properties of the handsheets formed from the pulp were tested and compared for each enzyme dosage. The morphology of fibres of each of the pulps was determined. It was found that increasing the enzyme dosage of the pulps resulted in the decrease of the dewatering ability of the handsheets. The number of fines in the pulp increased with increasing enzyme dosage. It was likely that fines were responsible for the restriction of pulp dewatering, as suggested by the choke-point hypothesis (Hubbe, 2007). Based on the extent of fibrillation and width of fibres, as well as the number of fines in the pulp, a model of the effect of the enzyme added on the dewatering of the pulp was proposed. Increasing the enzyme concentration in the pulp increased the susceptibility of the fibre to refining. An enzyme dosage of 100 g/t resulted in a more fibrillated fibre than found in the control pulp. However, the fibres of the pulp treated with the enzyme at a concentration of 200 g/t were less fibrillated, suggesting that the fibre surface was weakened to the extent that the fibrils were broke off during refining. The fibres of the pulp treated at 500 g/t were again more fibrillated, suggesting the occurrence of secondary fibrillation of the fibre at this high enzyme dosage. The fibres of the pulps treated at 500 and 200 g/t were also narrower, suggesting the loss of fibre material.

KEYWORDS: Pulp, drainage, dewatering, freeness, water retention, enzymatic refining

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Contents

Nomenclature

A	area	m^2
c	consistency	kg/m^3
D	diffusion coefficient	m^2/s
h	heat transfer coefficient	W/m^2K
k	thermal conductivity	W/m K
K	permeability	m^2
Q	heat transfer rate	W
m_{ev}	mass flow rate by evaporation	kg/s
m_{bw}	mass flow rate of bound water	kg/s
p_c	capillary pressure	Pa
S_0	hydrodynamic surface area	m^2/m^3
T	temperature	K
X	sheet thickness	m
$v_{superficial}$	superficial velocity	m/s
W_{dry}	dry solids mass	kg
z_{bw}	concentration of bound water	kg/m^3

Greek symbols

α	effective volume per unit mass	m^3/kg
ρ_w	density of water	kg/m^3
μ	viscosity	kg/s m

1 Literature review

1.1 Introduction

The drying of a pressed paper web to the final paper sheet is one of the most energy-intensive processes in the paper-making industry (Ghosh, 2011). Improving the drainage ability of the wet paper web could result in energy savings in the drying section of the paper machine. Mechanical refining and enzymatic refining of pulp, the structure and properties of the fibre and fibre web, the location of water within the fibre structure, as well as the properties of the water that needs to be removed, influence drainage ability of the wet paper web (Biermann, 1996, Ghosh, 2011, Karlsson, 2009). Typically, the Canadian Standard Freeness (CSF), or freeness, and Water Retention Value (WRV) of the pulp have been used to predict its dewatering performance on the paper machine (Hubbe, 2007). However, relating the pulp and fibre properties to drainage based on data from these tests is difficult as they were not designed to simulate the drainage of water from pulp on the paper-machine wire, or in the pressing section (TAPPI T227, 1999, Water Retention Value, 2000). Developing an understanding of the factors that affect pulp drainage at laboratory scale could prove useful in understanding and improving drainage on a commercial paper machine.

This literature review will discuss drying in the paper-making process and the need for improved drainage to save energy. The fibre and web properties that affect drainage, and how they are induced, as well as the properties and locations of water to be removed from the fibre will be examined in order to form a comprehensive understanding of pulp and paper dewatering.

1.2 Drying in the papermaking process

The dewatering of pulp to form paper commonly occurs in three sections of the paper machine: forming, pressing and drying (Figure 1-1). In the forming section, pulp at a low consistency (mass percentage of dry solids) of 0.2 to 1 % is applied to the forming fabric where water is removed by drainage due to the force of gravity and some pressure difference developed by suction equipment (Karlsson, 1996). Pulp at a low consistency is used to ensure uniform distribution of fibres to form a web of constant thickness across the forming fabric. The forming section removes between 100 and 170 kg of water per kilogram of pulp and a wet paper web at a consistency of 15 to 25 % enters the pressing section (Karlsson, 2009 & Biermann, 1996).

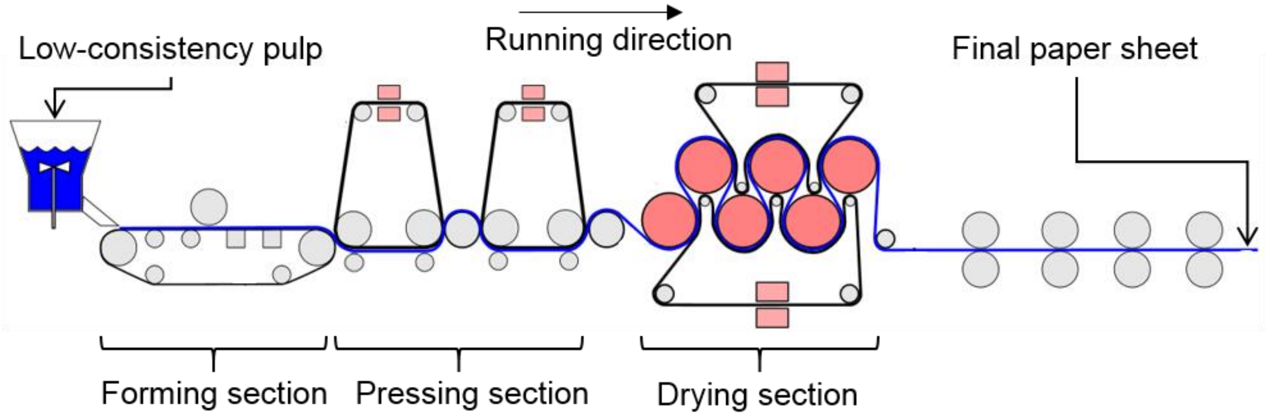


Figure 1-1: Schematic of a generic paper machine.

In the pressing section, the wet paper web is passed through a series of mechanical rolls and nips (Biermann, 1996). Here water is removed by mechanical force where pressure applied to the web expels the contained water. Approximately 2 to 4 kg of water per kilogram of dry pulp is removed in the pressing section and the web exits at a consistency of 33 to 55 % (Karlsson, 2009).

The drying section is the final stage of the dewatering process and is responsible for drying the paper to its final solids content of approximately 94 %. In this section, water is removed by heating and evaporation. The web is passed over a series of steam-heated rotating cylinders. Drying is the most energy-intensive, and therefore costly, section of the dewatering process. Despite the fact that the drying section only removes 1 to 1.5 kg of water per 1 kg of fibre, it is responsible for about 78 % of the total dewatering energy required (Figure 1-2) (Ghosh, 2011).

The energy required to remove one unit of water in each of the three sections (forming, pressing and drying) has a ratio of 1:5:220 (Karlsson, 2009). It is apparent that the more water that is removed before the web enters the drying section, the more energy will be saved in the drying stage. Therefore, the need for improving the drainage and pressing properties of pulp and the wet web respectively is paramount. De Beer, Worrell & Blok (1997) provided the breakdown of water removal and energy consumption in the different sections of the paper machine (Figure 1-2).

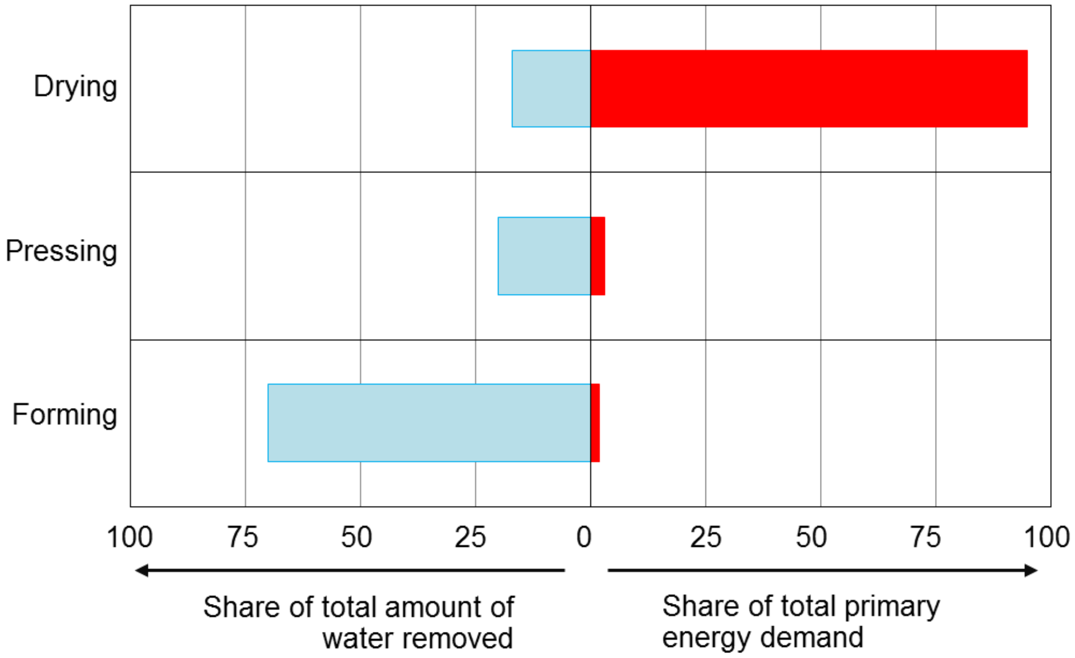


Figure 1-2: An approximation of the energy share of each section in dewatering against its respective water removal. Adapted from De Beer, *et al.* (1998).

1.2.1 Water in the web structure

Cellulose fibres found in paper are porous and can absorb water. Water can be found in the lumen of the fibre, on its surface and within the pores on the fibre wall (Häggkvist, Tie-Qiang & Öderberg, 1993). The wet paper web contains different types of water, the properties of which need to be considered for their removal. Broadly speaking, a wet web contains unbound water and bound water. Unbound water has the same thermodynamic properties as bulk water, and is situated in the voids between fibres and on the fibre surface, as well as inside the fibre lumen and in macropores on the fibre surface (Figure 1-3).

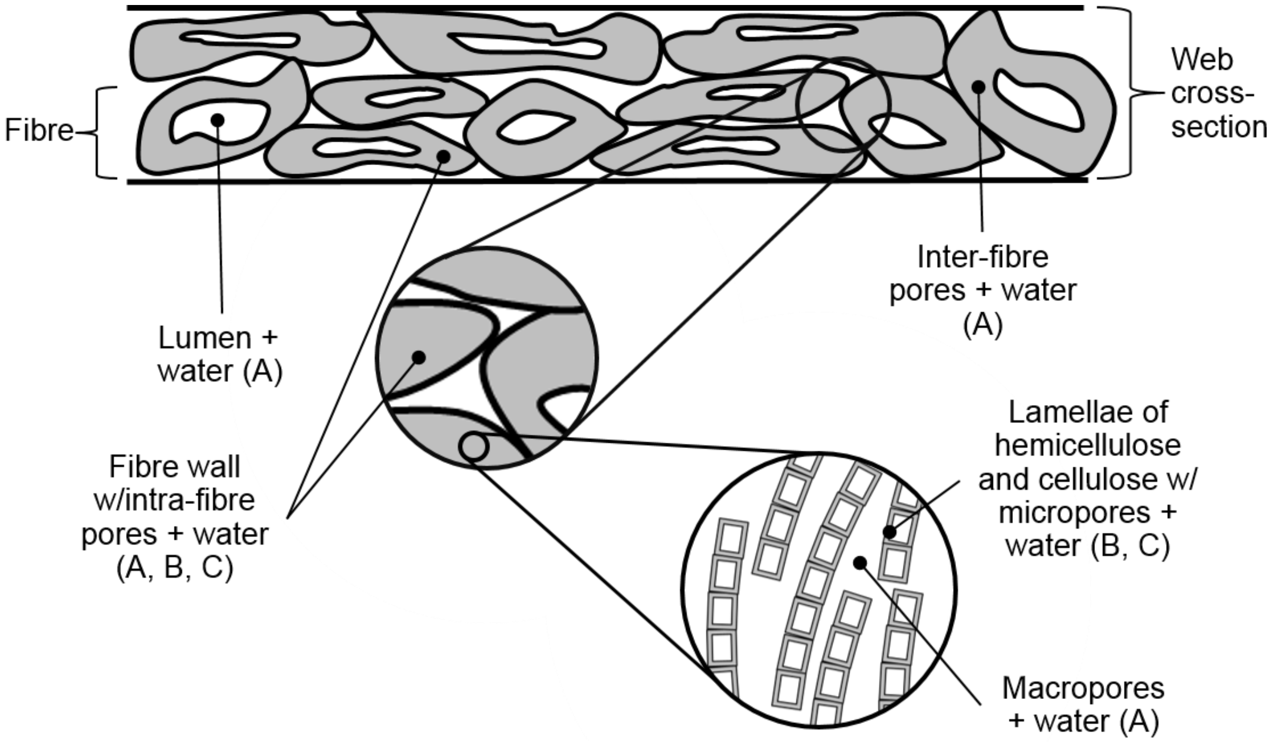


Figure 1-3: Schematic diagram of a cross section of a pulp-fibre web showing the locations of water. Unbound water (A), freezing bound water (B) and non-freezing bound water (C). Adapted from Karlsson (2007).

The remaining constituent of water is composed of freezing and non-freezing bound water located within the fibre walls: freezing bound water is situated in the micropores of the fibre wall, while non-freezing bound water is situated in the amorphous lamella region and in accessible hydrophilic groups (Weise, 1997). The bound water is present as mono or multi molecular layers and is considered to be in a state of interaction with the fibres (Weise *et al*., 1996). Therefore, bound water is difficult to remove and only removed after a significant fraction of the unbound water has been eliminated (Figure 1-4).

The sequence of water removal from the fibres during the drying of wet pulp was investigated by Weise *et al*. (1996) using differential scanning calorimetry (DSC) (Figure 1-4). It was determined that unbound water was removed first, followed by freezing bound water and lastly non-freezing bound water, with some overlap between the bound water components' removal. This can be related to the moisture content of the paper web in each section on the paper machine (forming, pressing and drying) to determine the type of water removed in each section (Figure 1-5).

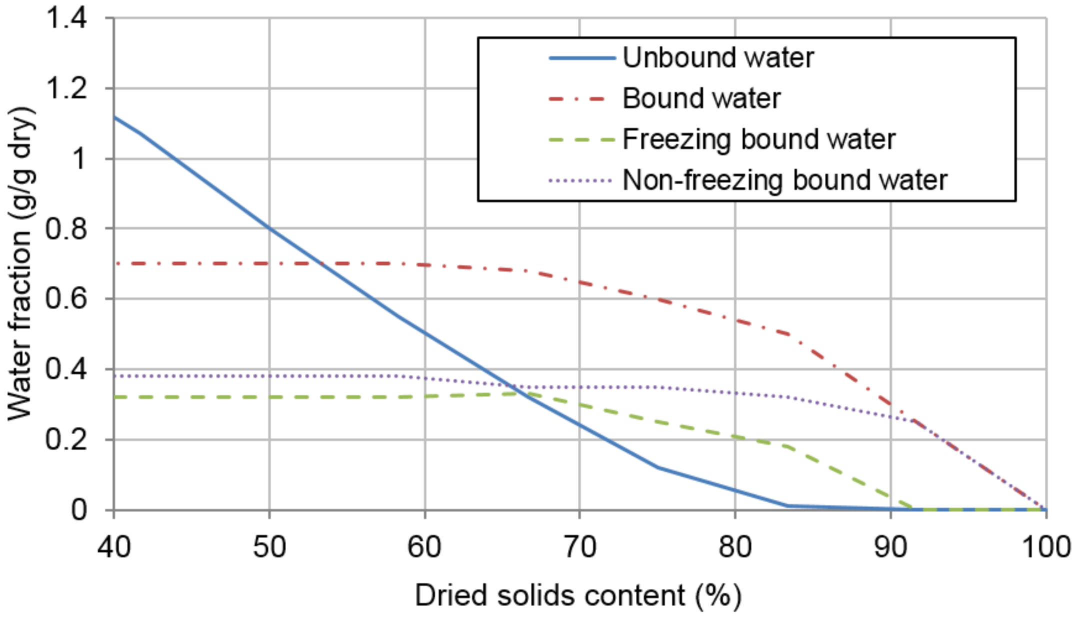


Figure 1-4: A schematic of the change in unbound and bound water during paper drying. Total bound water is shown as the sum of freezing and non-freezing bound water. Based on Weise *et al.* (1996).

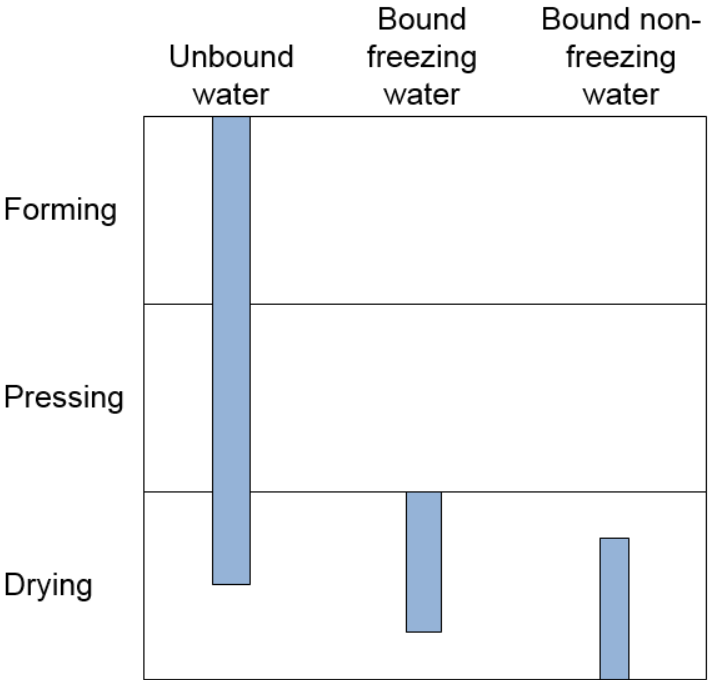


Figure 1-5: Types of water removed in the forming, pressing and drying sections of a paper machine. Adapted from Weise *et al.* (1996).

Park, Venditti, Jameel & Pawlak (2007) provided an effective illustration to aid in the visualisation of water removal and fibre shrinkage during the drying of the web on the paper machine (Figure 1-6). They emphasise the distinction between free water and trapped water as components of unbound water. It is stated that free water is the first to be removed, followed by trapped water which is difficult to evaporate but considered unbound water (Figure 1-6a). Trapped water can be found in the fibre lumen, between fibres and on the fibre walls (Figure 1-6c). The component of unbound water below the drying line in Figure 1-5 can therefore be considered to be trapped water.

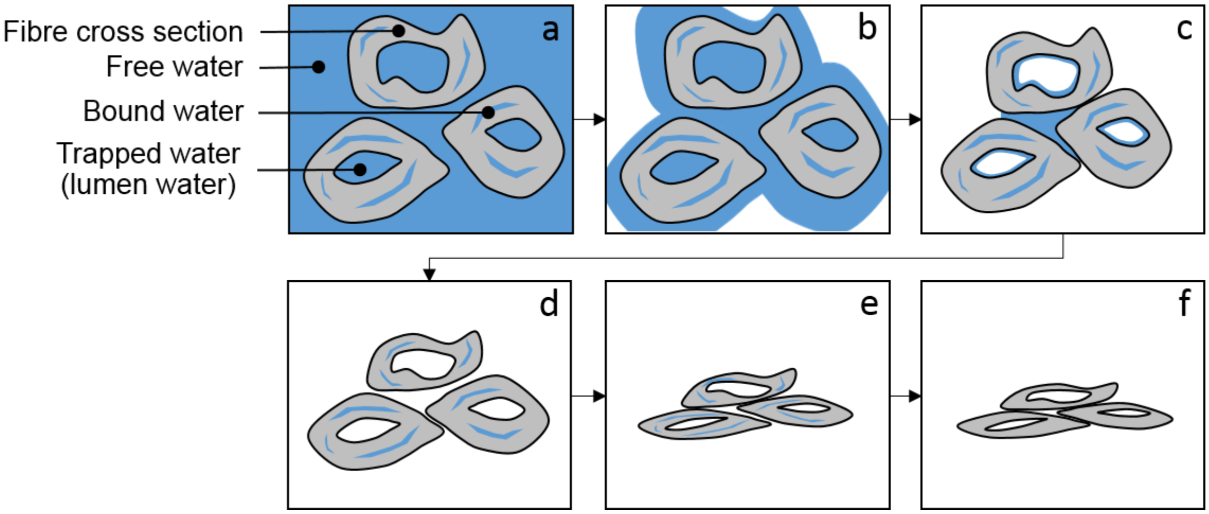


Figure 1-6: Cross section of fibres in a web as drying progresses from (a) to (f). Adapted from Park *et al.* (2007).

The apparent process of water removal from the wet web during drying can occur in two distinct phases (Park *et al*., 2007). First, free water is removed at a constant rate (Figure 1-6a-b) followed by the removal of “hard to remove” (HR) water at a falling rate (Figure 1-6b-f). HR water consists of water trapped in the lumen, which is unbound but difficult to evaporate, and bound water. The implication of this on dewatering on the paper machine is that the more trapped unbound water there is, the more difficult drying becomes. It is therefore important to minimise the occurrence of trapped water in the fibre.

1.2.2 Mass transfer of water in the wet web

During dewatering of the wet paper web in the forming and pressing sections, unbound water is removed from spaces between the fibres and macro pores on the fibre cell walls (Ghosh, 2011), driven by capillary pressure, and then from the lumen of the fibres (Karlsson, 2009). The unbound water constitutes a continuous phase in the web and its movement to the web surface can therefore be described by capillary flow during pressing of the web, as characterized by Darcy’s equation (Equation 1),

$$\frac{-m_w}{A} = \rho_w \frac{K}{\mu}\frac{dp_c}{dy} \tag{1}$$

Trapped unbound water remains as the wet paper web enters the drying section and is removed by evaporation, according to Equation 2:

$$\frac{m_{ev}}{A} = \rho_w \frac{K}{\mu}\frac{dp_c}{dy} \tag{2}$$

The capillary pressure gradient ($\frac{dp_c}{dy}$) in the web, generated by either pressure gradient over the web in the pressing section, or water loss by evaporation in the drying section, is the driving force for the mass flow rate of liquid water and water vapour respectively. Paper permeability (K), water viscosity (μ) water density (ρ_w) and the sheet surface area (A) are constants (Ghosh, 2011, Karlsson, 2009).

At the instant that all the unbound water is removed, the moisture content of the paper is equal to the fibre saturation point (FSP) (Topgaard & Soderman, 2002). The moisture at FSP is defined as the amount of water inside the fibre wall, according to Topgaard & Soderman (2002), and is considered bound water.

Once the moisture content of the paper web has reached FSP, continuous flow is no longer possible and surface diffusion occurs (Ghosh, 2011). The diffusion of bound water can be described by an equation similar to Equation 2 above, Equation 3 (Ghosh, 2011):

$$\frac{m_{bw}}{A} = D_{bw}\rho_d \frac{dz_{bw}}{dy} \tag{3}$$

The mass flow rate of bound water (m_{bw}) is directly proportional to the diffusivity coefficient of bound water (D_{bw}), dry material density (ρ_d) and the concentration gradient of bound water ($\frac{dz_{bw}}{dy}$). The diffusivity coefficient of bound water is indirectly proportional to the concentration of bound water and will also increase with increasing temperature, such as in the drying section (Ghosh, 2011).

1.3 Influence of fibre and pulp characteristics on water removal

A review paper by Hubbe & Heitmann (2007) summarized the current body of knowledge on the factors affecting the drainage ability of pulp on the wire. Generally, the increase in fine fibres content of the pulp, conformability of pulp fibres and increased basis weight of the paper all inhibit drainage. An increase in fibre stiffness and tendency of the fibres to flocculate, possibly with the addition of bridging polyelectrolytes and retention aids, promote drainage. Hubbe (2002) explained that there are three mechanisms that affect drainage resistance in the wet paper web: the choke-point hypothesis, mat density effects and the surface area effect.

Hubbe & Heitmann (2007) described phenomena that occur during drainage of water from pulp and their effects on further, high-consistency, drainage within the web. The choke-point hypothesis theorises that fine particles in the pulp move through the web during drainage only to become trapped in vital passageways between the fibres, blocking them, and inhibiting the flow of water through these channels (Figure 1-7a). In industry, polyelectrolyte additives are sometimes added to the pulp. These are used to attach the loose particles to the fibre surface by charging them, such that their mobility ceases high up in the web and they do not move down and block vital flow passageways (Figure 1-7b).

The flocculation of fines can improve drainage at low consistencies as the flocs tend to become trapped high up in the web and leave passageways below open for water to exit (Figure 1-7b). However, at high consistencies, these passageways also allow air to pass through the web. As a result, an appreciable pressure difference cannot be developed across the web, either by suction or gravity, and the driving force for water removal is mitigated. Corollary, high fines content aids in sheet consolidation at high consistency, improving the dewatering ability of the web as a more appreciable pressure difference is developed (Britt & Unbehend, 1980; Hubbe & Heitmann, 2007). Therefore, a contradictory relationship is observed where the effectiveness of drainage of water from the web is indirectly proportional to the effectiveness of the vacuum drawn across the web (Unbehend & Britt, 1982).

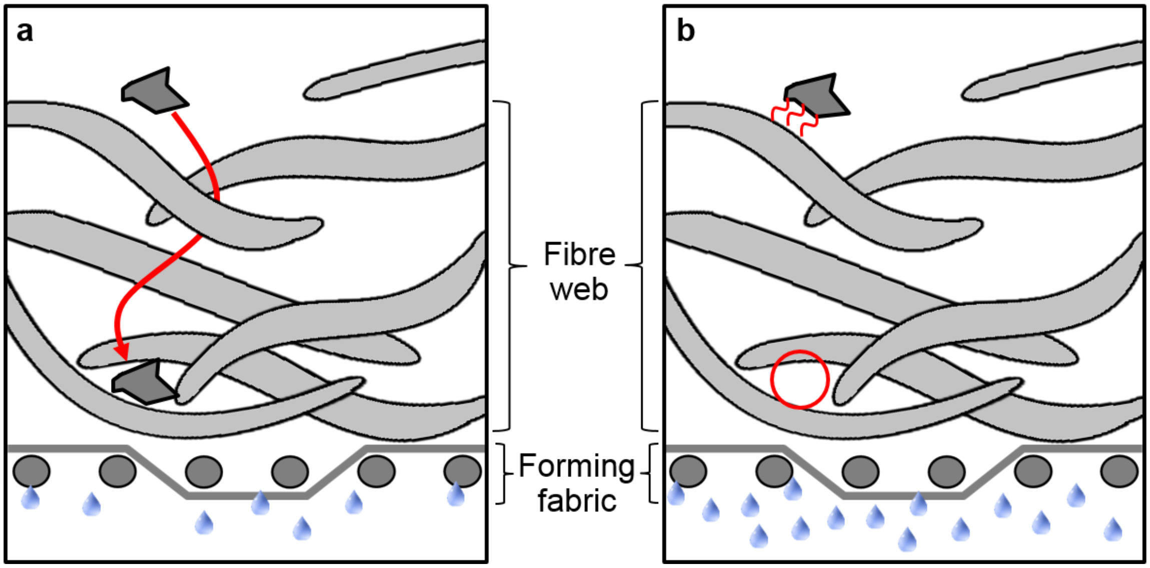


Figure 1-7: Schematic illustration of the choke-point hypothesis. A cross-section of the paper web on the forming fabric is shown, with the extent of water drainage represented by the number of water droplets below the forming fabric. Adapted from Hubbe & Heitmann (2007).

As previously mentioned, the conformation of fibres in the sheet, influenced by the extent of refining, affects the drainage properties of the web. More flexible fibres are able to bend and conform, lying closely together and thereby sealing passageways where water could exit the web (Figure 1-8b). Stiffer fibres pack in a manner that leaves voids in the web for water to pass through during drainage (Figure 1-8a) (Hubbe & Heitmann, 2007). The cross-sectional area, wall thickness and degree of internal fibrillation of the fibre affect its ability to conform (Karlsson, 2009).

Hubbe (2002) stated that an increasing fibre specific surface area, occurring with smaller particles or fines, inhibits drainage to a larger extent. Patel & Travedi (2004) noted greater resistance to drainage with a decrease in the size range of fine particles (Figure 1-9). The

Darcy equation has been used to model the relationship between fibre specific surface area and drainage rate.

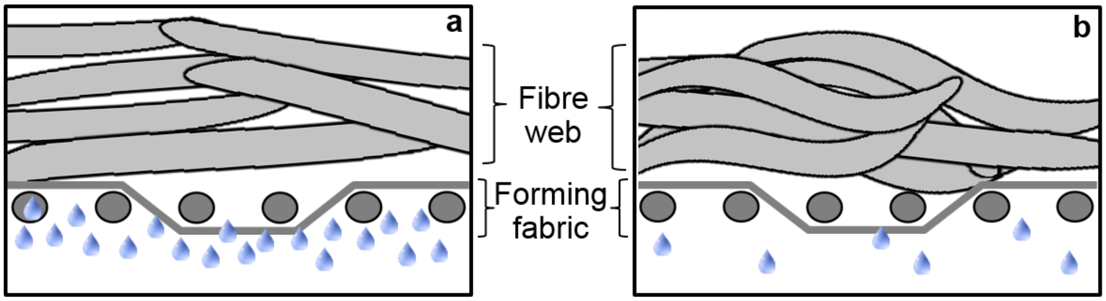


Figure 1-8: A schematic representation of fibre conformability in the paper web. Cross sections of a web comprised of non-conformable (stiff) fibres (a) and a web comprised of conformable fibres (b) are shown. Adapted from Hubbe & Heitmann (2007).

The Darcy Equation (4) is used to model the superficial velocity ($v_{superficial}$) of water through a packed bed of solids, a fibre web in this case, and the Kozeny-Carman Equation 5 can be used to determine the permeability coefficient (K) of the web (Hubbe, 2002). The Darcy equation was first applied to a fibre mat (essentially a wet paper web) by Lindsay (1994) and it predicts that drainage resistance is a function of the specific surface area of the fines; an increase in specific surface area yields an increase in resistance to drainage.

$$v_{superficial} = \frac{K\Delta P}{\mu X} \tag{4}$$

$$K = \left(\frac{1}{5.55\, {S_0}^2}\right) \times \frac{(1 - \propto c)^3}{(\propto c)^2} \tag{5}$$

Where ΔP is the pressure decrease across the mat of thickness X, while μ is the viscosity of the water draining through it. The permeability constant (K) is a function of the hydrodynamic surface area per unit volume (S_0); this increases with decreasing fibre or fine size, decreasing K and in turn decreasing $v_{superficial}$, the speed of water drainage. The remaining parameters are the effective volume per unit mass (α), and the consistency of the matt (*c*) expressed as mass of solids per unit volume (Hubbe, 2002). According to this theory, the presence of smaller fibres and fines result in higher resistance to drainage in the paper web.

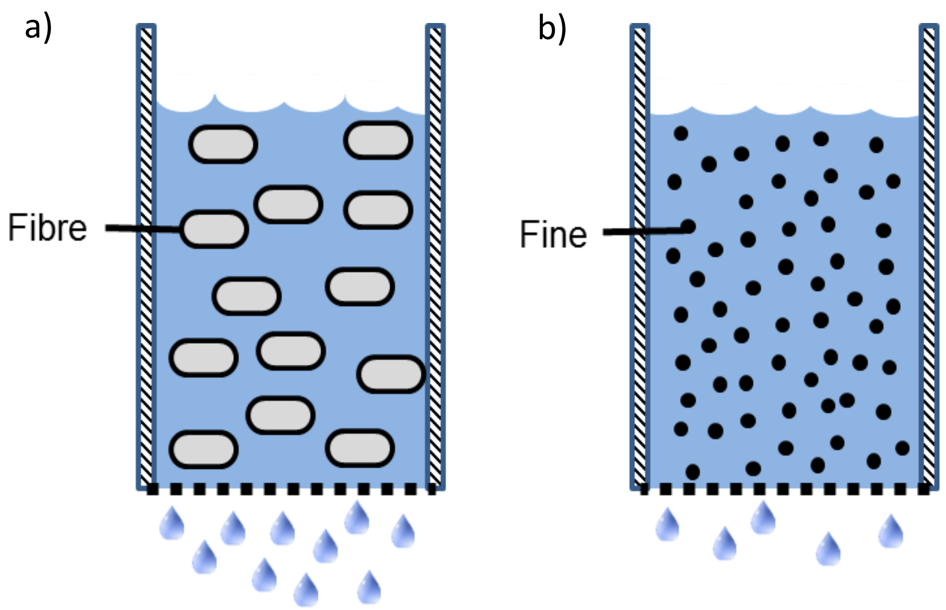


Figure 1-9: A model illustrating the difference in drainage performance between larger fibres (a) and fines (b). Adapted from Hubbe (2002).

The drainage properties of the wet web are also influenced by the makeup of the fibres, including their lignin content. Lignin is a high-molecular mass, polymeric component of wood. Lignin is responsible for binding fibres together; during wood pulping it is desireable to remove lignin and thereby improve fibre separation (Biermann, 1996). Lignin gives strength and rigidity to the fibre wall, but limits its water permeability (Moore & Jung, 2001) and therefore decreases the water drainability of the fibre. In practice, an increased Kappa number (a measure of lignin content) generally results in more refining of pulp to achieve the desired properties. This over-refining could result in increased fibre swelling by internal fibrillation (see 1.4.1: Refining), and therefore increase water absorption by the fibre, making drying more difficult.

1.4 Processes affecting water removal

1.4.1 Refining

In the paper-making process, mechanical refining of pulp is common practice. During refining, pulp is passed between metal discs, one of which is rotating, in an effort to alter the fibre properties by abrasion and cutting (Biermann, 1996). The aim of refining is to optimise the final paper properties. It is especially important to increase bond strength between fibres and enhance sheet formation, achieved by increasing the surface area of the fibres and improving their pliability. Refining therefore results in a denser sheet (Biermann, 1996; Fardim & Duran, 2003). Morphologically, refining results in the shortening of fibres by cutting and external fibrillation of the fibre surface. Evidence of fibrillation can be seen under microscope as fine structures protruding from the fibre surface (Figure 1-10). Internal fibrillation may also occur where the inner walls of the fibre become delaminated and the fibre may collapse (Figure 1-11), improving its flexibility. This can result in better inter-fibre bonding and conformability (Biermann, 1996; Fardim & Duran, 2003).

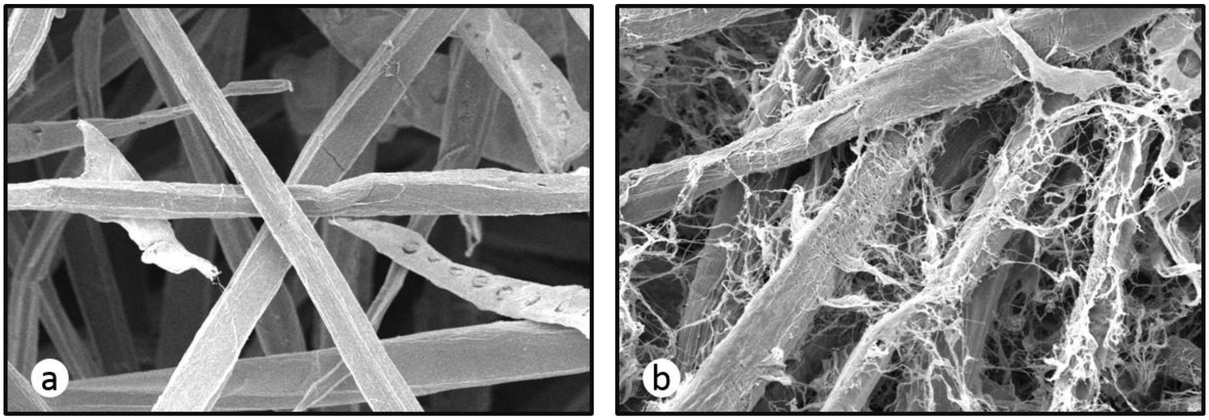


Figure 1-10: Scanning Electron micrographs of unrefined fibres (a) and refined fibres (b). Fibrils are visible on the surface of the refined fibres (b) (Wesley-Smith, 2012).

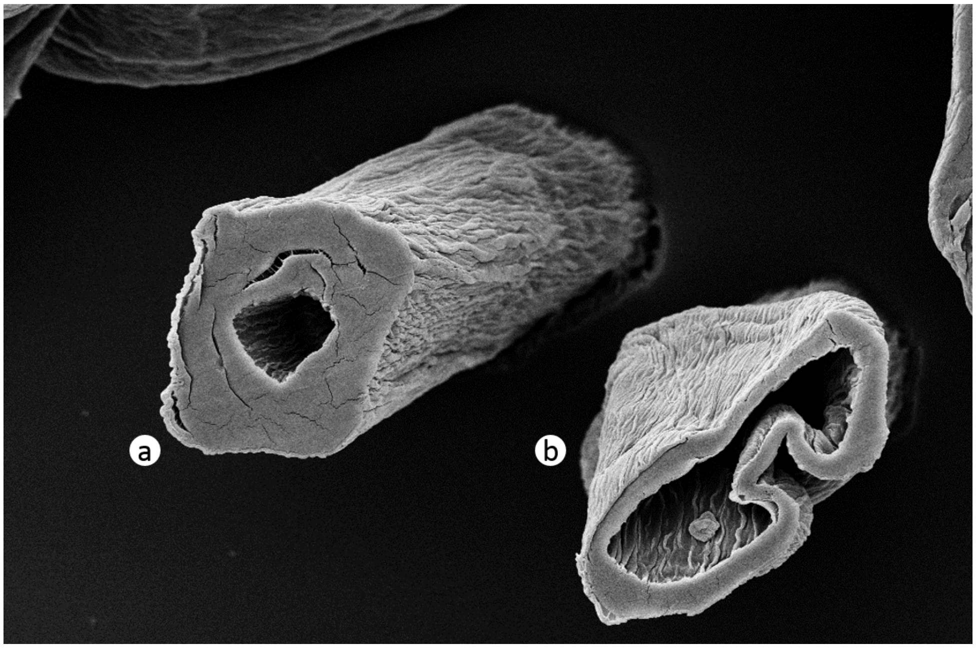


Figure 1-11: Scanning Electron micrograph of the cross section of a normal fibre (a) and a collapsed fibre (b) (Wesley-Smith, 2007).

Generally, inter-fibre bonding is improved during refining as fibre surface area and conformability is increased, resulting in the enhancement of tensile and z-direction (perpendicular to the sheet surface) strength of the final paper. However, individual fibres are weakened and shortened by cutting, diminishing the tear strength of the paper product as the average fibre length decreases (Biermann, 1996).

Refining influences the drainage performance of the pulp on the paper machine as well as the final paper properties. An increase in refining level yields a decrease in freeness, a measure of pulp drainage at low consistencies, as a denser web is formed (Biermann, 1996). In addition, fines created in the refining process may block drainage channels as described previously under the choke-point hypothesis of Hubbe & Heitmann (2007). Refining can also cause internal fibrillation in fibres which can induce swelling of fibres as their water absorption capacity is increased (Hui, L, Liu, Z and Ni, Y, 2009), making drying more difficult.

1.4.2 Enzymatic refining

In the paper industry, enzymes can be used for the modification of fibres in order to save energy and enhance fibre properties. Enzymes can be used to improve the susceptibility of the pulp to refining, as well as the drainability of the web (Blomstedt, Asikainen, Lahdeniemi, Ylonen, Paltakari & Hakala (2010). Enzymatic refining saves energy in refining as less mechanical action is required to modify the fibre to the same extent as untreated pulp. Bajpai, Mishra, Mishra, Kumar, S and Bajpai (2006) reported energy savings of approximately 19 % in refining energy on industrial scale with the use of enzymatic pretreatment of the pulp. Endoglucanase (a cellulase) and hemicellulase in particular are used to treat pulps in order to improve their drainage properties, lowering the energy demand in the drying section, and thereby increasing the running speed of the machine and saving costs (Blomstedt *et al.*, 2010). Bajpai *et al.* (2006) achieved savings of approximately 20 % in the drying section with enzyme-treated pulp.

The enhancement of pulp drainage by enzymatic action was first discovered by Fuente & Robert (1990). According to Mooney, Mansfield, Beatson & Saddler (1999), drainability of pulp is improved with enzyme treatment as fines and other small particles (from here-on referred to as crill) are removed. The Pulp & Paper Dictionary (1986) defines crill as: “very small pieces of fibre that occur on the surface of the pulp which are rubbed off during the refining process”. Crill is generated during mechanical refining as a result of enzymatic cleaving of amorphous cellulosic material on the fibre surface (Stork, Pereira, Wood, Dusterhof, Toft & Puls, 1995). Crill absorbs far more enzyme per gram than coarser fibres due to its greater specific surface area and is, therefore, more easily hydrolyzed (Mooney *et al.*, 1999), aiding in the dewatering of the pulp according to the choke-point hypothesis and surface area effect as previously discussed. It is possible that an insufficient enzyme dosage could result in the formation of crill which will not be consumed and, therefore, inhibit drainability of the pulp.

There is evidently a need for controlled enzyme dosage. Sprey (1990) performed a transmission electron-microscope study of the degradation of fibrous cellulose with enzyme treatment. It was determined that the degree of degradation of the amorphous regions was dependent on the enzyme dosage, with excessive treatment yielding highly eroded and fibrillated fibres (Figure 1-12) resulting in the loss of paper tear strength properties and inhibiting drainage; similar to observations made by Bajpai (1999). Bajpai (1999) stressed the need for an optimum enzyme dosage and stated that a controlled amount of xylanases and cellulases peel the fibre surface, removing hydrophilic microfibrils, reducing pulp-water interactions and thereby increasing drainage. Bajpai (1999) further stated that xylanase removes hemicellulose, which is responsible for interfibre bonding, which may result in poor paper properties.

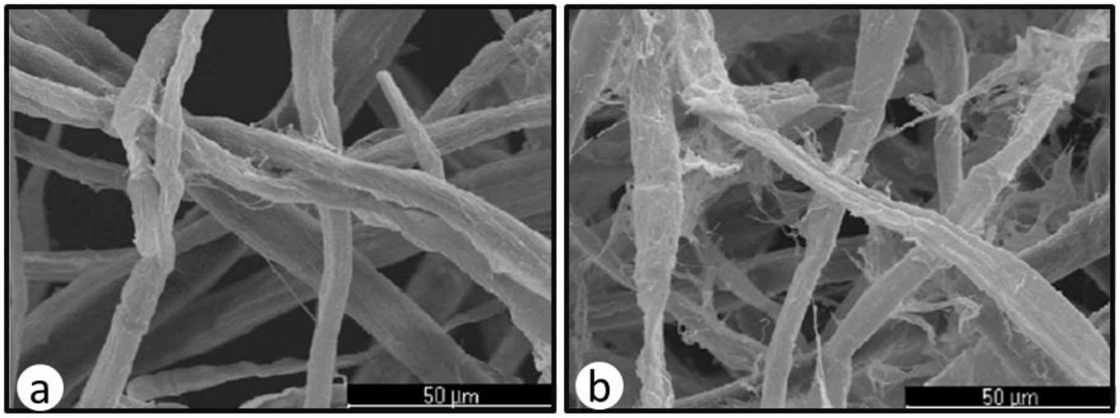


Figure 1-12: An untreated sample (a) compared with an enzyme-treated sample (b) (Gil *et al.*, 2009)

Gil, Gil, Amaral, Costa & Duarte (2009) studied the effect of cellulase and beta-glucanase pretreatment in pulp refining on energy savings in the paper-making process. It was determined that enzyme treatment improved drainage of pulp and did not negatively impact paper quality and it improved internal paper bonding. Gil *et al.* (2009) further stated that the pulp drainage at low consistency improved more than (WRV), an indication of water retention of the fibres. It was argued that the enzymes fibrillated the fibre surface, enhancing drainage, while not improving the WRV significantly, indicating minimal internal fibrillation due to a lack of enzyme penetration into the fibre.

Oksanen, Pere, Paavilainen, Buchert & Viikari (1999) made a similar observation: an increased drainage for softwood kraft pulps after endoglucanase treatment. The WRV did not change significantly even at high enzyme dosages. Oksanen *et al.* (1999) stated that endoglucanases improve drainage or dewatering by hydrolyzing the major constituent of fines (fibres less than 200 µm in length) formed during refining: amorphous hydrophillic cellulose. It was also found that endoglucanase I and II and hemicellulose treatment had a detrimental effect on the strength properties of the pulp.

Kamaya (1996) investigated the effect of cellulase on the drainability of never-dried bleached hardwood kraft pulp. The cellulase was also fractionated to determine which enzyme constituent was primarily responsible for improved drainage. Endoglucanase II was found to enhance the susceptibility to refining and drainability of the pulp the most effectively. However, as noted by Oksanen *et al.*, it was also found that the hydrolysis of the cellulose fibres by enzymatic action weakened the fibres.

Blomstedt *et al.*, (2010) investigated the use of xylanase on hardwood kraft pulps and concluded that the enzymatic action results in a notable increase in solids content of the enzyme-treated pulp, both after secondary pressing (pressing at appreciable pressure) and during drying. However, Blomstedt *et al.* (2010) noted a lack of difference in the formed solids content, suggesting this could have been due to the fact that faster initial drainage in the enzyme-treated pulp resulted in the formation of a denser web with a higher filtration resistance and lower porosity, offsetting the initial effect of faster drainage. However, according to Unbehend & Britt (1982), a denser web is formed as a result of increased fibre conformability and the effect of the speed of drainage on web density is not discussed. Therefore, it is possible that in the study by Blomstedt *et al.* (2010), the increase in fibre mat density was caused by an increase in fibre conformability (as previously discussed) due to enzymatic action.

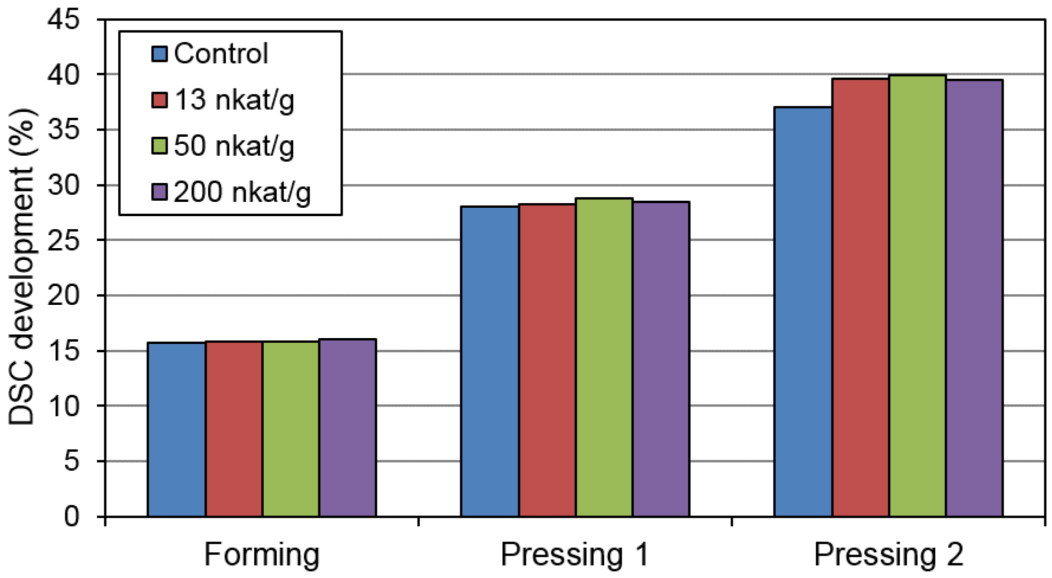


Figure 1-13: The dry solids content (DSC) of hardwood kraft pulp treated with different dosages of enzyme. Adapted from Blomstedt *et al*. (2010).

Blomstedt *et al.* (2010) speculated that enzyme treatment enhanced the collapsibility of fibres (evidence of internal fibrillation), contributing to the denser web structure as fibre conformability improved. The secondary pressing caused the collapse of pores in the cell wall. The increased dewatering of the enzyme-treated pulp at this stage was indicative of a loss in cell wall rigidity, caused by the enzymatic action, as water was able to exit the fibre structure. The fibre was thus more responsive to compression. Blomstedt *et al*. (2010) also measured an increase in the drying rate of enzyme-treated fibres (Figure 1-14). They speculated that structural changes to the fibre surface and pore structures caused by the enzyme treatment allowed for easier water removal by drying. The solids content was higher entering the drying section in the enzyme-treated pulps, but still increased at a faster rate than the untreated pulps.

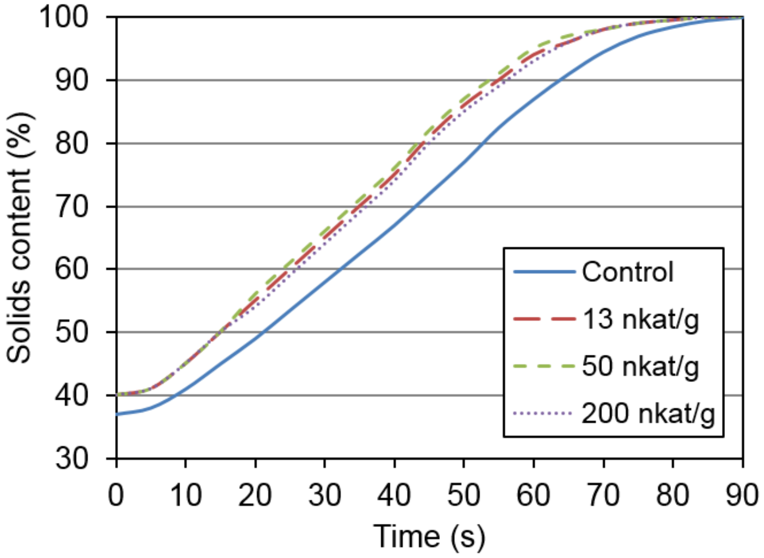


Figure 1-14: The increase in solids conent of untreated and enzyme-treated pulps during infrared drying. Adapted from Blomstedt *et al.* (2010).

Pala, Mota & Gama (2001) determined that the drainage properties of recycled container pulp and primary hardwood pulp were improved in dilute suspensions by enzyme treatment. However, it was also determined that the permeability of a fibre cake at high consistency decreased with enzyme treatment: a similar observation to that of Blomstedt *et al*. (2010).

1.5 Changes in web structure during drying

During drying, the amount of water in the sheet decreases significantly and spaces in the fibre walls where water was removed undergo irreversible shrinkage and closure, or are filled with gas (Ghosh, 2011). According to Karlsson (2009), the web shrinks by 30 to 40 % in the z-direction and up to 10 % in the plane direction depending on the amount of sheet strain in the machine and cross-directions. The density of the paper increases with drying and consequent shrinkage as the relative amount of solids increases (Figure 1-15).

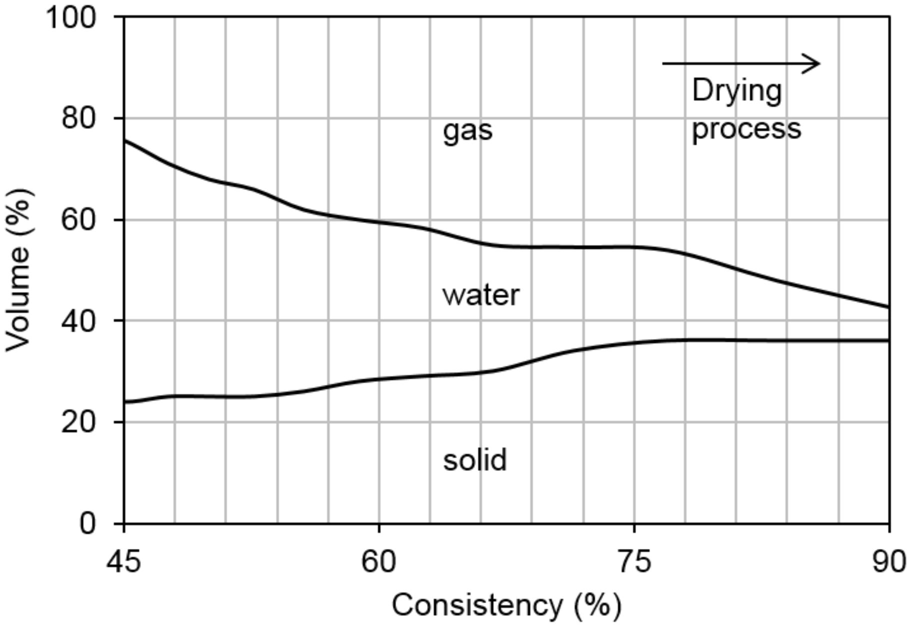


Figure 1-15: The distances between the lines represent the volume fractions of gas, water, and solids in a web with increasing consistency. Adapted from Karlsson (2009).

1.5.1 Hornification

Hornification is the phenomenon that occurs when drying fibres become tortuous and harden. According to Diniz, Gil & Castro (2004), hornification refers to the stiffening of the polymer structure that occurs upon drying in lignocellulosic materials. Weise *et al.* (1996) defines hornification as the decrease in WRV as a percentage of the original WRV (Figure 1-16). The most drastic increase in hornification occurs between 40 % and 75 % solids content.

Weise *et al.* (1996) argued that that wet pressing affects WRV; this is indicative of hornification, or ‘wet hornification’, and it is not a result of heat drying. It was found that upon pressing to a ‘critical solids content’ of 25 to 45 % (for bleached kraft pulp), there is an irreversible decrease in WRV- the manifestation of hornification.

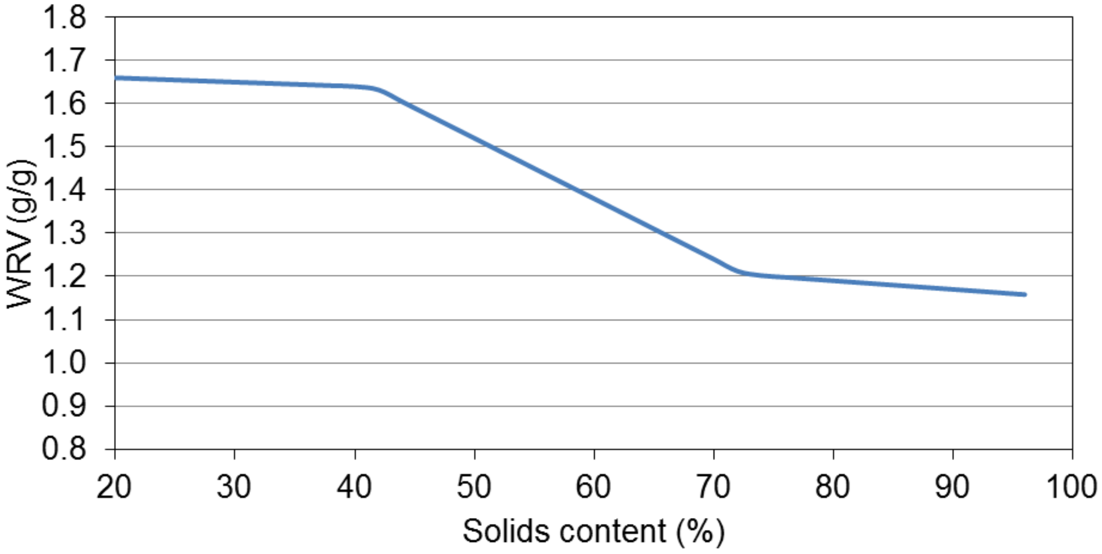


Figure 1-16: A graph of the decrease in WRV of fibres as drying progresses. Adapted from Weise *et al*. (1996).

Early text suggests that hornification is as a result of fibril packing upon drying and interfibril hydrogen bonding, Diniz *et al.* (2004) suggested that the data shows that hornification is a symptom of lactone bridge formation in lignocellulosic materials. Diniz *et al.* (2004) further stated that hydrogen bonds in amorphous lignocellulosic materials are completely broken by water, suggesting that hydrogen bonding is not a cause of the lowering of WRV. Diniz *et al.* (2004) suggested that carboxylic acid groups interact with hydroxyl groups in the lignocellulosic structure and form covalent lactone bridges, resulting in crosslinking; this is responsible for the symptoms of hornification, and that hornification can be reversed only under specific chemical conditions.

1.6 Laboratory Methods

In the paper industry, a means of measuring the drainage ability of pulp would be useful in predicting the performance of the pulp on the paper machine. Canadian Standard Freeness (CSF) was developed to measure the drainage ability of pulp at low consistency (0.3 %). According to TAPPI (T227, 1999), CSF is used to follow the changes in drainage rate of various pulps during beating/refining, but it also states that the CSF measurement does not necessarily correlate with the pulp sheet on paper machine, as it is not a direct measurement of sheet drainage. The CSF apparatus consists of a chamber and a funnel (Figure 2-5). A pulp suspension is poured into the chamber and then allowed to drain through the screen plate into a funnel. The CSF measurement, in mL, is the volume of water exiting the drainage chamber at high velocity from the side orifice while the remainder of the water exits via the bottom orifice. A higher CSF, or freeness, measurement indicates a pulp with faster drainage.

The Water Retention Value (WRV) is representative of the fibre saturation point of pulp fibres (Scallan & Carles, 1972). It is an empirical measure of the mass of water per mass of dry pulp after centrifugation under specific conditions (Water Retention Value, 2000). The WRV of pulps increases with beating or refining. According to Scallan & Carles (1972), the WRV may be used as an estimate for the FSP except for highly-swollen pulps (whose fibre walls are swollen with water). As previously mentioned, the FSP is an indication of the amount of bound water in the fibre: water in the lumen and in the pores on the fibre wall. Therefore, the WRV was intended to determine the amount of bound water rather than free water in the pulp.

1.7 Conclusion

Further to the evaluation of the need for energy saving in drying by improvement of pulp dewatering properties, the inhibition of pulp drainage by mechanical refining as well as the potential for improvement of drainage by enzymatic refining have been examined.

The literature has presented models that enable visualisation and understanding of the phenomena that affect drainage, such as fibrillation, the choke-point hypothesis and fibre conformability. Linking these models to pulp drainage behaviour in the laboratory, making use of the Canadian Standard Freeness and Water Retention Value tests, could serve to enhance the understanding of drainage properties of the paper web.

Furthermore, the understanding of the location and quantity of water within the fibre lumen, as well as bound water on the fibre wall, could aid in interpreting difficulty of water removal in the forming and pressing sections. For example, excessive refining can cause internal fibrillation, which can enhance the water absorption ability of the fibres, increased the amount of drying required. Ultimately, better prediction and control of dewatering on a commercial paper machine could enable the sending of a lower-consistency web into the drying section.

2 Materials and methods

2.1 Introduction

The methods applied throughout the current study are described in this chapter, while the specific designs of individual trials are discussed in their respective chapters. Pulp batches were mechanically refined in a pilot refining circuit, over a range of refining energies. Some pulps were refined with an enzyme additive. Each batch yielded six pulp samples at refining levels evenly spread over a range of energy levels, which encompass those used on commercial scale. Each pulp sample was subjected to freeness and WRV tests (Figure 2-1).

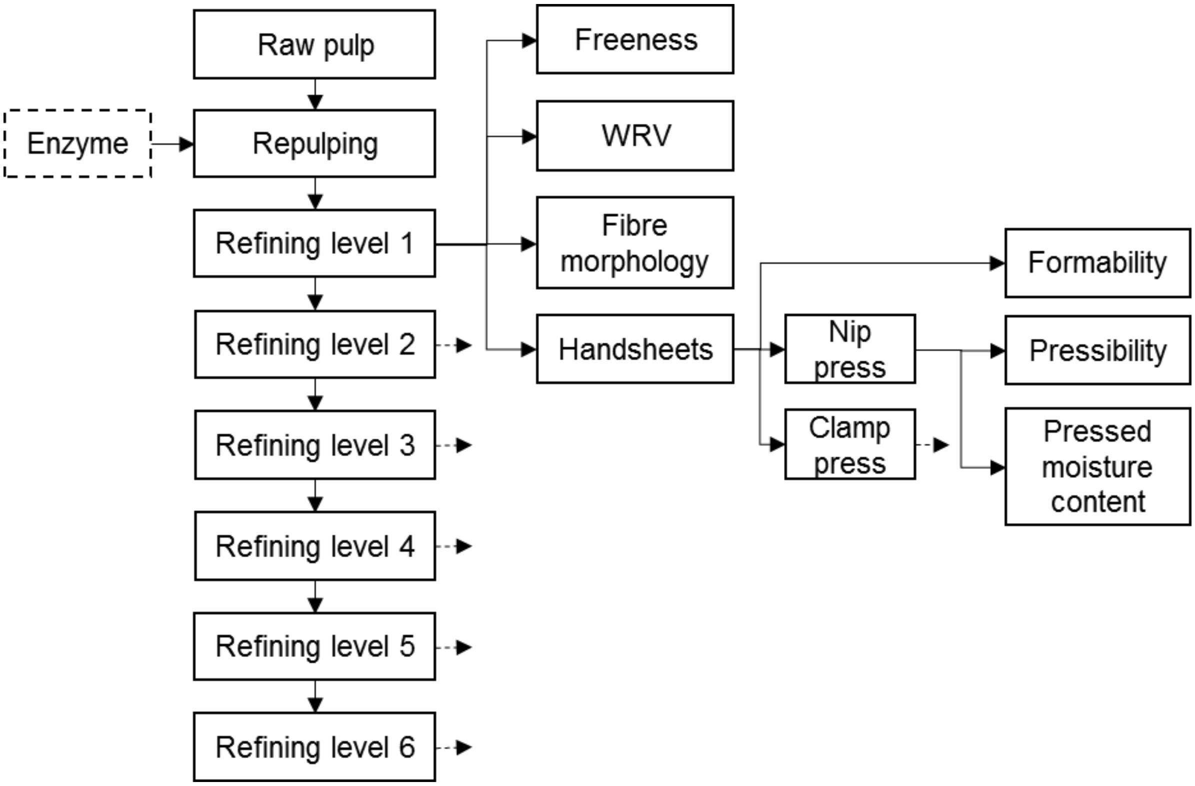


Figure 2-1: Schematic diagram of the experimental design for the refining and testing of pulp.

Handsheets were produced from each pulp sample in order to calculate the formability, pressibility and the moisture content of the pressed handsheet. Handsheets were pressed with a nip press and a clamp press. Fibre-morphology measurements were also performed to relate to drainage properties of the pulp.

2.2 Procedure

2.2.1 Repulping, refining and enzymatic refining

Pulp was refined in order to develop fibre properties and analyze their effect on the dewatering properties of the pulp. Pulp is refined on commercial scale to improve the properties of the paper made from it. Therefore, refining on pilot scale was also done to simulate the dewatering performance of commercial-scale pulps.

Figure 2-2: The pilot refiner circuit at the Sappi Technology Centre (Pretoria).

Repulping, refining and sampling of the pulp was performed in a pilot-scale refining circuit (Figure 2-2), consisting of a replulping vessel, a 12” single-disk pilot refiner and a sampling carrousel (Figure 2-3). Raw pulp fibre was obtained in the form of dry sheets and was converted into slurry form (repulped) by mixing it with water at 50 ºC in the repulper for 30 minutes where the sheet was disintegrated into individual fibres. Approximately 8.5 kg of dry pulp was repulped in 200 L of water. The repulping of the raw fibres was achieved with constant mixing for 30 minutes at a temperature of 50 ºC.

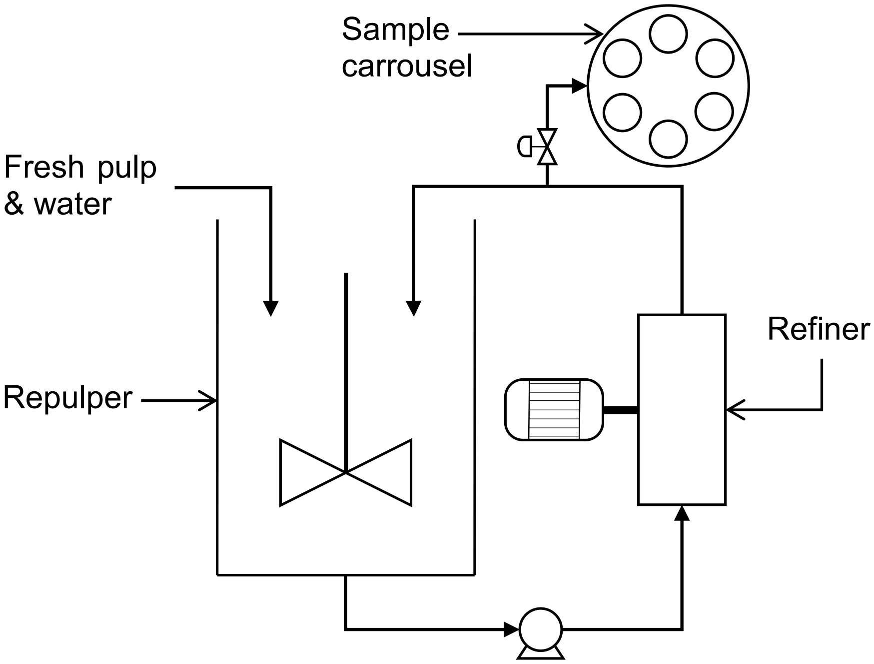


Figure 2-3: Schematic diagram of the pilot refiner circuit used for the present work.

The sampling of the pulp was conducted automatically, as samples were dispensed into 5 L buckets in the sample carrousel. The first pulp sample for each batch was dispensed after the repulping step (at zero refining energy). The mechanical refining was started by pumping the pulp through the refining circuit and having it pass through a Matech 12” single-disk pilot refiner (Figure 2-4). The refining continued with samples being dispensed regularly into the carrousel until the necessary amount of energy (measured in kWh/t) had been transferred to the pulp for the last sample. The refiner was operated in hydracycle mode and used a classical hardwood plate, with a bar angle of 10º, and bar width, groove width and bar height of 2, 2 and 4 mm, respectively. The intensity of the refining was 0.5 Ws/m.

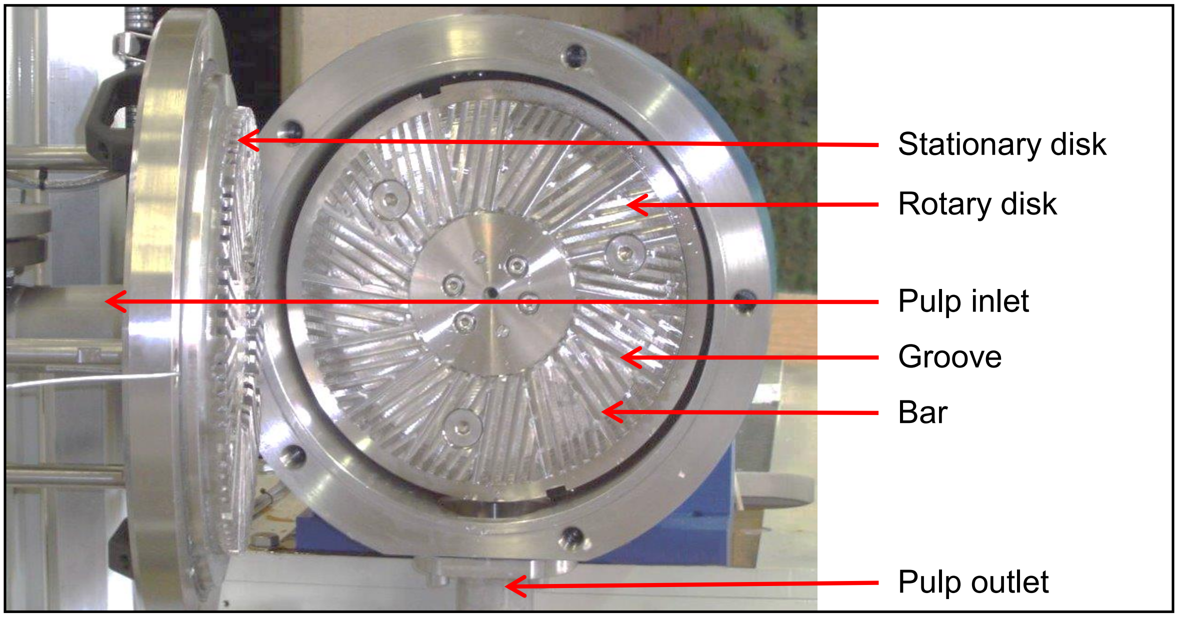


Figure 2-4: Open Matech 12” single disk pilot refiner.

Some pulp samples were refined with an enzyme added (Figure 2-1) in order to modify fibre properties and enable the evaluation of their effect on the dewatering properties of the pulp. Enzymes have been used to modify pulps on commercial scale in order to improve their refining and dewatering properties (Blomstedt, *et al*., 2010). An endoglucanase (Ecopulp R from AB Enzymes) was added to the pulp 10 minutes into the repulping stage, and was allowed to incubate for 20 minutes until the start of refining. The pulp was dosed with the enzyme at concentrations of 100, 200 and 500 g/t. The remainder of the refining process, as well as the methods applied thereafter, were the same as for untreated pulps.

2.2.2 Freeness, WRV and fibre morphology

The freeness of the pulp samples was tested immediately upon their dispensation from the refiner circuit, in order to acquire accurate readings. Canadian Standard Freeness (CSF), or freeness, was developed to measure the drainage ability of pulp at low consistency (0.3 %). According to TAPPI (T227, 1999), freeness is used to follow the changes in drainage rate of various pulps during refining, but the freeness measurement does not necessarily correlate with the pulp behavior on the paper-machine fabric, as it is not a direct measurement of sheet drainage.

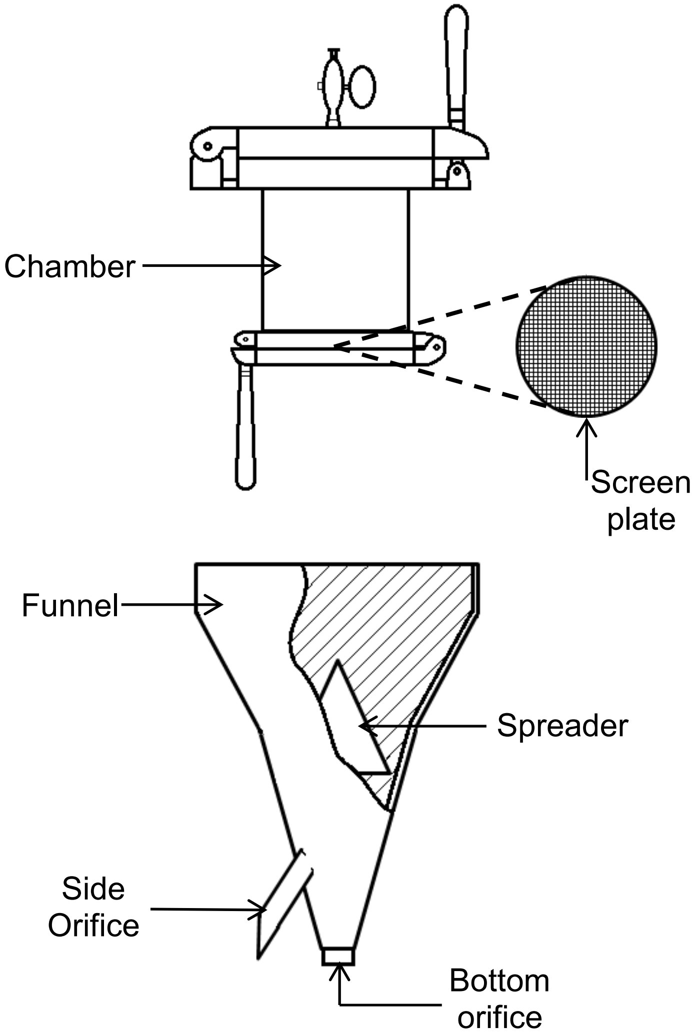


Figure 2-5: A schematic of the Canadian Standard Freeness apparatus. Adapted from TAPPI T227 (1999).

The freeness apparatus consists of a chamber and a funnel (Figure 2-5). A diluted pulp suspension of 1 L, containing 3 g of dry pulp, was poured into the chamber and then allowed to drain through the screen plate into a funnel. The freeness measurement (in mL) reflected the volume of water exiting the drainage chamber at high velocity from the side orifice while the remainder of the water exited via the bottom orifice. A higher freeness measurement indicated a pulp with faster drainage.

The water-retention value (WRV) is an empirical measurement of the ability of a pad of fibres to retain water after centrifugation under specific conditions (Water Retention Value, 2000). A mass of wet pulp, containing the equivalent of 1.125 g of dry pulp, was placed in a sintered glass crucible and drained of water using a vacuum pump attached to a Buchner funnel. The crucible containing the wet pulp pad was then centrifuged for 30 minutes at 900 x g and 2350 rpm and the expelled water was absorbed by tissue paper placed under the sintered glass crucible. The pulp pad was then weighed and dried (overnight at 105 °C). The mass of water retained after centrifugation per gram of dry pulp represents its WRV.

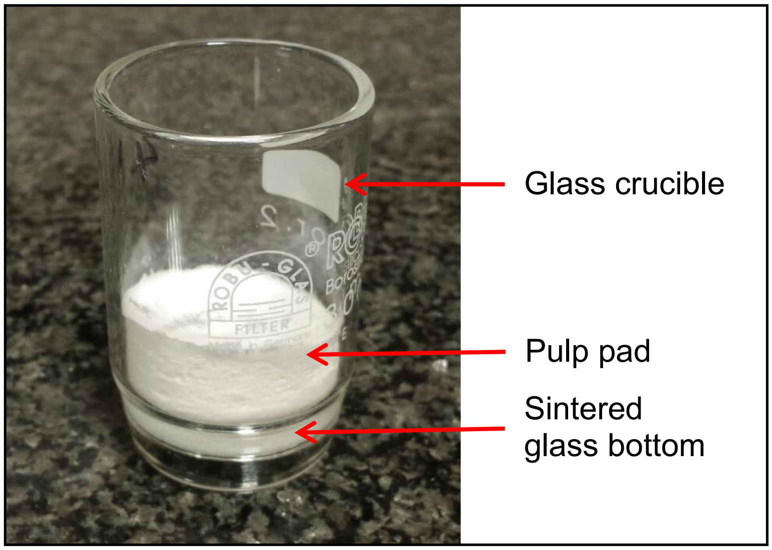


Figure 2-6: A sintered glass crucible containing a dry pulp pad, used in the determination of WRV.

Some morphological properties of the fibres from each sample were analysed in order to relate them to the dewatering performance of the pulp, with the aid of the MorFi Fibre Analyser (Techpap, France). Each sample analysis required 0.4 g of dry pulp in a suspension of water. Fibre properties that were considered as valuable in explaining the dewatering behavior of the pulp that were analysed included fibre width, coarseness, curl and the extent of fibrillation on the fibre surface. The number of fines (fibres less than 200 µm in length) present in the pulp was also determined.

2.2.3 Handsheets and pressing

The pulp sheet on the paper machine was simulated at laboratory scale by using handsheets. Handsheets are round paper sheets, with a diameter of 20 cm, made in the laboratory from the pulp samples acquired after refining. Handsheets were made using the Blattbildner Rapid Köthen System PTI handsheet former (Germany) (Figure 2-7).

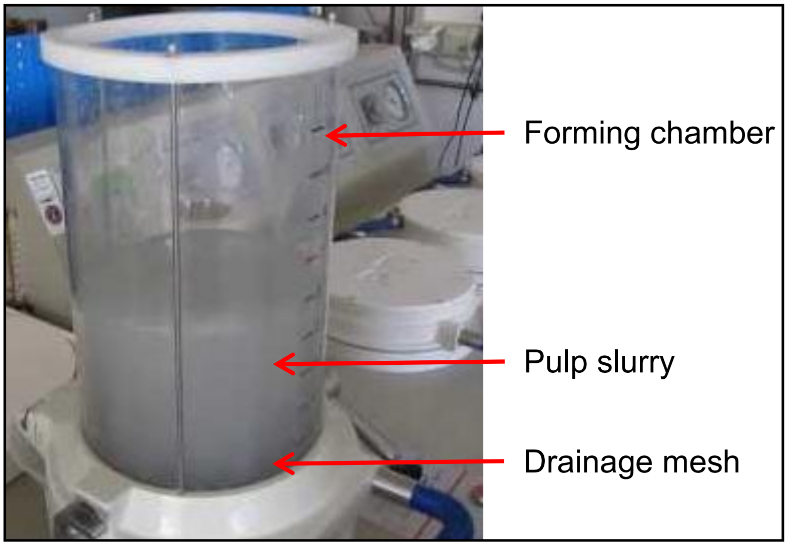


Figure 2-7: The handsheet former is shown. The pulp slurry drains through the drainage mesh leaving behind a wet handsheet.

The handsheet former is a cylindrical container in which the pulp sample is diluted and distributed over a screen, and drained with the aid of a suction force applied from underneath the screen. The grammage (mass per unit area) of handsheets produced was approximately 80 g/m^2. Handsheets were weighed immediately after forming as well as after pressing, to determine the respective moisture contents of sheets that were subjected to different pressing methods.

The formed handsheets were pressed using two different apparatus, a Techpap laboratory-scale nip press and a William's Apparatus clamp press, enabling the effect of different pressing techniques to be observed. An F-test was performed with a 95 % confidence interval to analyse the significance of the difference between handsheets pressed by each pressing technique. Each handsheet was pressed with two sheets of blotting paper (approximately 250 g/m^2 each) on either side, to provide a medium to absorb the expelled water. The nip press squeezed water from the sheet by passing it between two heavy rollers at an approximate pressure of 10000 kPa (Figure 2-8). The handsheets were weighed immediately after pressing.

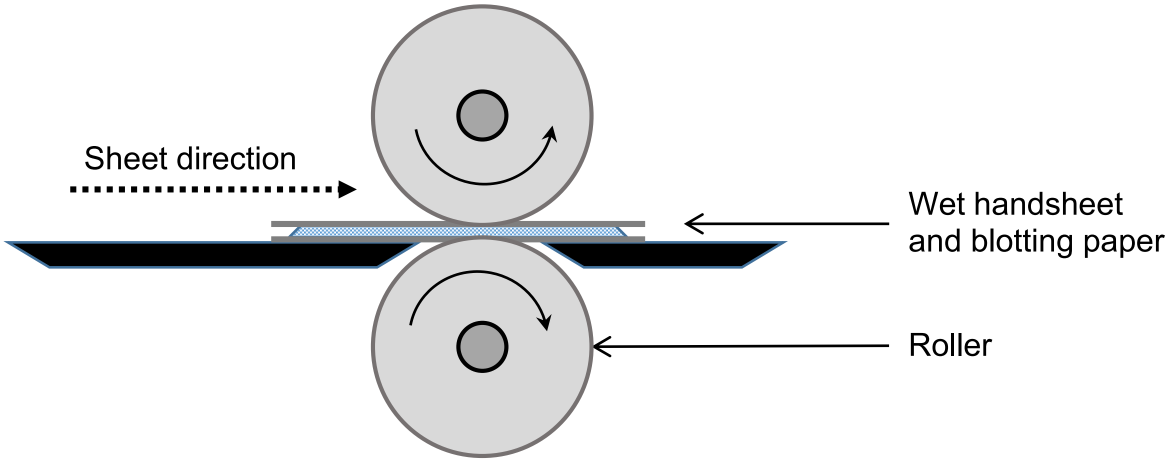


Figure 2-8: Side view of the laboratory-scale nip press. The wet handsheet was placed between sheets of blotting paper.

The hydraulic press clamps and presses the sheet in the z-direction (perpendicular to the sheet surface) and was operated at a maximum pressure of 4240 kPa. Once the handsheet and blotting paper was placed in the clamp press, the pressure setting was immediately adjusted to its maximum pressure, and left for one minute before the handsheet was removed and weighed.

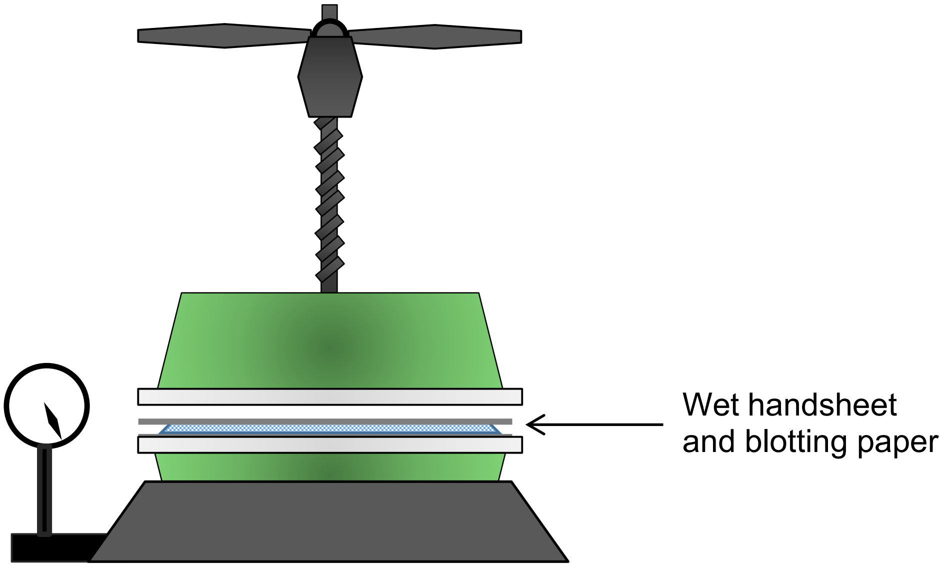


Figure 2-9: Schematic of the laboratory scale nip (a) and hydraulic press (b). The wet handsheet was placed between sheets of blotting paper.

2.2.4 Formability, pressibility and moisture content determination

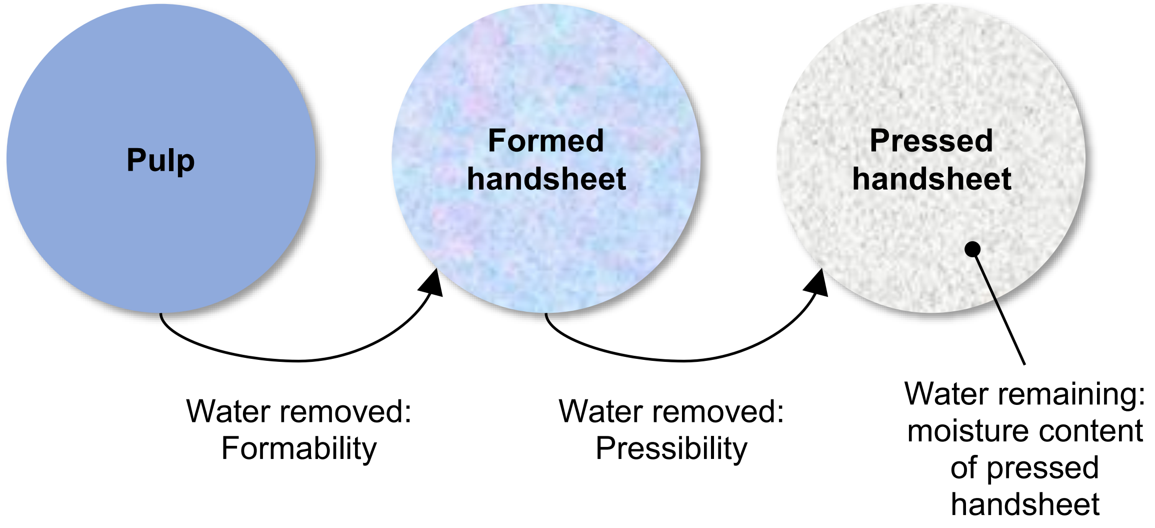


Figure 2-10: Schematic showing the pulp as well as the formed and pressed handsheets.

The formability of the handsheets was determined by measuring the moisture content of the formed handsheet gravimetrically and subtracting it from the moisture content of the pulp sample from which it was formed. The pressibility of the handsheets, or the amount of water removed by pressing, was determined by subtracting the moisture content of the formed handsheet from that of the pressed handsheet. The moisture content of the pressed handsheet was also determined gravimetrically (Figure 2-10).

3 Assessment of dewatering of pulp

3.1 Introduction

The ability to predict and control the dewatering properties of the wet paper sheet during its forming and pressing on the paper machine could result in significant energy savings in the paper-making process (Ghosh, 2011, Karlsson, 2009). Typically, the Canadian Standard Freeness (CSF), or freeness, and Water Retention Value (WRV) of the pulp have been used to predict its dewatering performance on the paper machine (Hubbe, 2007). However, these conventional dewatering tests were not designed to simulate the dewatering of pulp on the wire or in the pressing section (TAPPI T227, 1999, Scallan & Carles, 1972) and there is opportunity for a more accurate technique to be developed.

The freeness of pulp is used to follow the changes in its drainage rate during refining, but it does not necessarily correlate with the dewatering of pulp on the paper machine as it is not a direct measurement of sheet drainage on the wire or in the pressing section (TAPPI T227, 1999). Furthermore, the Water Retention Value (WRV) is representative of the fibre saturation point of pulp fibres (Scallan & Carles, 1972) and not its dewatering ability. The WRV is an empirical measure of the mass of water per mass of dry pulp after centrifugation under specific conditions (Water Retention Value, 2000).

The aim of the current investigation was to examine formability (the water removed by forming) of handsheets made from hardwood pulp refined on pilot-scale as a means for predicting their dewatering performance. The correlations that the formability of the handsheets and the conventional dewatering measurements (freeness and WRV) of the pulp had with the pressibility (the water removed by pressing) and total dewatering of the handsheets were compared. The handsheets were pressed using a clamp press or a nip press in order to analyse the effect of different pressing techniques and pressures.

3.2 Materials and methods

Baycel, a hardwood pulp composed of *Eucalyptus grandis*, was refined in a pilot-scale refiner. Each refining run produced six samples of pulp, each at a different level of refining, ranging from 0 to 80 kWh/t. Pulp treatments were not repeated due to the expense involved in the refining of pulp in the pilot circuit. The pulps were each subjected to formability, pressibility, WRV and freeness tests. The moisture content of the handsheets after pressing was determined gravimetrically. Handsheets were produced in triplicate for each pulp sample. The specifications of the refining and dewatering tests of the pulp are covered in Chapter 2.

3.3 Results and discussion

When handsheets were pressed, there was a strong correlation ($R^2 = 0.94$) between handsheet formability and relative handsheet pressibility (Figure 3-1). Therefore, the amount of water removed in pressing was determined by the water content of the sheet after forming. The more water that the handsheet contained after forming, the more water there was that was available to be removed by pressing and vice versa. There was no significant difference ($p \leq 0.05$) between the pressing data sets obtained from the nip press and the clamp press. Therefore, the two data sets can be represented by one line (Figure 3-1) and the pressing technique did not affect the moisture removed during pressing of the handsheets. Only the nip press data were considered as it more closely replicated the pressing mechanism on a paper machine, which also makes use of the nip press. The relevant data for handsheets pressed using the clamp press are available in the Appendix (Figure 7-1 to 7-6).

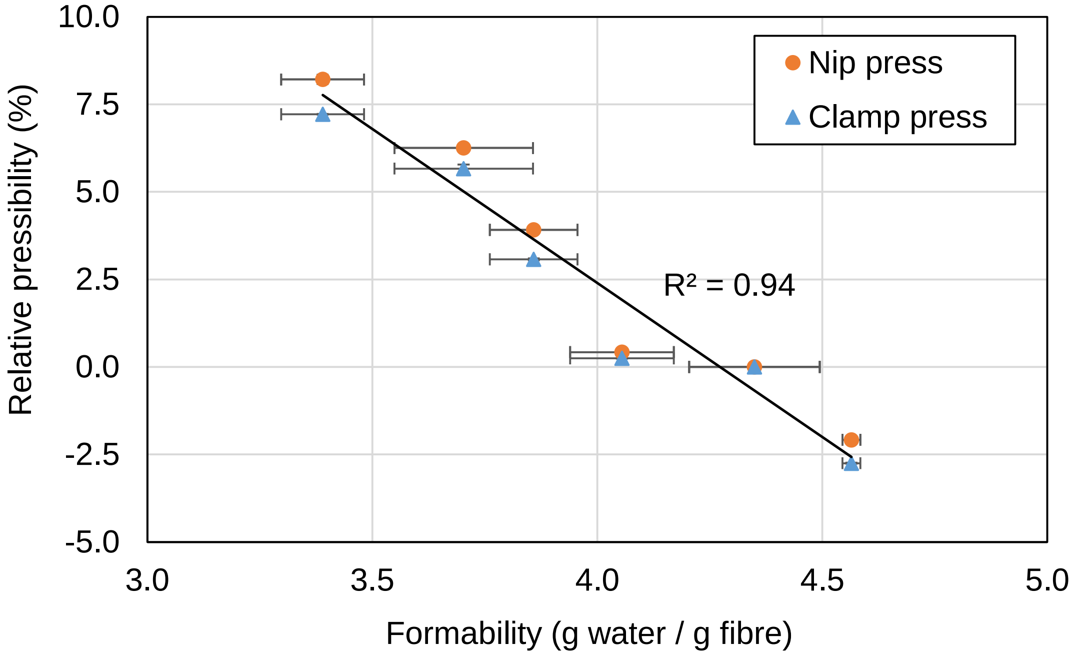


Figure 3-1: The influence of handsheet formability on relative handsheet pressibility obtained by using two different pressing techniques.

The formability of the handsheet was not as effective in predicting its moisture content after pressing as it was in predicting its pressibility. However, the formability of the handsheet still gave some indication of its total dewatering behavior (Figure 3-2), with an R^2 value of 0.65. A higher formability resulted in a lower handsheet moisture content after pressing, indicating that more water removed during forming improved the total dewatering of the handsheet.

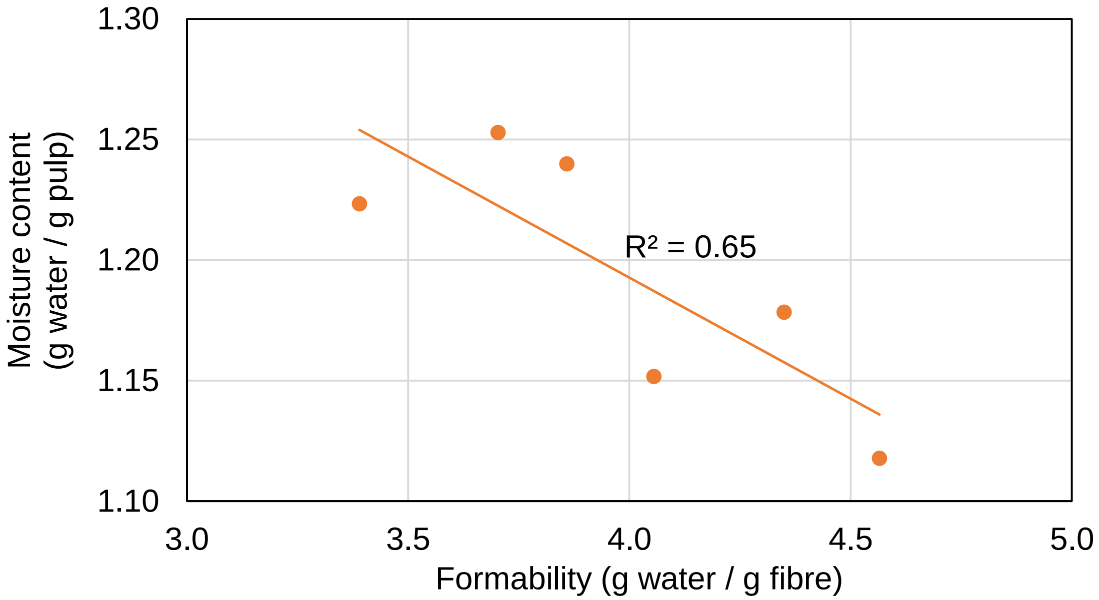


Figure 3-2: The influence of handsheet formability on its moisture content after pressing.

Although the formability of the handsheet was a good predictor of its pressibility (Figure 3-1), the freeness and WRV were not (Figure 3-3), with R^2 values of 0.15 and 0.18 respectively. Therefore, conventional dewatering measurements did not correlate well with handsheet formability either (Figure 3-4) with R^2 values of 0.34 and 0.39 for freeness and WRV respectively. However, conventional dewatering measurements do give an indication of the drainage performance during forming of the handsheet. The reason for the weak correlation between freeness and handsheet formability could have been that the freeness test was performed by draining water from pulp only with the aid of gravity, whereas handsheets were formed with the aid of a suction force to drain the water and through a much thinner layer of pulp than used for the freeness test.

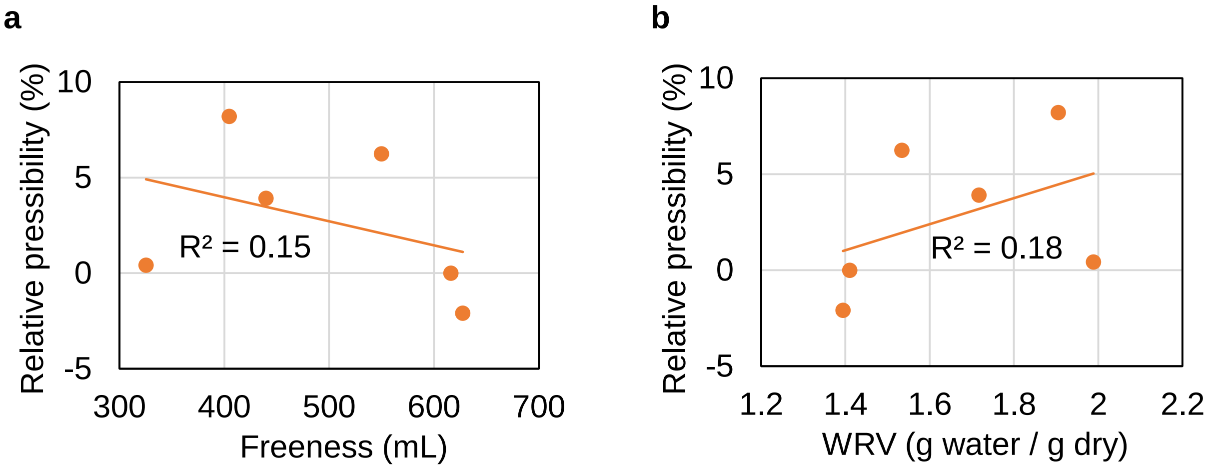


Figure 3-3: The influence of freeness (a) and WRV (b) on relative pressibility of handsheets pressed by the nip press. Each pressibility is given relative to the pressibility of the handsheet formed from the unrefined pulp.

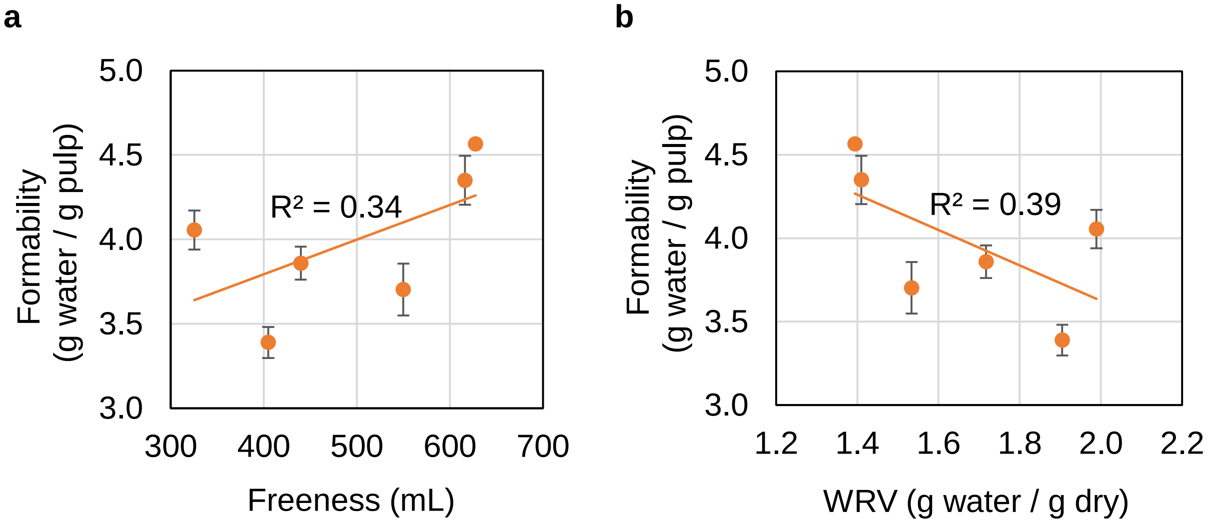


Figure 3-4: The influence of freeness (a) and WRV (b) on handsheet formability.

There were very weak correlations between the moisture content of the pressed handsheet and both freeness and WRV (Figure 3-5) with R^2 values of 0.04 and 0.03 respectively. Therefore, conventional dewatering measurements were very poor predictors of the total dewatering performance of the handsheet. Formability offered a better prediction of the total dewatering performance of the handsheet than did either of the conventional dewatering techniques (Figure 3-2).

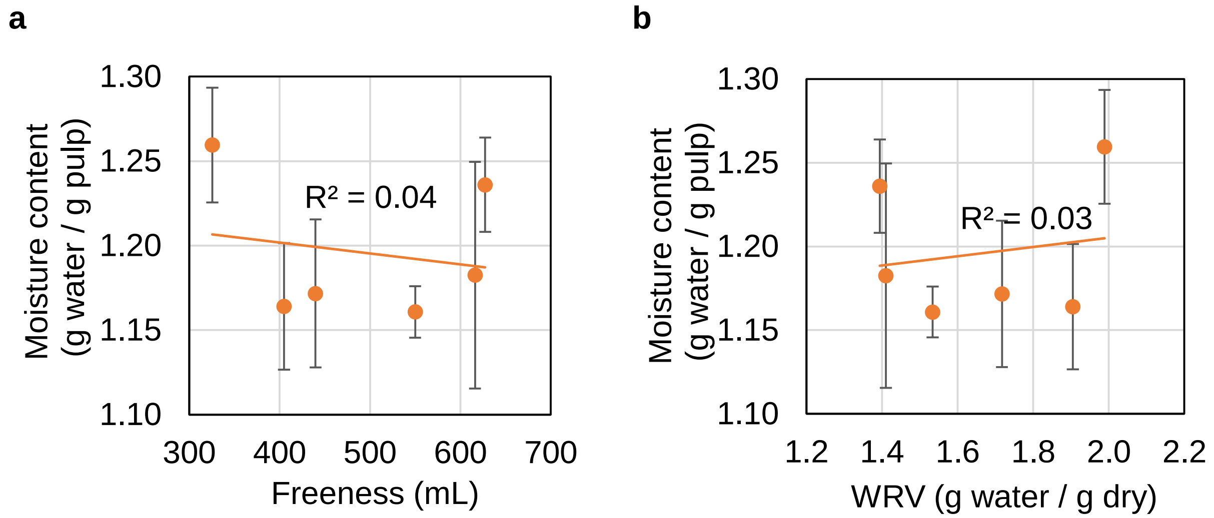


Figure 3-5: The influence of freeness (a) and WRV (b) on the moisture content of the handsheet after pressing by the nip press for a range of refining energies.

The WRV of pulp correlated very well with its freeness (Figure 3-6) but water is drained from the pulp with the aid of centrifugal force during the WRV test. The reason for the strong correlation of WRV with freeness and the weak correlation between WRV and handsheet formability (Figure 3-4b) could be due to water drainage through a thicker layer of pulp during the WRV test, something it has in common with the freeness test.

In general, the WRV data was not distinct from freeness data, and the two measurements were very strongly correlated with each other (Figure 3-6). Therefore, it is advisable to conduct only freeness measurements in future work because they are less expensive and faster to perform than WRV measurements.

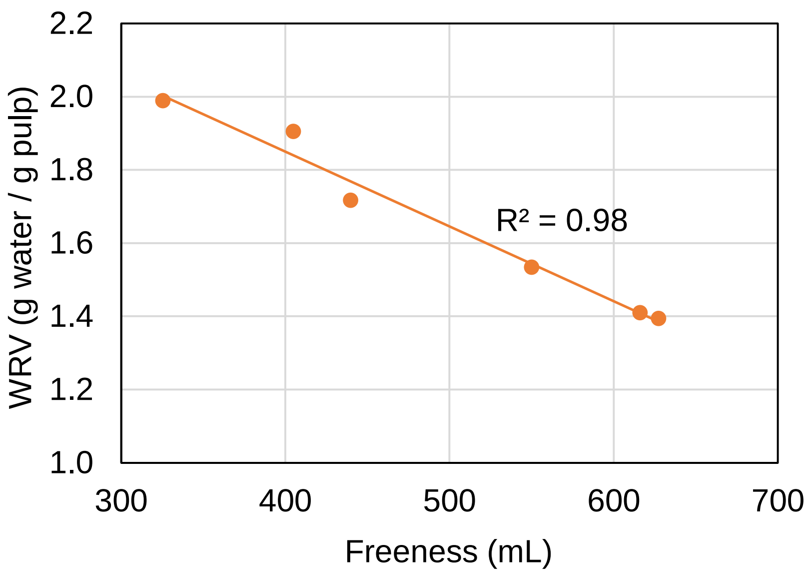


Figure 3-6: The relationship between freeness and WRV.

3.4 Conclusions

Conventional dewatering measurements and handsheet formability both predicted the relative pressibility of handsheets, but handsheet formability had a much stronger correlation with it than did freeness or WRV. The formability of a handsheet is a direct measurement of its drainage during forming, and so it correlated well with its pressibility. The amount of water removed during forming of the handsheet determined the amount of water available for removal by pressing. The freeness and WRV of pulp were not direct measurements of the drainage of a sheet on the wire, and therefore did not yield strong correlations with the pressibility of handsheets.

The freeness and WRV of pulps had very weak correlations with the moisture content of the pressed handsheets, and were unable to predict their total dewatering performance. Neither of these conventional dewatering measurements were designed to predict the dewatering performance of the sheet on the paper machine (TAPPI T227, 1999, Scallan & Carles, 1972), and they were not able to do so in the present work. Freeness and WRV measure the drainage from a pulp pad, which is significantly thicker than a handsheet, possibly explaining their inability to predict it the dewatering of the handsheet. Handsheet formability was able to predict the moisture content of the pressed handsheets. The formability of handsheets is a direct measurement of their drainage performance, and correlated well with their total dewatering performance.

There was little difference in the effect of pressing on the handsheets between the nip press and the clamp press, despite the differences in the force and type of pressing applied by each method. It is likely that the handsheet was pressed to a greater extent than was required for the removal of any water that could possibly be removed, by pressing, for either technique. It is recommended that the nip press be used for future work as it is faster to use than the clamp press and more closely simulates pressing on a paper machine.

There was a good correlation between WRV and freeness data, rendering one of them redundant. In future work it is recommended that only the freeness measurement be used to estimate drainage of the pulp, as this test was significantly faster and less expensive to perform than were WRV measurements.

4 Influence of enzymatic refining on pulp dewatering

4.1 Introduction

Treating the pulp with an enzyme before refining has been done in the paper industry to enhance sheet dewatering on the paper machine and save energy in the mechanical refining of pulp (Blomstedt, *et al*., 2010; Bajpai, *et al*., 2006; Fuente & Robert, 1990). Enzymatic treatment of pulp increases its susceptibility to refining, resulting in lower energy consumption during refining as the desired fibre properties are achieved at a lower refining energy (Blomstedt, *et al*., 2006). According to Fuente & Robert (1990), and Mooney, *et al.* (1999), the enzyme consumes fines and other small particles resulting in improved drainage of water from the wet web. The improvement of pulp dewatering on the forming fabric could result in a decrease of energy consumption in the drying section of the paper machine as a web of higher consistency is achieved, mitigating the amount of water to be removed by thermal drying. The drying section of the paper machine is responsible for a significant portion of energy consumption in the paper-making process and small decreases in the moisture content of the sheet after pressing could result in large energy savings (Ghosh, 2011, Karlsson, 2009).

The objective of the current study was to determine the effect of enzymatic refining on the dewatering properties of handsheets produced from a pilot-refined hardwood pulp. The pulps were refined in batches, at different enzyme dosages. The dewatering properties of handsheets, including their formability, pressibility and moisture content after pressing, were compared for pulps over the range of enzyme dosages. The freeness and Water Retention Values (WRV) of the pulps were also determined. The effect of the enzyme additive on fibre morphology was also analysed and compared for all pulps, and an attempt was made to relate it to their dewatering behaviour.

4.2 Materials and methods

An endoglucanase (Ecopulp R from AB Enzymes) was added to a hardwood pulp (Baycel, composed of *Eucalyptus grandis*) at several dosages and was refined with a 12" single-disk pilot refiner. Each refining run produced six samples of pulp, each at a different level of refining, ranging from 0 to 80 kWh/t. The control pulp was not treated with an enzyme, while pulps treated with an enzyme at concentrations of 100, 200 and 500 g/t were also refined. The enzyme was allowed to incubate for 20 minutes at 50 ºC for each batch of pulp. Pulp treatments were not repeated due to the expense involved in the refining of pulp in the pilot circuit.

The pulps were each subjected to formability, pressibility, WRV and freeness tests. The handsheets were pressed using a nip press as well as a clamp press. The moisture content of the handsheet after pressing was determined gravimetrically. Fibre morphology was determined for each of the samples by using a MorFi Fibre Analyser instrument (Techpap, France). The pressing treatments, formability determination and fibre morphology testing were each replicated three times on different handsheets. The procedures for repulping and refining, as well as all of the tests performed on the pulps were covered in Chapter 2.

4.3 Results and discussion

4.3.1 Effect of enzyme on pulp dewatering

Only the results for the handsheets pressed in the nip pressed are shown due to the similarity between the data from the different pressing methods, as observed in Chapter 3, while results for the clamp press are in the Appendix (Figure 7-7 to 7-12). The handsheets produced from the control sample of pulp had the best dewatering performance of all those produced (Figure 4-1), as they had the lowest moisture content after pressing. In general, increasing the enzyme concentration in the pulp increased the moisture content of the pressed handsheets, indicating a decrease in dewatering performance during forming and pressing. The increase in refining energy did not have a notable impact on the moisture content of the handsheets after pressing.

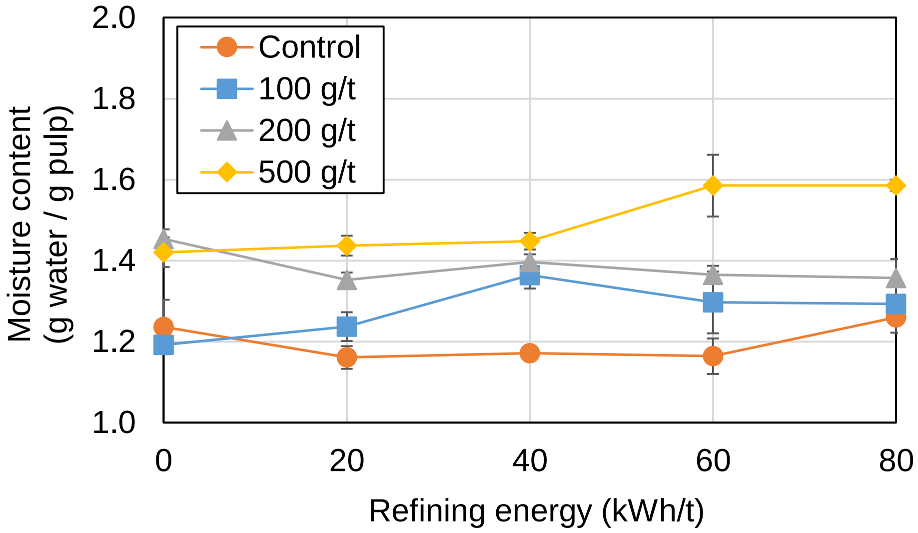


Figure 4-1: The influence of enzyme dosage and refining energy on the moisture content of handsheets after pressing with a nip press. Error bars are based on the standard deviation for each point, based on pressing replicated three times.

The pulp treated with the enzyme at a concentration of 200 g/t produced handsheets with the worst formability (Figure 4-2). The control pulp and the pulp treated with the enzyme at 500 g/t produced handsheets with the best formability. The effect of enzyme dosage on handsheet formability (Figure 4-2) did not correspond with its effect on the moisture content of the handsheets after pressing (Figure 4-1). The formability of the pulp treated with the enzyme at a concentration of 200 g/t was notably high compared to the other pulp (Figure 4-2), but the pulp did not have the lowest moisture content after pressing (Figure 4-1). The pattern of an increasing enzyme concentration resulting in an increase in the moisture content of the handsheet after pressing (Figure 4-1) was not observed in the formability data (Figure 4-2).

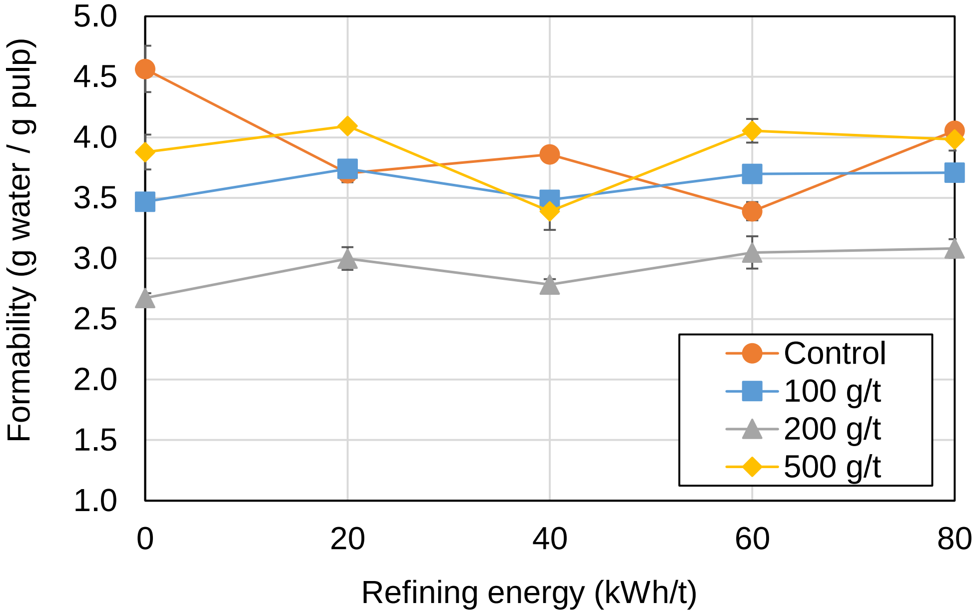


Figure 4-2: The influence of enzyme dosage on the formability of handsheets over a range of refining energies. Error bars are based on the standard deviation for each point, based on pressing replicated three times.

The pressibility of handsheets reflected their formability (Figure 4-3), suggesting that formability was the determining factor for handsheet pressibility and consequently the total dewatering performance of the handsheets. There were strong correlations between handsheet formability and pressibility for each pulp (Figure 4-4), suggesting that the more water available to be pressed in the handsheet after forming, the more water was removed by pressing. The increase in refining energy did not have a significant impact on the formability or the pressibility of the handsheets when compared to the enzyme dosage.

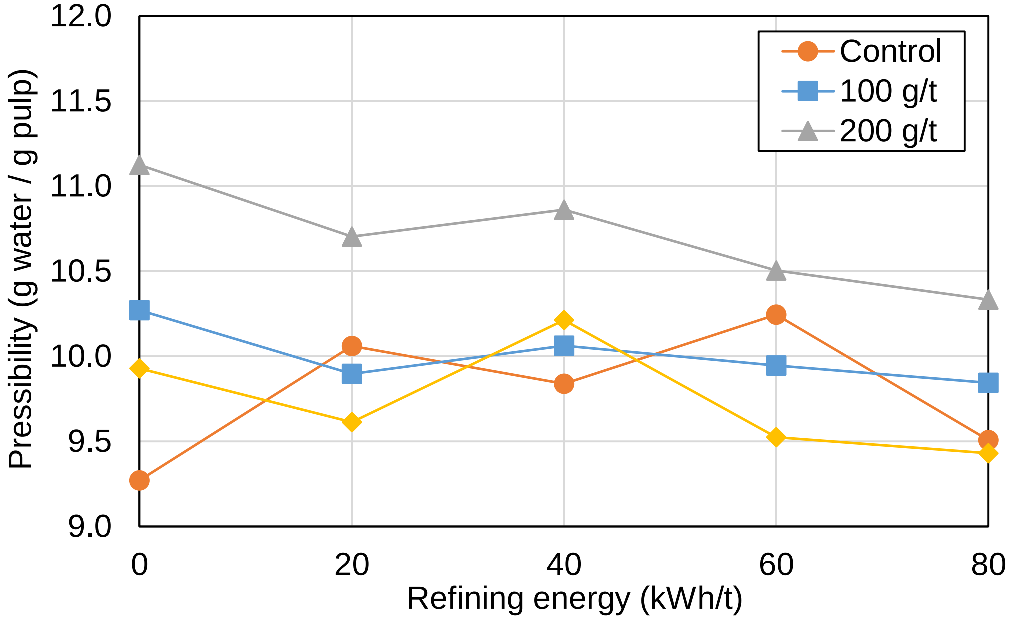


Figure 4-3: The influence of enzyme dosage on the pressibility of handsheets over a range of refining energies. Handsheets were pressed with a nip press.

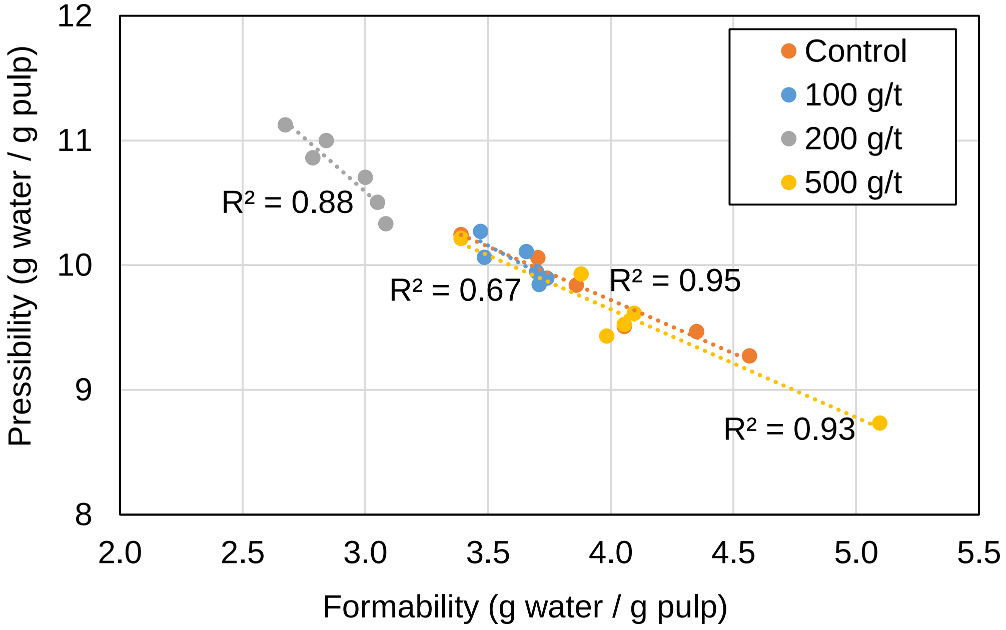


Figure 4-4: The correlation between the formability and pressibility of handsheets for three different enzyme-treated pulps as well as an untreated pulp. Handsheets were pressed with a nip press.

The pulp treated with the enzyme at a concentration of 500 g/t had the lowest freeness and highest WRV of the pulps tested (Figure 4-5). Moreover, the handsheets produced from the pulp treated with the enzyme at 500 g/t also had the worst total dewatering performance (Figure 4-1). The remaining pulps achieved similar freeness and WRV results. The freeness and WRV data support the observations of the moisture content of handsheets after pressing, in that a higher enzyme dosage resulted in the inhibition of the drainage of water. It was also noted that the freeness and WRV correlated strongly for pulps treated with different concentrations of the enzyme (Appendix, Figure 7-15), similar to observations made in Chapter 3.

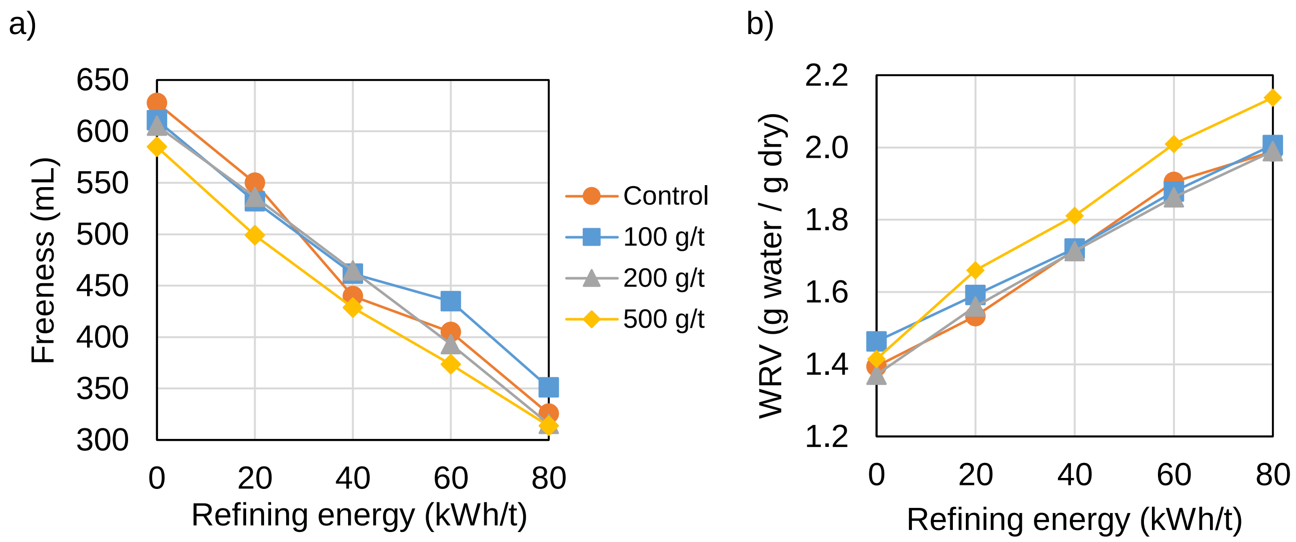


Figure 4-5: The influence of the enzyme dosage on the freeness (a) and WRV (b) of the pulps.

4.3.2 Analysis of fibre properties

The amount of enzyme applied to the pulp influenced the number of fines it contained (Figure 4-6). A higher enzyme dosage resulted in the generation of more fines in the pulp. It was likely that the reaction of the enzyme with the fibre surface, and consequent weakening the amorphous cellulose structure (Sprey, 1990), made the fibre more susceptible to refining. In turn, more fines were produced during refining as they were more easily removed from the fibre surface at higher enzyme concentrations (Figure 4-12).

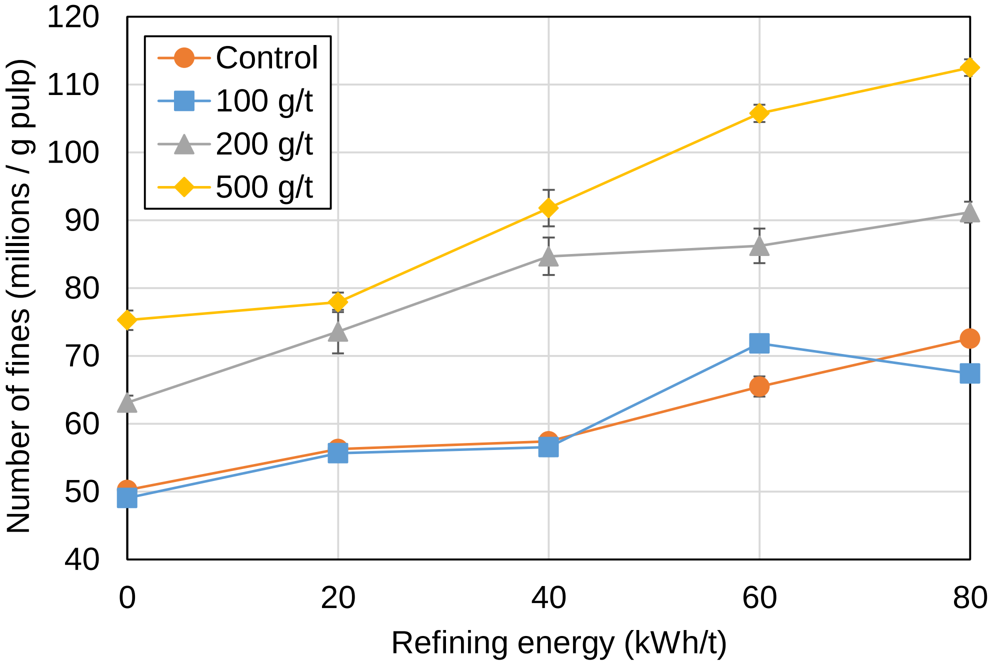


Figure 4-6: The influence of enzyme dosage on the number of fines in the handsheet. Error bars are based on the standard deviation for each point, based on pressing replicated three times.

An increase in the number of fines in the pulp was strongly correlated (R^2 = 0.70) to the increase in moisture content of the pressed handsheet (Figure 4-7). The high concentration of fines in the high-enzyme-dosage pulp likely inhibited dewatering of the pulp and handsheet by blocking drainage channels between the fibres, as described by the choke-point hypothesis (Hubbe, 2007). The inhibition of pulp dewatering due to the high number of fines in the pulp was also evident in the high moisture content of the pulp treated with the enzyme at 500 g/t (Figure 4-1), as well as its low freeness and high WRV (Figure 4-5). The coarseness of the fibres was also measured, and was not correlated to the moisture contents of the pressed handsheet (Appendix, Figure 7-13 & 7-14). Therefore, it was likely that the effect of stiff fibres stacking in a manner that allowed better drainage from the web, or pliable fibres that conformed and restricted drainage, as described in Chapter 1 (Hubbe, 2007) was not the cause for the inhibition of drainage during handsheet forming and pressing.

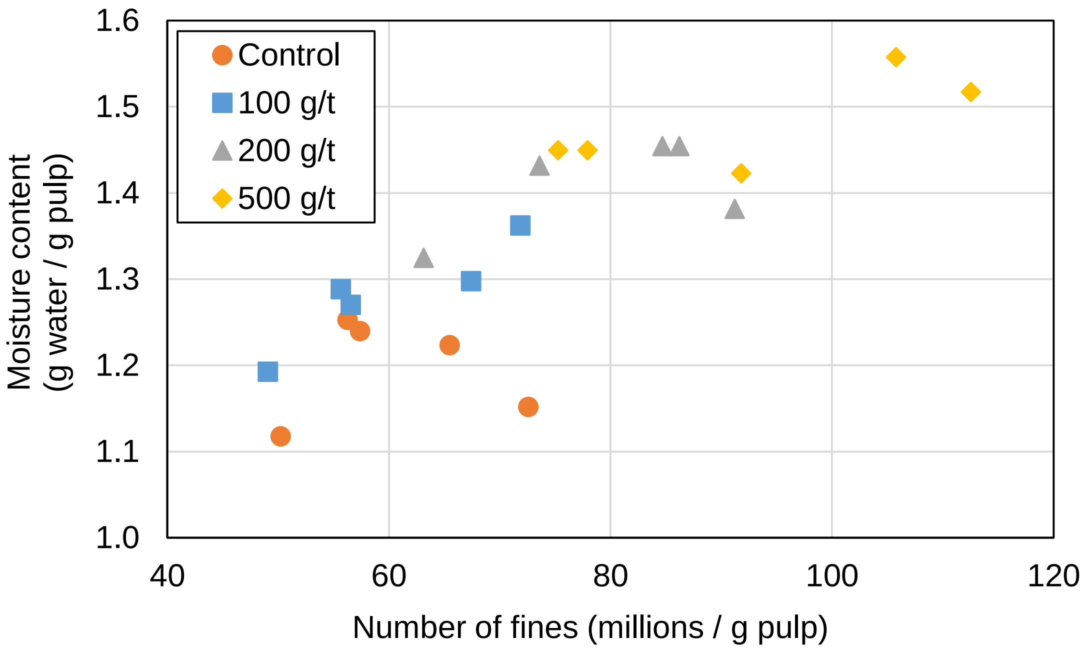


Figure 4-7: The influence of the number of fines in the pulp on the moisture content of the handsheets after pressing with the nip press.

The amount of fibrillation present on the fibre surface was highest for the pulp with the enzyme at 100 g/t, and lowest for the pulp treated at 200 g/t (Figure 4-8). It was likely that the low enzyme dosage of 100 g/t weakened the amorphous cellulose of the fibre so that more fibrillation occurred (Sprey, 1990) when compared to the fibres of the control pulp (Figure 4-12a, b). However, the 200 g/t enzyme-treated pulped yielded fibres with far less fibrillation then the control sample. It was possible that this higher enzyme dosage of 200 g/t yielded fibres which were weakened to the extent that fibrils broke off from the fibre surface during refining, resulting in fibres with very little fibrillation on their surface (Figure 4-12c). The pulp treated with the enzyme at 500 g/t yielded fibres with a similar amount of fibrillation to those of the control sample. The high enzyme dosage could have caused secondary, more extensive, fibrillation of the fibre, after the elimination of the initial fibrils and erosion of the outer-most amorphous cellulose (Sprey, 1990) (Figure 4-12d).

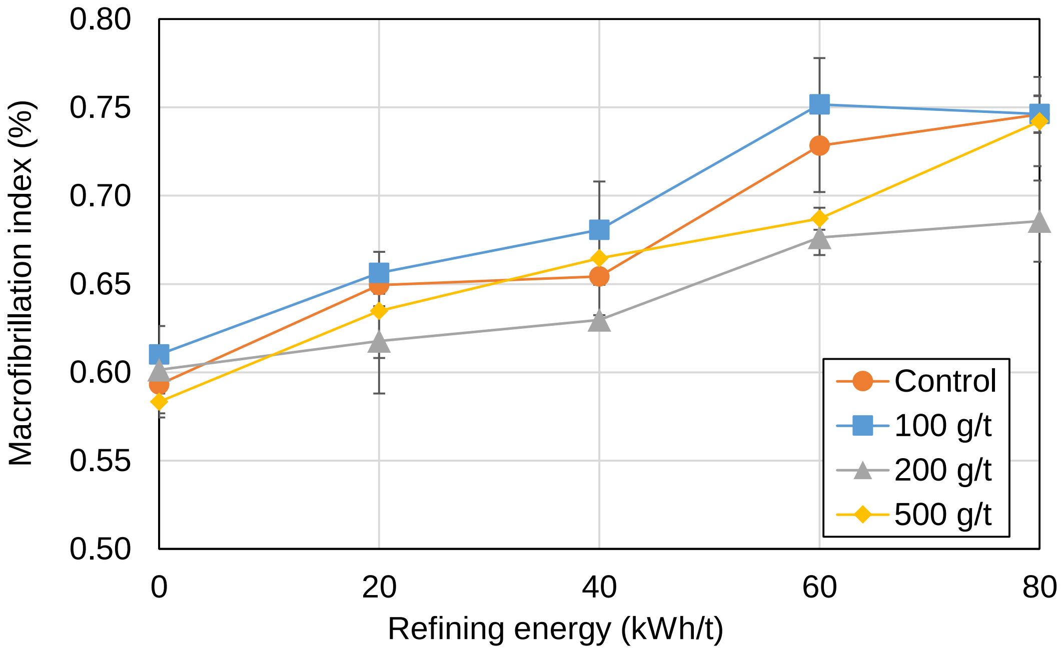


Figure 4-8: The influence of enzyme dosage on the amount of fibre fibrillation, defined as the macrofibrillation index, in the handsheet. Error bars are based on the standard deviation for each point, based on pressing replicated three times.

The pulp treated with the enzyme at 200 g/t had the lowest amount of fibrillation on the fibre surface (Figure 4-8), possibly resulting in smoother fibres. The formability of the handsheets could have been inhibited as these smoother fibres packed in such a manner that sealing of drainage channels occurred (Hubbe, 2007), in which case it could have been difficult for water to exit the pulp. The low formability of the handsheets produced from the 200 g/t enzyme-treated pulp reflected this scenario (Figure 4-2). However, it was not supported by the moisture content, pressibility, freeness or WRV data (Figure 4-1, Figure 4-3 and Figure 4-5), as the dewatering of the pulp was not lowest for the 200 g/t sample. There was not a strong correlation ($R^2 = 0.01$) between the extent of fibrillation on the fibre surface and the total dewatering performance of the handsheet (Figure 4-9).

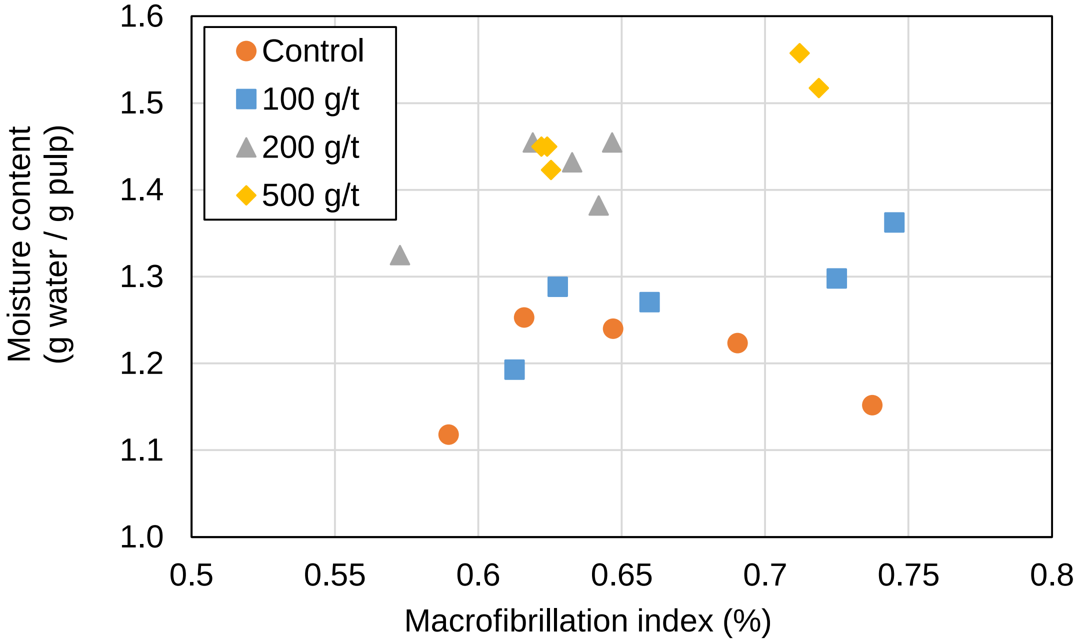


Figure 4-9: The influence of the extent of fibrillation of the fibres on the moisture content of the handsheets after pressing with the nip press.

The average width of the fibres was lowest for the pulps treated with the enzyme at 200 and 500 g/t (Figure 4-10), indicating that a high enzyme dosage results in the loss of amorphous cellulose on the fibre surface during refining, causing it to become narrower. The low fibre width of these high-dosage pulps supported the loss of fibre material. Fibre width was not strongly correlated (R^2 = 0.01) with the moisture content of the handsheet after pressing (Figure 4-11) and was not a determining factor in the dewatering of handsheets.

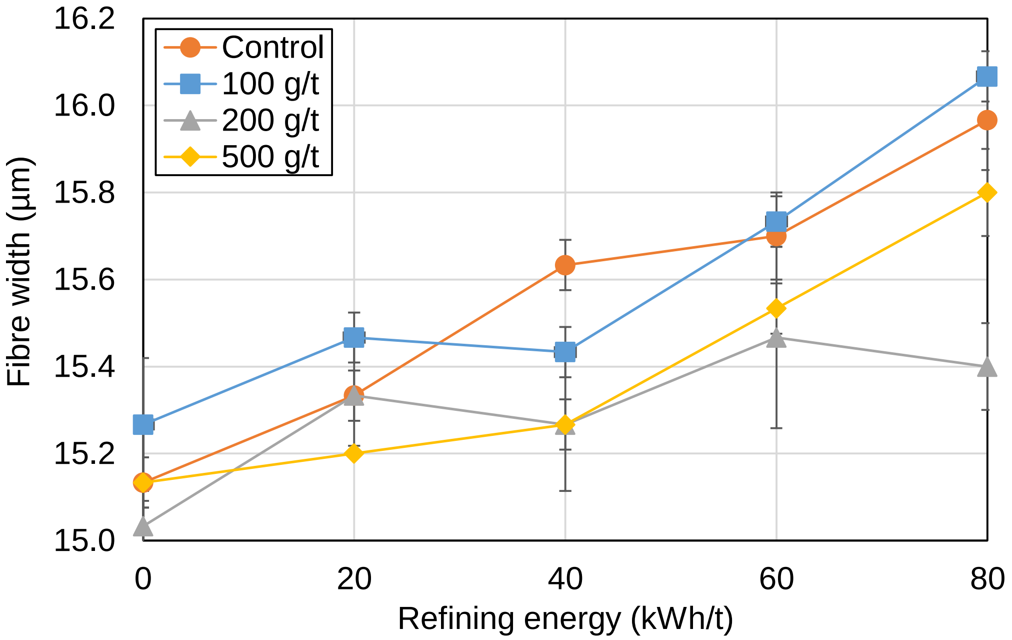


Figure 4-10: The influence of enzyme concentration on the average fibre width of the pulp. Error bars are based on the standard deviation for each point, based on pressing replicated three times.

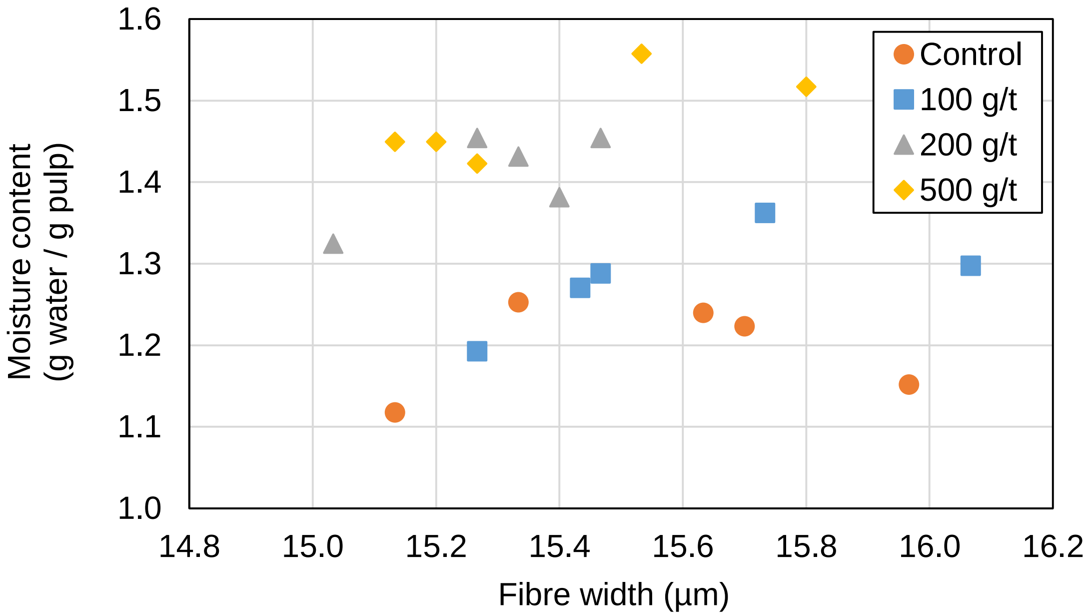
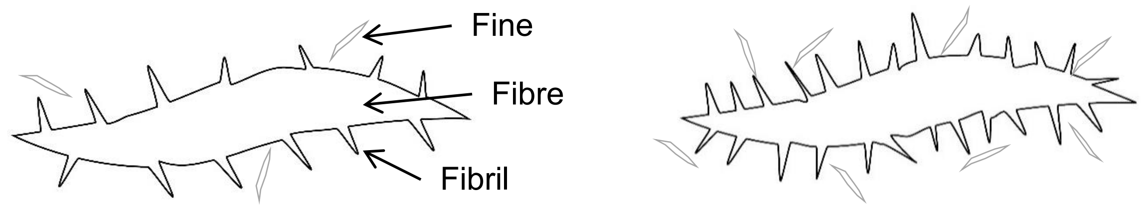


Figure 4-11: The influence of the width of the fibres in the pulp on the moisture content of the handsheets after pressing with the nip press. Error bars are based on the standard deviation for each point.

A model for the modification of fibres by enzyme treatment and refining was proposed in the current study. The extent of fibrillation on the fibre surface (Figure 4-8), the number of fines in the pulp (Figure 4-6), and the width of the fibres (Figure 4-10) was considered for the control pulp and the three enzyme-treated pulps (Table 4-1). A visual representation of the fibres in their respective pulps was constructed (Figure 4-10).

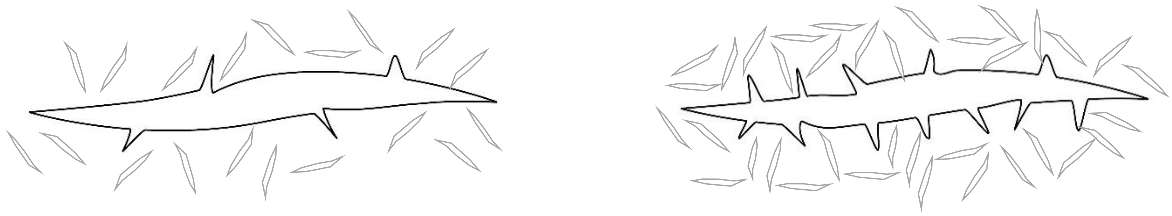
Table 4-1: Summary of the proposed model of the effect of the enzyme dosage in the pulp on its fibre properties.

Pulp	Number of fines	Extent of fibre fibrillation	Fibre width
Control	Low	Medium	High
100 g/t	Medium	High	High
200 g/t	High	Low	Low
500 g/t	Very high	Medium	Low



a: control pulp

b: 100 g/t



c: 200 g/t

d: 500 g/t

Figure 4-12: Proposed model of the extent of fibre fibrillation after refining of the control pulp (a), and pulps with enzyme dosages of 100 g/t (b), 200 g/t (c) and 500 g/t (d).

The model of the effect of an increasing enzyme dosage on fibre morphology was supported by the data. Increasing the enzyme dosage results in an increased number of fines (Figure 4-12, Figure 4-6), and corresponded with decreasing fibre width, as seen for the 200 and 500 g/t samples (Figure 4-10), as the fibre material was eroded from its surface (Sprey, 1990). There was an increase in the amount of fibrillation on the surface for the 100 g/t sample, but a loss of fibre fibrillation for the 200 g/t sample (Figure 4-8), which corresponded with an increased number of fines in the pulp, as fibrils were broken from the surface of the fibre. The increased amount of fibrils on the fibre surface from the 500 g/t could have resulted from secondary, extensive, fibrillation (Sprey, 1990).

4.4 Conclusion

Pulps with a higher enzyme dosage yielded handsheets with the highest moisture content after pressing, indicating a poor dewatering performance, and had the worst performance in conventional dewatering tests (freeness and WRV). The pressibility of handsheets was dependent on their formability, as the more water that remained in the sheet after forming, the more water that was removed. The pressibility measurement should not be considered for future work. The formability of handsheets did not reflect their total dewatering performance. It was possible that the low formability of the pulp treated with the enzyme at 200 g/t was due to the sealing of the sheet (Hubbe, 2007), as these fibres had the lowest amount of fibrillation.

Increasing the enzyme dosage caused an increase in the number of fines present in the pulp. As stated by the choke-point hypothesis (Hubbe, 2007), it was likely that the fines were primarily responsible for the decrease in dewatering performance of the pulps that were treated with the enzyme. It was likely that fines blocked drainage channels in the web of fibres and consequently restricted the dewatering of pulp and handsheets.

The extent of fibrillation on the fibre surface was highest for the pulp treated with the enzyme at 100 g/t and lowest for the 200 g/t pulp. The control pulp and pulp treated with the enzyme at 500 g/t had a similar extent of fibrillation to each other. A model was proposed whereby with increasing enzyme dosage in the pulp (to 100 g/t), the extent of degradation and consequent fibrillation of the fibre surface was increased. With enzyme treatment at a higher concentration (200 g/t), fibrillation was minimal as the amorphous cellulose was eroded further and broke off the fibre surface (Sprey, 1990). With an excessively high enzyme dosage, secondary fibrillation occurred, resulting in an higher fibrillation presence on the fibre surface. This model was supported by the fibre width data, which showed that pulps treated with 200 and 500 g/t of enzyme were narrower, possibly due to the erosion of the cellulose material of the fibre. The model was also supported by the increasing number of fines generated by the increasing enzyme dosage, as fibrils were removed from the fibre surface.

5 General conclusions

The freeness and Water Retention Value (WRV) of pilot-refined pulps were compared to the formability (water removed during forming) and pressibility (water removed during pressing) of handsheets made from the pulps in an effort to determine the best predictor of their dewatering performance. The formability of the handsheet was able to provide an indication of its moisture content after pressing. The conventional dewatering measurements, freeness and WRV, provided no correlation to the moisture content of the handsheet after pressing. Furthermore, the pressibility of the handsheets was determined to be dependent on the formability of the handsheet and provided no value in predicting its dewatering.

It was recommended that the formability of handsheets be used as a predictor of the dewatering performance of pulp on the paper machine, as it is a direct measure of the draining of a paper sheet. Neither freeness nor WRV measurements were designed to predict the dewatering of the paper sheet (TAPPI T227, 1999, Scallan & Carles, 1972). The freeness of the pulp was strongly correlated to its WRV, and it was recommended that only freeness is used in future work as it is a faster to perform and relatively inexpensive.

Pulps were also refined at pilot scale, and with an enzyme additive (endogucanase) at several dosages, in an effort to determine the effect of enzyme treatment on the morphology of the fibres and the consequent dewatering properties of the pulp. It was found that an increasing enzyme dosage resulted in the inhibition of dewatering of the handsheet, as its moisture content after pressing increased. Similarly, the freeness of the pulp decreased, and its WRV increased, with an increasing dosage of the enzyme. An increasing enzyme concentration in the pulp resulted in more fines present, and the number of fines was strongly correlated to the moisture content of the handsheets after pressing. As stated by the choke-point hypothesis (Hubbe. 2007), it was possible that fines blocked vital drainage channels in the wet web of pulp, inhibiting its dewatering. It was possible that the low formability of the pulp treated with the enzyme at 200 g/t was due to the sealing of the sheet (Hubbe, 2007), as these fibres had the lowest amount of fibrillation.

A model was proposed to relate the number of fines in the pulp, the extent of fibrillation on the fibre surface, and the fibre width to the structure of the fibre with an increasing enzyme dosage. The extent of fibrillation increased with the initial enzyme dosage, but fibrils were removed from the surface of the fibre at a higher dosage, likely due to the weakening of the amorphous cellulose constituting the fibre surface. Secondary fibrillation occurred with a further-increase in concentration of the enzyme in the pulp. The fibrillation data tied in with the fines and fibre width data, as more fines were generated with the removal of fibrils from the fibre surface, and fibre width decreased at high enzyme dosage, all indicating a loss of fibre material in the form of fines.

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7 Appendix

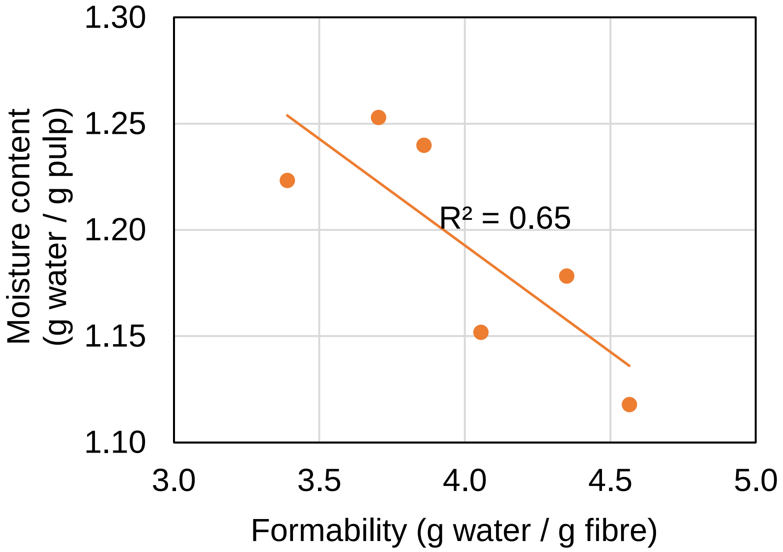

Figure 7-1: The influence of handsheet formability on its moisture content after pressing for a range of refining energies. Handsheets were pressed using a clamp press.

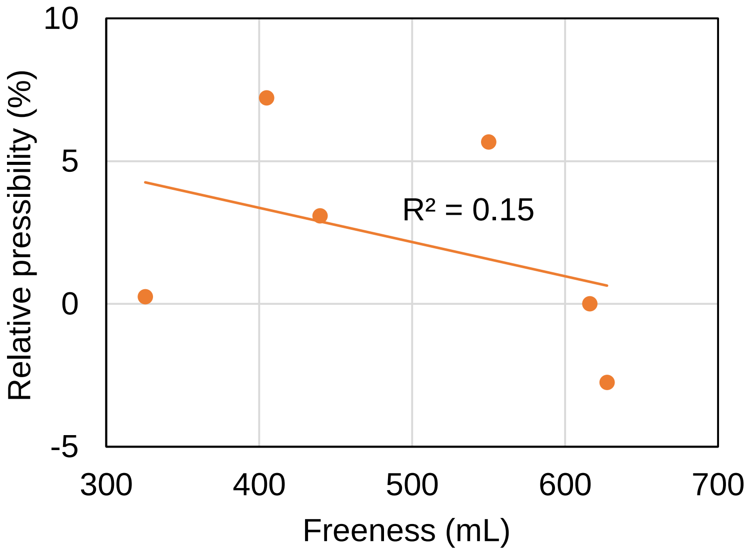

Figure 7-2: The influence of freeness on relative handsheet pressibility for handsheets pressed by the clamp press. Each pressibility is given relative to the pressibility of the handsheet formed from the unrefined pulp.

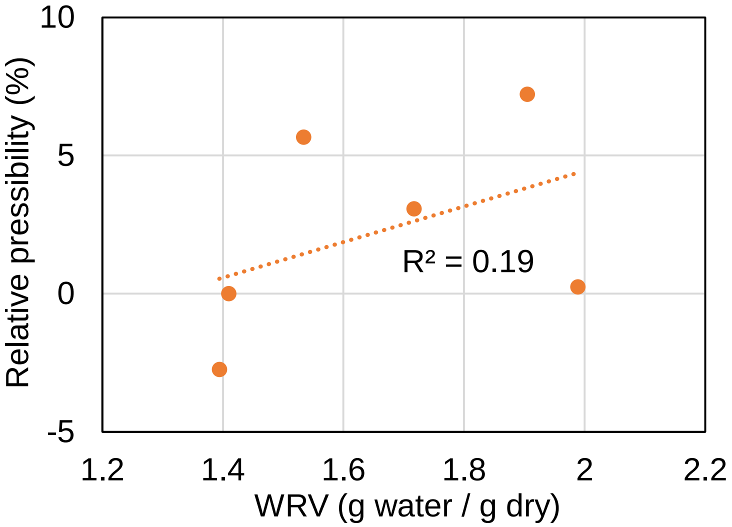

Figure 7-3: The influence of WRV on relative handsheet pressibility for handsheets pressed by the clamp press. Each pressibility is given relative to the pressibility of the handsheet formed from the unrefined pulp.

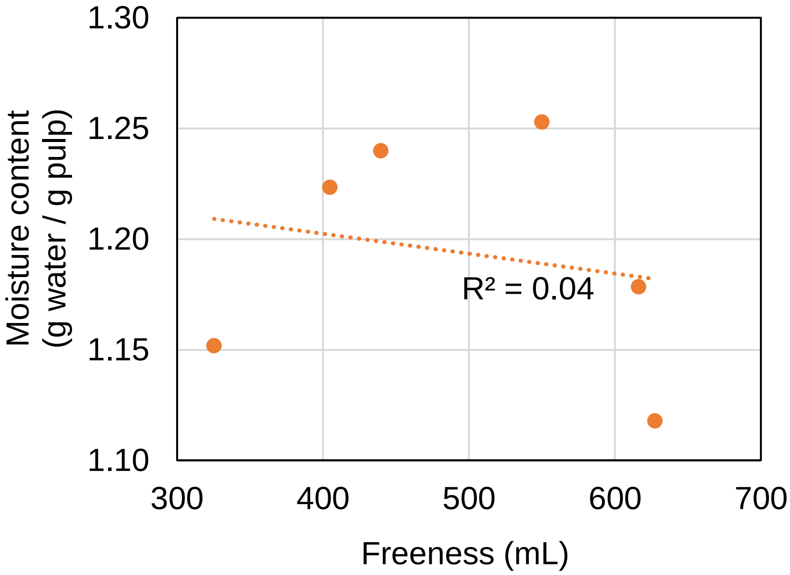

Figure 7-4: The influence of freeness on the moisture content of the handsheet after pressing with the clamp press for a range of refining energies.

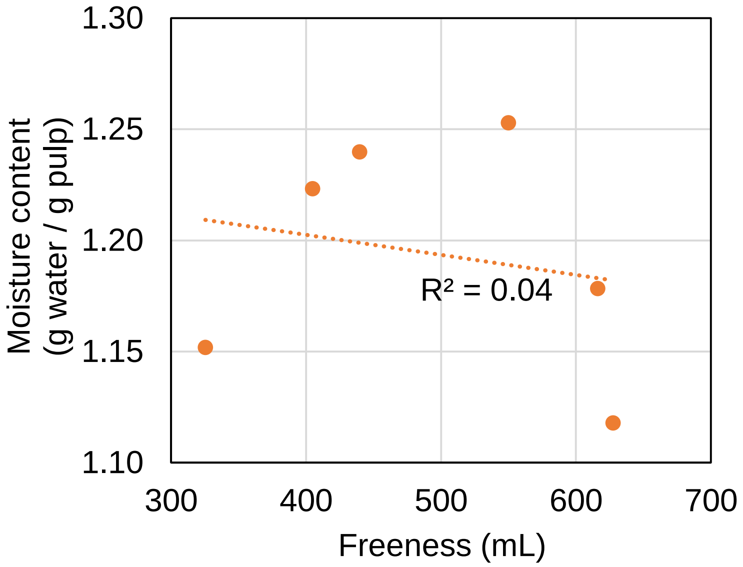

Figure 7-5: The influence of freeness on the moisture content of the handsheet after pressing with the clamp press for a range of refining energies.

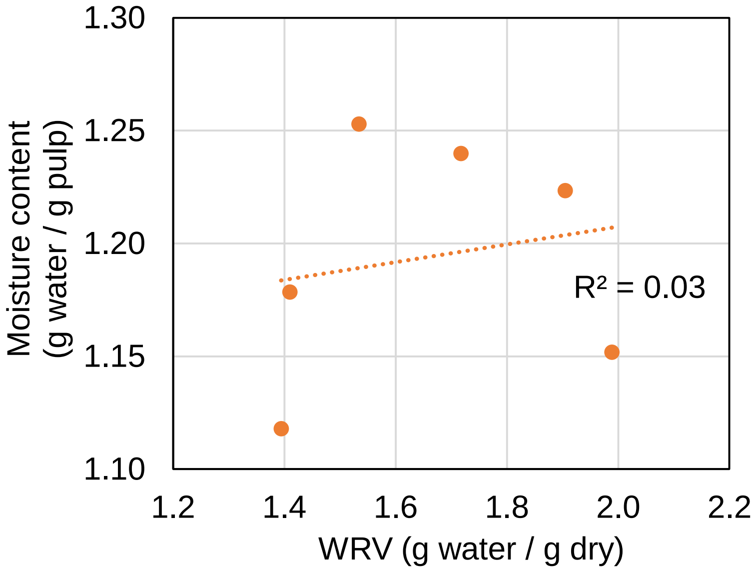

Figure 7-6: The influence of WRV on the moisture content of the handsheet after pressing with the clamp press for a range of refining energies.

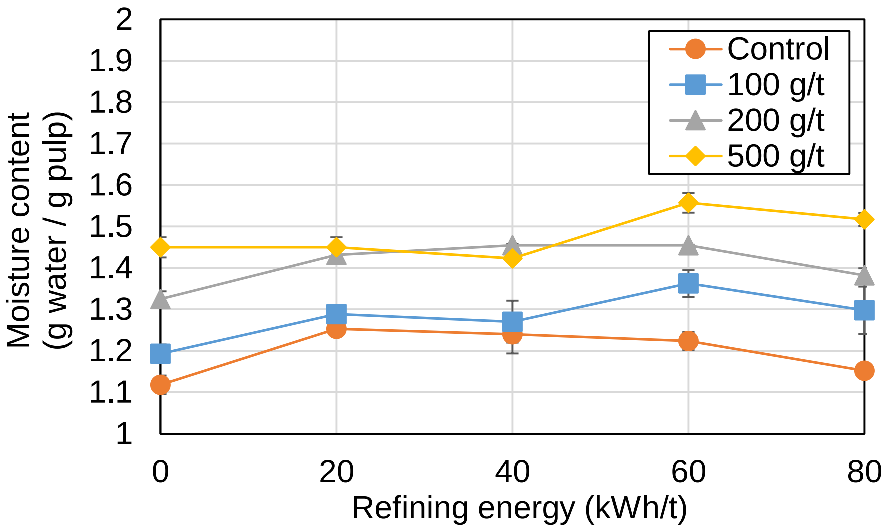

Figure 7-7: The influence of enzyme dosage on the moisture content of handsheets after pressing over a range of refining energies. Handsheets were pressed with a clamp press.

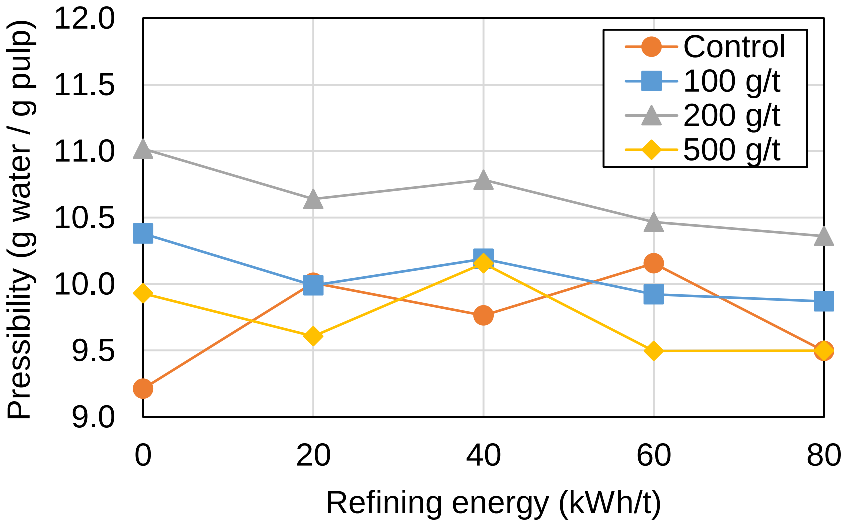

Figure 7-8: The influence of enzyme dosage on the pressibility of handsheets over a range of refining energies. Handsheets were pressed with a clamp press.

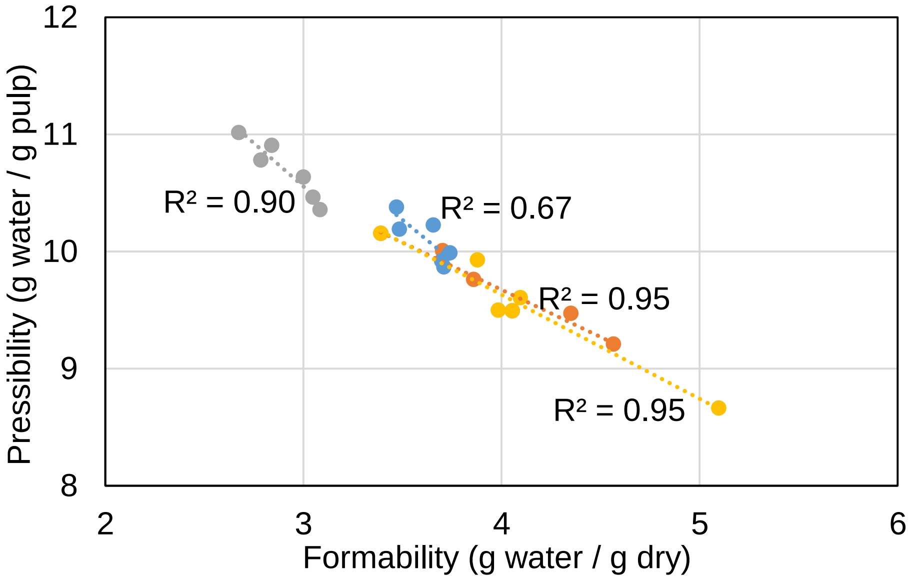

Figure 7-9: The correlation between the formability and pressibility of handsheets for three different enzyme-treated pulps as well as an untreated pulp. Handsheets were pressed with a clamp press.

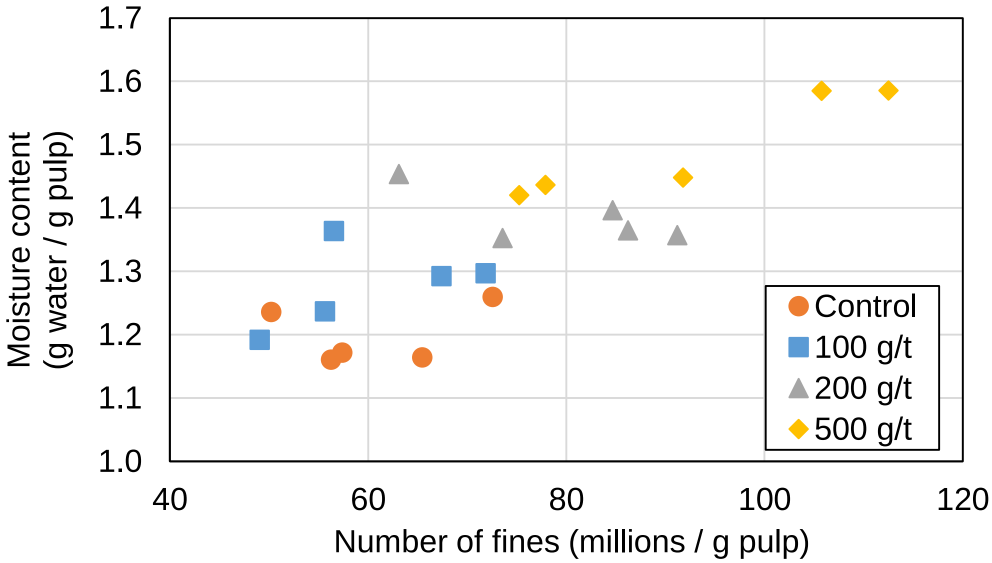

Figure 7-10: The influence of the number of fines in the pulp on the moisture content of the handsheets after pressing with the clamp press.

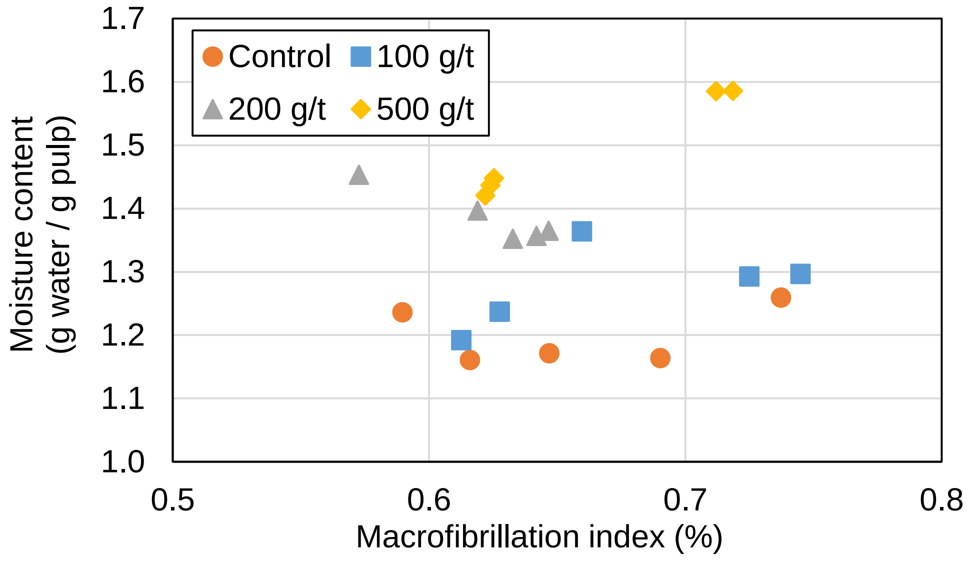

Figure 7-11: The influence of the extent of fibrillation of the fibres on the moisture content of the handsheets after pressing with the clamp press. Error bars are based on the standard deviation for each point.

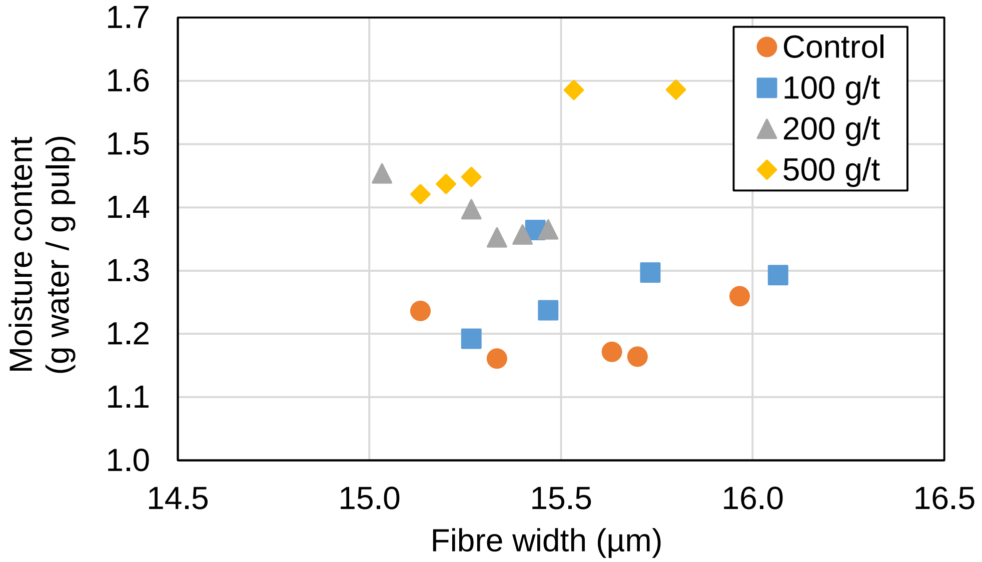

Figure 7-12: The influence of the width of the fibres in the pulp on the moisture content of the handsheets after pressing with the clamp press. Error bars are based on the standard deviation for each point.

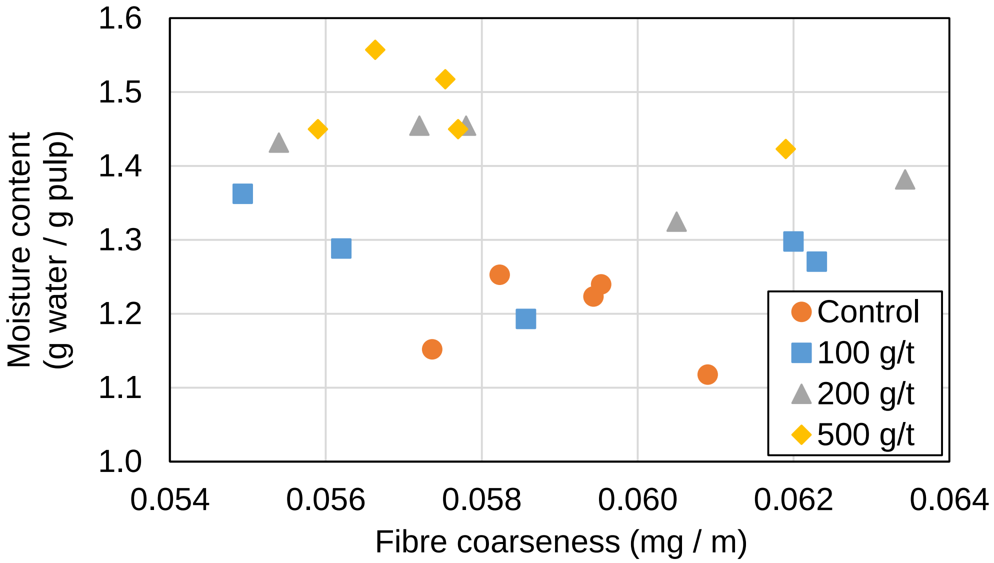

Figure 7-13: The influence of the coarseness of the fibres in the pulp on the moisture content of the handsheets after pressing with the nip press.

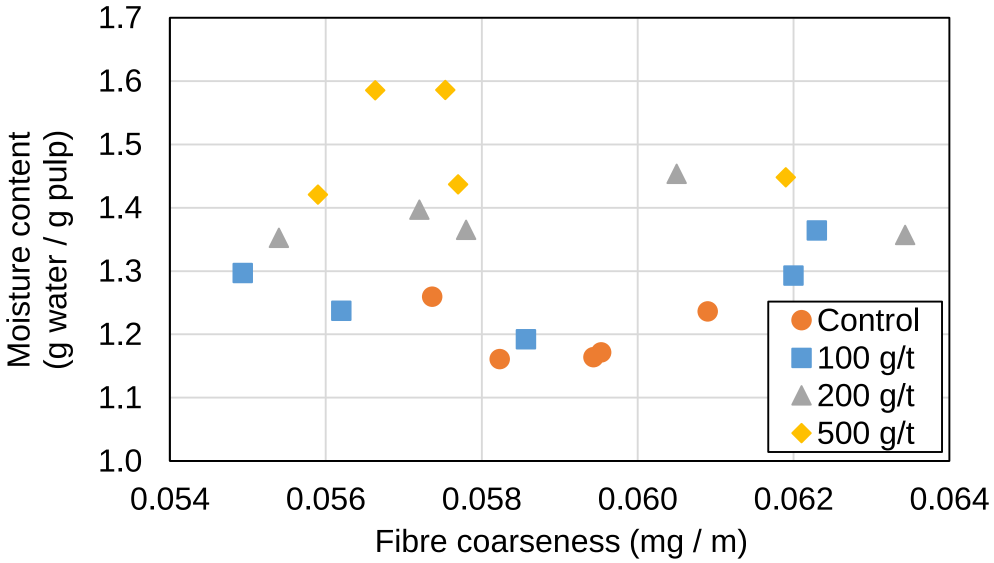

Figure 7-14: The influence of the coarseness of the fibres in the pulp on the moisture content of the handsheets after pressing with the clamp press.

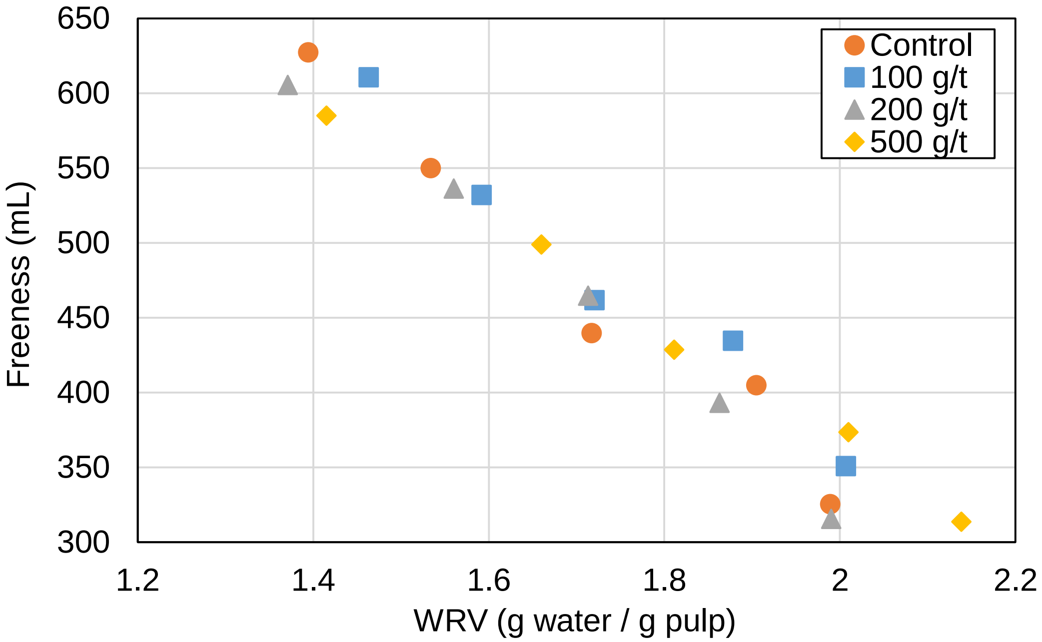

Figure 7-15: The relationship between freeness and WRV for pulps treated with different amounts of enzyme.