Czech University of Life Sciences Prague

Faculty of Forestry and Wood Sciences

Department of Wood Processing and Biomaterials

Master Thesis

Influence of cracks in wood chips on characteristics of pulp produced from those chips

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CZECH UNIVERSITY OF LIFE SCIENCES PRAGUE

Faculty of Forestry and Wood Sciences

DIPLOMA THESIS ASSIGNMENT

Benjamín Petržela

Wood Engineering

Thesis title

Influence of cracks in wood chips on characteristics of pulp produced from those chips

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Objectives of thesis

The first goal of this master's thesis is to develop a laboratory method for measuring cracks in wood chips. The second goal is to quantify cracks in wood chips produced by different conditions and estimate an influence of the cracks on characteristics of pulp produced from those wood chips.

Methodology

The thesis is conducted in cooperation with the Mondi company and is written in the English language.

The student will formulate theoretical starting points based on the study of literature and comparison of the results from previously published research.

During the experimental part of the work, the student will first develop a method for measuring cracks in wood chips. Furthermore, using the developed method, the student will quantify the cracks in the different variants of wood chips supplied by the Mondi company. Subsequently, the student will determine the effect of cracks in the chips on the selected characteristics of the pulp produced from these chips. The data for this comparison will be provided by Mondi company.

Time schedule:

May – October 2023: literature search

June – October 2023: methodology design, development of a laboratory method for measuring cracks

July – December 2023: quantification of cracks in chips produced by different conditions

December 2023 – March 2024: statistical data processing, discussion of results

April 2024: thesis submission

The proposed extent of the thesis

80 printed pages

Keywords

papermaking; residual lignin; microcracks; pulping liquor

Recommended information sources

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Dean

Prague on 21. 10. 2023

Declaration

I declare that I have worked on my master thesis titled "Influence of cracks in wood chips on characteristics of pulp produced from those chips" by myself and that I have used only the sources mentioned at the end of the thesis. As the author of the master thesis, I declare that the thesis does not violate the copyrights of any third party.

In Prague on the 5th of April 2024 _____

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I would like to express my gratitude to the supervisor of this master thesis Ing. et Ing. Štěpán Hýsek, Ph.D. for patience and mentoring. My gratitude also belongs to co-supervisor Dr. Johannes Leitner (Mondi AG), who initiated the idea of this research and provided practical knowledge, and to Ing. Jan Gojný, Ph.D. for his expertise in conduction of practical part of this research at University of Pardubice. Finally, I would like to humbly thank to my family and friends for endless support.

Vliv trhlin v dřevěných štěpkách na vlastnosti buničiny vyrobené z těchto štěpek

Abstrakt

Diplomová práce pojednává o vlivu trhlin ve dřevěných štěpkách a zkoumá vybrané vlastnosti buničiny vyrobené z těchto štěpek. Klíčovým aspektem této práce je vývoj reprodukovatelné laboratorní metody pro analýzu a kvantifikaci trhlin ve dřevní štěpce pro akademické i profesní využití. Závěrečná práce byla realizována ve spolupráci s firmou Mondi Group. V práci byly analyzovány dvě sady vzorků dřevních štěpek odebraných z výrobního závodu Mondi Štětí. První sada vzorků byla štěpkována ostrým nožem a druhá sada vzorků byla štěpkována nožem tupým, dle specifikovaných parametrů. Na těchto vzorcích byla následně vyvinuta obrazová analytická metoda pro kvantifikaci trhlin ve dřevních štěpkách. Dané vzorky byly zpracovány na buničinu procesem korespondujícím s výrobním procesem produkční linky Kamyr-line v Mondi Štětí. Vyrobená buničina byla nadále podrobena zkoumání vybraných vlastností jako jsou výtěž s hrubými neprovary, množství hrubých neprovarů, výtěž bez hrubých neprovarů, kappa číslo, obsah zbytkových alkálií a pevnost vláken při nulové vzdálenosti. Výsledky práce porovnávají dvě sady vzorků řezané tupým a ostrým nožem, které reprezentují vzorky s větším a menším množstvím trhlin. Tyto výsledky dokazují, že trhliny ve dřevních štěpkách mají vliv na vlastnosti buničiny vyrobené z těchto štěpek. Bylo dokázáno, že větší množství trhlin ve štěpkách může pozitivně ovlivnit proces výroby buničiny z hlediska efektivity a časové náročnosti procesu. Větší přítomnost trhlin u vzorku řezaného tupým nožem zapříčinila snížení požadovaného H-faktoru o -8,16 % při stejném cílovém kappa čísle. Výtěž s hrubými neprovary byla zvýšena o 1,50 % u vzorku s větší přítomností trhlin při stejném cílovém kappa čísle. Přílohou práce je vyvinutá laboratorní metoda pro obrazovou analýzu trhlin ve dřevní štěpce. Tato metoda byla použita pro stanovení podílu oblasti, kterou zaujímají trhliny ve štěpce s výsledným rozdílem 0,79 % většího podílu oblasti trhlin u vzorku řezaného tupým nožem, oproti vzorku řezaného nožem ostrým. Vyvinutá laboratorní metoda může sloužit jako podklad pro akademické i profesní účely zkoumání této problematiky.

Klíčová slova: papírenský průmysl; zbytkový lignin; trhliny; bílý louh

Influence of cracks in wood chips on characteristics of pulp produced from those chips

Abstract

The master's thesis deals with the influence of cracks in wood chips and examines selected characteristics of pulp produced from those chips. A key aspect of this paper is the development of a reproducible laboratory method for the analysis and quantification of cracks in wood chips for academic and professional use. The thesis was carried out in collaboration with Mondi Group. Two sets of wood chip samples taken from the Mondi Štětí production plant were analysed in the thesis, where the first set of samples was chipped with a sharp knife and the second set of samples was chipped with a dull knife. An image analysis method for quantifying cracks in wood chips was subsequently developed on these samples. The samples were processed into pulp using a process corresponding to the production process of the Kamyr-line production line at Mondi Štětí. The produced pulp was further subjected to the examination of selected characteristics such as unscreened yield, shive´s content, screened yield, kappa number, residual alkali, and Zero-span fibre strength. Results of the study compare two sets of samples cut with a dull and a sharp knife, representing samples with larger and smaller amounts of cracks presence. These results demonstrate that the cracks in wood chips influence the properties of pulp produced from these chips. It was proven that a larger number of cracks in the chips can positively affect the pulp production process in terms of efficiency and time consumption. A greater presence of cracks in the sample cut with a dull knife resulted in a decrease in H-factor by -8,16% at the same desired kappa number target. The unscreened yield was increased by 1,50% in the sample with a greater presence of cracks at the same target kappa number. An attachment to the thesis is the developed laboratory method for image analysis of cracks in wood chips. This method was used to determine the proportion of the chip crack area, resulting in a 0,79% greater proportion of crack area in the sample cut with a dull knife compared to the sample cut with a sharp knife. The developed laboratory method can serve as a basis for academic and professional purposes in researching this issue.

Keywords: papermaking; residual lignin; microcracks; pulping liquor

Content

1 Introduction

When introducing ecological solutions to our everyday live, one may think about renewable energy, reusing cups or innovative insulation materials. For me, it is paper and its irreplaceable role in details of everyday life.

Paper industry has made a huge progress in past decades, but could it go even further? Could paper solutions be produced using less material or less energy with the same output characteristics? Mondi, one of the market leaders in pulp and paper industry, has introduced project with a target of reducing 1 % of input material with conservation of same pulp output with same characteristics. While investigating potential approaches, chip, and its preparation process as well as its impact on final product were considered as one of important research topics.

Wood is a unique element – so organic, complex and inhomogeneous – that it is still not fully described on a chemical base. Wood, main input material for pulp and paper industry, begins its journey at huge chippers, where many parameters can influence the outcome. Some external as temperature or relative humidity and some internal or rather specified as knife sharpness, cutting angle, chipping speed etc. Those parameters have a significant impact on characteristics of chip produced as its dimension, surface processing or internal structure – especially internal cracks.

As small element as a crack in the wood chip may seem, the more important role it may play in cooking process of pulp. But does it really? Could more homogenous structure of chip with adequate number of cracks positively influence a white liquor penetration and thus making cooking process more efficient? And how could one identify or quantify these cracks?

Cracks in wood chips are a highway for white liquor ($NaOH + Na_2S$) for quick penetration of the chip. Wood chips with homogenous structure – cracks including – have fast and desired liquor uptake resulting in less overcooked and uncooked wood chips leading to less uncooked chips which must be recirculated and thus energy savings and yield increase. For investigation of chip cracks, further development of methods used needs to be done. Thus, this thesis is focused on development of such laboratory method based on image analysis of wood chip cracks and it´s comparation to selected pulp characteristics produces from same chips.

2 Aims of the study

Based on the hypothesis that more cracks and their homogenous distribution in wood chips have positive impact on efficiency of pulping process and subsequently for pulp yield, the first goal of this master thesis is to develop a laboratory method for quantification of cracks in wood chips. The second goal is to compare the laboratory method for chip quantification to selected characteristics of pulp produced by specified conditions and to estimate an influence of wood chip cracks on those characteristics of pulp produced from the same chips.

3 Literature review

Overall pulp production in Europe has increasing trend by 0,1 % with the baseline in year 1991 (Cepi, 2022), while overall world production is increasing faster with increase between years 2000 and 2022 by 15,5 % (Statista, 2024), resulting in growth by 0,73 % per year. The world production of pulp could be distinguished by different pulping processes. Chemical pulp covered 79 % of world production, while mechanical and semi-chemical pulp covered 15 % and other processes 6 % of world production in 2021 (Statista, 2024). European countries cover 20,3 % of global pulp production, while the most important region according to the pulp production is North America with 31,3 % of whole global production (Cepi, 2024). There are two main chemical pulp production processes – sulphite process and kraft (sulphate) process. The most widely used system nowadays is kraft process (Biermann, 1996) which covered approximately 75 % of European pulp production in 2022 according to Cepi (2022).

3.1 Company´s introduction

Mondi Group is a global leader in packaging and paper industry. Mondi employs around 21,000 people at approximately 100 production sites across 30 countries, with key operations located in Europe, North America, and Africa (Mondi Group, 2022). Mondi´s business structure permeates the value chain – from managing forests and producing pulp, paper and films, to developing and manufacturing effective industrial and consumer packaging solutions. Mondi produces a wide range of packaging and paper products, including corrugated packaging, flexible packaging, industrial bags, containerboard, pulp, and specialty papers (Mondi Group, 2022). The company is committed to sustainability and operates according to a sustainable development approach. The company focuses on reducing its environmental footprint (reflected by Smith, 1997), promoting responsible sourcing of raw materials, and developing innovative, sustainable products (Mondi Group, 2022). Additionally, Mondi invests in research and development to innovate its product offerings and processes continually (Mondi Group, 2022). It aims to provide solutions that meet customer needs while also addressing sustainability challenges. Company owners are confronting even socio-economic problems and global geopolitical affairs as e.g. the company has sold its assets in Russian Federation (Mondi plc, 2023).

Figure 1: Mondi Group logo

Source: https://cs.m.wikipedia.org/wiki/Soubor:Mondi_Logo.svg [cit. 2023-01-23]

Whole production process begins at pulp production sites where Mondi uses a kraft processes of manufacturing. Pulp could be both sold to costumers or additionally processed to final product which is a paper or various paper solutions. Main paper products are uncoated fine paper, lightweight paper-based packaging such as paper bags, functional paper and films which protect adhesive surfaces or provide protective barriers to papers for packaging and other applications as plastic-based flexible packaging solutions (Mondi Group, 2022). At the very beginning of a production is wood, which is FSC or PEFC certified and by other certificates related to region of production and processing assuring the origin of a material (Mondi Group, 2022).

3.2 Kraft process

Important variables during kraft pulping according to Biermann (1996) are:

1. Wood species and chip geometry.
2. Ratio of effective alkali to wood weight.
3. Concentration of effective alkali and liquor to wood.
4. Sulfidity.
5. H-factor (function of cooking time and temperature).

Despite the effort to create a uniform wood chip with specified parameters (wood species, chip thickness, chip geometry) variability in wood means that some of chips will be over-cooked and some of it will be undercooked (Sixta, 2006). Electron microscopy shows that over-thick chips have incomplete fibre liberation (kappa number of 94.7), whereas fine chips overcook (kappa number of 36.9), leading to lignin condensation and dark, rigid fibre clusters (Biermann, 1996). The penetration of liquors at pH above 13 in

wood is about the same in all three directions (Biermann, 1996; Hagiopol and Johnston, 2011). Decreasing variability in wood chips cooking procedure has potential to increase yield and efficiency of whole pulping process, regarding to Mondi research manager Dr. Johannes Leitner.

3.2.1 Pulping fundaments

The main active chemicals in the kraft process are hydroxide and hydrosulphide anions that are present in the kraft liquor, an aqueous solution of carbon disulphide CS_2 and sulphate sodium hydroxide NaOH and sodium sulphide Na_2S, referred to as white liquor (Rance, 1980; Sixta, 2006; Hagiopol and Johnston, 2011; Brännvall, 2017). The hydrosulphide ion plays an important role in kraft pulping by accelerating delignification and rendering nonselective soda cooking into a selective delignifying process (Hagiopol and Johnston, 2011).

H-factor

The H-factor is a pulping variable that combines cooking temperature and time into a single variable that indicates the extent of reaction (Biermann, 1996). Although different temperatures might be used, the degree of cook can be accurately estimated by H-factor calculation while other variables such as active alkali, sulfidity, and the liquor to wood ratio remain constant. There is no analogous variable for sulphite or soda pulping. The rate of delignification approximately doubles for an increase in reaction temperature of 8°C (Biermann, 1996). To obtain a bleachable kraft softwood pulp of about 5% lignin, one typically cooks for about 1,5 hours at 170°C, which corresponds to 0,75 hour at 178°C or 3 hours at 162°C (Biermann, 1996).

White liquor

White liquor is fresh pulping liquor for the kraft process, consisting of the active pulping substances NaOH (sodium hydroxide) and Na_2S (sodium sulphate), small amounts of Na_2CO_3 (sodium carbonate) left over from the recovery process, and oxidized sulphur compounds and other chemical impurities from wood, salt water, corrosion, etc. (Casey, 1960; Biermann, 1996; Bajpai, 2010).

Black liquor

Black liquor is the waste liquor from the kraft pulping process after pulping is completed (Ek, 2009; Bajpai, 2015). It contains most of the original cooking inorganic

elements and the degraded, dissolved wood substance. The latter include acetic acid, formic acid, saccharine acids, numerous other carboxylic acids (all as the sodium salts), dissolved hemicelluloses, methanol, and hundreds of other components. It is an extremely complex mixture (Ek, 2009; Hagiopol and Johnston, 2011). About 7 tons of black liquor at 15 % solids (about 10 % organic chemicals and 5 % inorganic chemicals with a total heat content of 13.5-14.5 MJ/kg solid) are produced per ton of pulp (Biermann, 1996). The viscosity rises rapidly with concentration above 50 %, with softwood black liquors being more viscous than hardwood black liquors (Biermann, 1996, Bajpai, 2015).

Green liquor

Green liquor is partially recovered form of kraft liquor. It is obtained after combustion of the black liquor in a recovery boiler (Bajpai, 2010). Green liquor is produced by dissolving the smelt from the recovery boiler (Na_2S, Na_2CO_3, and any impurities) in water (Hagiopol and Johnston, 2011). Further processing of the green liquor converts it to white liquor (Fengel and Wegener, 1989; Biermann, 1996).

Total chemical or total alkali (TA)

Total alkali is the sum of all sodium salts in the liquors (as Na_2O) that contribute to AA (i.e., NaOH or Na_2S) or are capable of being converted to AA in the kraft cycle (Sixta, 2006). Specifically, NaOH, Na_2S, Na_2CO_3, and $Na_2S_xO_y$ (as Na_2O) are included. All chemical amounts may be reported as concentrations of g/L or as a percent relative to oven-dry wood, regarding Biermann (1996). Following equation (3.1) represents computation of total alkali according to Biremann (1996).

$$TA = NaOH + Na_2S + Na_2CO_3 + Na_2S_xO_y \left[\frac{g}{L}\right] \tag{3.1}$$

Active Alkali (AA)

AA includes the active ingredients in the pulping process, i.e., NaOH + Na_2S, both as equivalent of Na_2O, expressed by equation (3.2) (Sixta, 2006). A typical value for AA is 100 g/L. Active alkali should be kept constant during cooking by adding alkali during the cooking process (Biermann, 1996).

$$AA = NaOH + Na_2S\ (as\ Na_2O)\ \left[\frac{g}{L}\right] \tag{3.2}$$

Effective alkali (EA)

EA are the ingredients that will produce alkali under pulping conditions. The effective alkali is about 12-18 % on wood to produce unbleached kraft and 18-24 % on wood for the production of bleached grades, with hardwoods using the lower amounts due to their lower lignin contents (Sixta, 2006). Biermann (1996) states that above 55 g/L EA, cellulose decomposition relative to lignin removal increases dramatically; consequently, a liquor to wood ratio above 3:1 should be used. Following equation (3.3) represents computation of effective alkali regarding to Sixta (2006).

$$EA = NaOH + \frac{1}{2} Na_2S \ (as\ Na_2O) \ [\frac{g}{L}] \tag{3.3}$$

Sulfidity

Sulfidity (of white liquor) is the ratio of Na_2S to the active alkali, expressed as percent, represented by equation (3.4) by Sixta (2006). Typically, a mill runs in the vicinity of 24-28 % sulfidity, depending largely on the wood species pulped. Sulfidity increases the rate of delignification which occurs by nucleophilic action of the hydrosulphide anion (HS^-) (Hagiopol and Johnston, 2011). The net effect is cleavage of β-aryl-ether linkages of lignin as well as the methoxy groups, the latter leading to the production of mercaptans (Hagiopol and Johnston, 2011). Sulfidity may protect against carbohydrate degradation directly, but the decreased cooking times also result in less carbohydrate degradation and all chemicals are expressed as Na_2O (Biermann, 1996).

$$Sulfidity = \frac{Na_2S}{NaOH + Na_2S} * 100\% \ [\%] \tag{3.4}$$

3.2.2 Chipping process

Chipping process begins with debarking. Barkers are used to strip bark from wood before chipping which is a crucial step. Bark contains minimal useful fibre, can darken pulp, requires extra chemicals, and introduces contaminants. Bark adhesion varies by season. Drum barkers, large rotating steel drums, remove bark by having logs abrade against each other (Sixta, 2006). While they are effective, they have high power and maintenance costs. Cambial shear barkers work with knives, but they need more supervision (Biermann, 1996). Cutterhead barkers, with toothed wheels, are suitable for difficult-to-debark logs but have higher wood losses and need supervision according to Biermann (1996). Hydraulic barkers use high-pressure water jets. In ring barkers, logs

move at speed while water at high pressure removes bark regarding to Biermann (1996). Understanding these methods is essential for wood processing. Other types of barkers are e.g. Bellingham barker or Flail debarkers (Biermann, 1996).

Chippers are used to turn wood into chips mechanically. Chipping requires around 7-14 kWh per ton (Biermann, 1996). Hardwoods are harder to chip, yielding fewer fine chips but more large ones compared to softwoods (Duffield timber, 2021). Traditional chippers for 1,5-3 m logs are gravity feed disk chippers. However, for tree-length wood, horizontal feed disk chippers are used (Biermann, 1996). They require more knives to avoid producing excessive fine and pin chips. Chips can be discharged via gravity, which increases costs due to elevation, or through blowing discharge, which can damage chips. Effective chipping of softwood logs should result in 85% acceptable chips, with 4% over-thick and 2% overlength chips, 7% pin chips, and 2% fines (Biermann, 1996). Certain chippers, like drum chipper and double cone (V-drum) chippers, are less suitable for smaller wood sizes according to Sixta (2006). They generate more pin chips (15-25%) and fines (5% or higher) due to their design. Other chippers, like veneer and core chippers, serve specific purposes in wood processing. In chip size classification vibratory, round-holed screens are used (Figure 2).

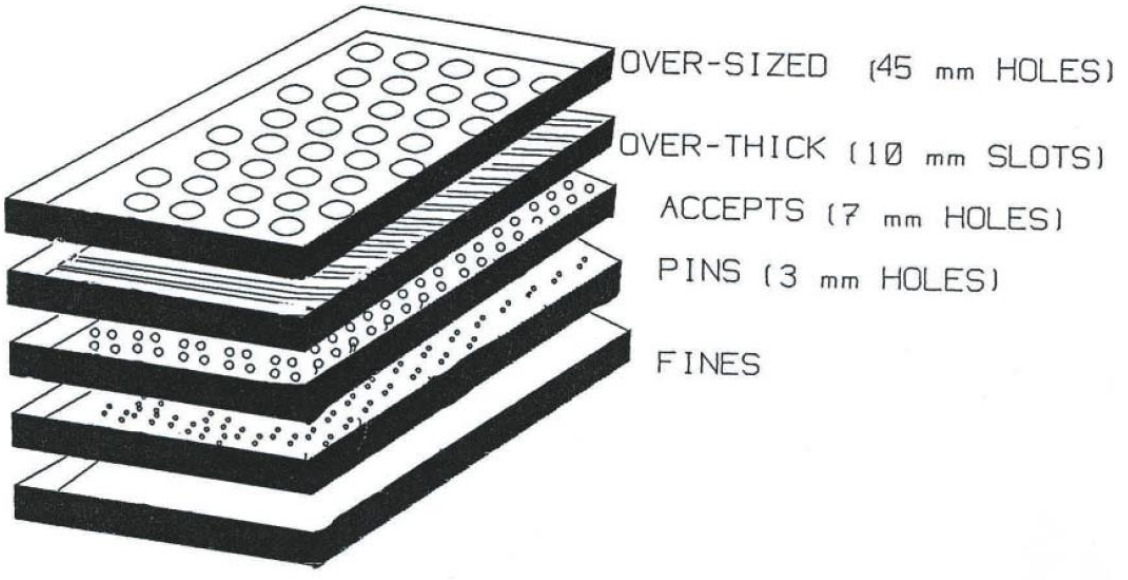


Figure 2: Vibratory, round-holed screens scheme

Source: Biermann (1996) [cit. 2023-10-21]

It has been concluded by Hellström (2010), that the friction between the chipping tool and wood is probably one crucial factor for the chip formation process. Three

different types of chip formation were identified in this study (Figure 3, Figure 4 and Figure 5). Hellström (2010) suggests that the stress field over the entire crack, i.e. not only the stress field close to the tip of the chipping tool, is critical for chip creation rather than just the latter. Finally, an analytical model was developed by Hellström´s study (2010) for predicting the normal and shear strain distribution in the crack prior to crack initiation and the analytical distributions were found to be in reasonable agreement with the corresponding distributions obtained from a FE analysis. Study (Hellström, 2010) assumes that the specific water adsorption was larger for chips produced at the spout angle 50°, as compared to chips produced at spout angles 30° and 40°, which implies that chipping at spout angle 50° will give a larger specific surface area than does chipping at lower spout angles. This was beneficial regarding the energy consumption during refining. It may be implied, that proper parameters may positively influence not only input material consumption in pulping process, but also energy consumption during pulp cooking.

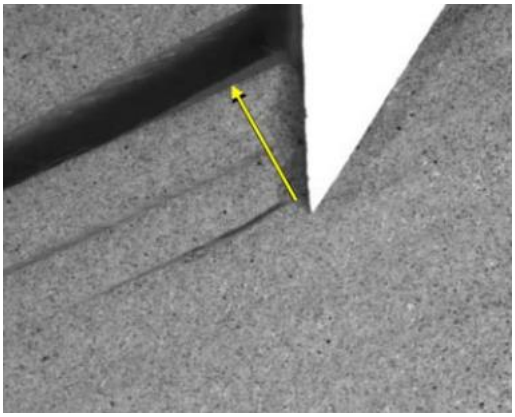


Figure 3: Opening mode

Source: Hellström (2010) [cit. 2024-02-17]

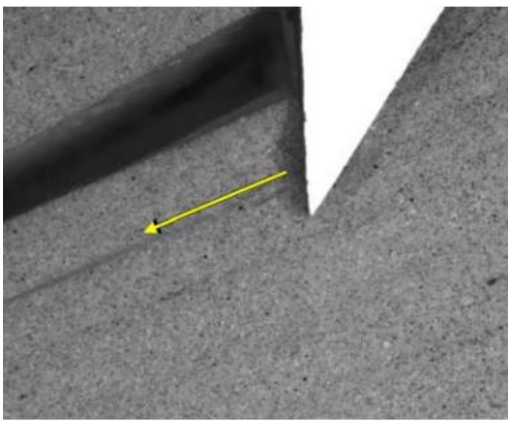


Figure 4: Forward shear mode

Source: Hellström (2010) [cit. 2024-02-17]

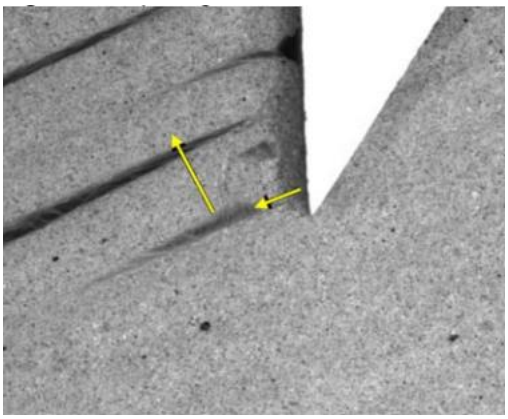


Figure 5: Remote opening mode

Source: Hellström (2010) [cit. 2024-02-17]

For further chip analysis miscellaneous analysis are used. TAPPI standards are essential for analysing wood samples, be it logs, chips, or sawdust. TAPPI Standard T 257 outlines the procedures for sampling and preparing wood for analysis. When it comes to assessing basic density and moisture content of pulpwood, TAPPI Standard T 258 comes into play. In this test, volume is determined through water displacement, while moisture content is calculated based on the change in mass before and after drying at 105°C ± 3°C. For evaluating the weight-volume of stacked roundwood, one should adhere to TAPPI Standard T 268. Moreover, TAPPI Standard T 265 is a valuable resource for measuring the natural dirt content in wood chips, which can originate from elements like outer and inner barks, knots, stains, and rot. Identifying dirt can be aided by colour photographs and involves ignition in a muffle furnace at 575°C ± 25°C, as detailed in TAPPI Standard T 211. Additionally, to determine the ash content, encompassing minerals, metals, anions, and silicates in wood and pulp, TAPPI Standard T 263 is indispensable. It provides comprehensive guidance, complete with diagrams and photomicrographs, for identifying wood and fibres from conifers.

3.2.3 Liquid penetration of chips

On a fibre level, the first criterion towards fibre liberation is for cooking chemicals to reach the middle lamellae. Cooking liquor introduced to wood chips initially penetrates through capillaries that have been revealed in the chipping process (Figure 6). In softwood, these are the lumen openings, and in hardwood these are mainly the vessel

elements (Brännvall, 2017) Moreover, ray cell channels make it possible for radial liquid penetration into wood chips (Malkov, 2002; Brännvall, 2017). Also, resin canals provide openings for longitudinal liquor flow.

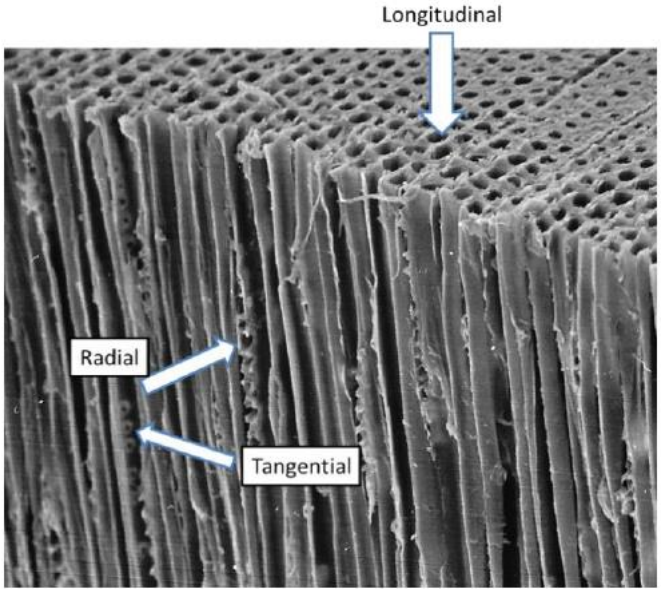
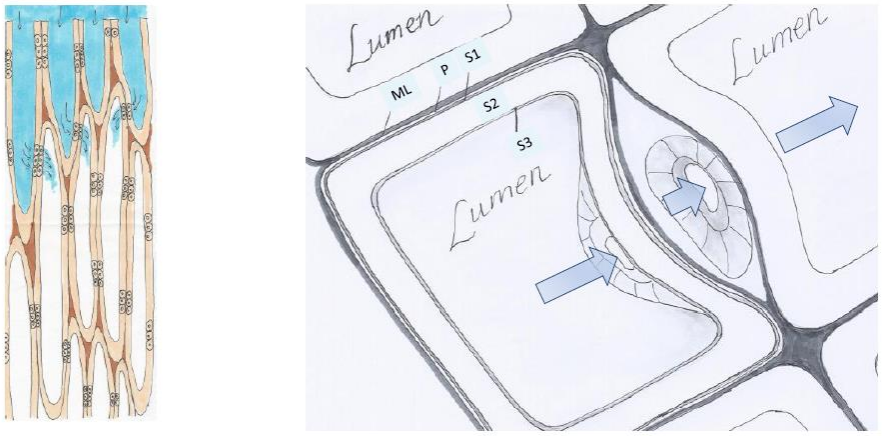


Figure 6: Softwood. Microscopic level. Directions of impregnation flow.

Source: Brännvall (2017) [cit. 2023-01-25]

The surface shows the lumen openings of the tracheids, and the tangential cut shows the lumen capillaries. Inside the lumen capillaries, the pith openings connect one tracheid with the adjacent tracheid. Ray cells seen between some fibres permit radial flow into the wood (Brännvall, 2017).

When the exposed capillaries have been filled, continued liquor flow in hardwood takes place from vessel elements to libriform cells through the scalariform perforation plates states Brännvall (2017). In softwood, liquid flow occurs through bordered pits in the softwood cell wall (Figure 7a). Bordered pits are arranged in pairs, such that one pit in the softwood fibre wall is connected to a pit opening in the adjacent fibre. As can be seen in Figure 7b, the passage through the pits brings the cooking chemicals in direct contact with the middle lamellae (Brännvall, 2017).



a) b)

Figure 7: Liquid penetration of softwoods via pit openings.

Source: Brännvall (2017) [cit. 2023-01-25]

Schematic Figure 7a shows a liquid flow through the open lumen at the chip surface followed by liquid entering the adjacent fibre through pit openings in the fibre wall (Brännvall, 2017). Where liquid flow (blue arrows) from one lumen cavity to the next lumen of the adjacent fibre through the bordered pits is represented by Figure 7b. With a shortcuts explanation: ML - middle lamellae, P-primary wall, S1-transition lamellae, S2-secondary wall, S3-tertiary wall. Drawings by the author. Figure 2b is inspired by Neagu et al. (2006).

When all empty capillaries have been filled, liquid penetration comes to an end and transport of cooking chemicals continues by diffusion (Brännvall, 2017). If the cooking liquor reaches a fibre with its lumen filled with chip moisture, transport of cooking chemicals will take place by diffusion. At a moisture content in fresh chips of around 50 %, approximately 45 % of the voids are filled with chip moisture (Brännvall, 2017).

In kraft cooking, the active chemical species are hydroxide and hydrogen sulphide ions, and the environment is highly alkaline, with pH levels ranging from 14 down to 11 according to Brännvall (2017). At these high pH levels, diffusion rates of chemicals are quite similar in longitudinal and radial directions, as alkali will swell the wood (Brännvall, 2017).

Deprotonation leads to electrostatic repulsion and swelling of the fibre wall and enlarges the paths for ionic movement (Brännvall, 2017). The hydroxide ions will split

off acetyl groups, and deacetylation is one of the main alkali-consuming reactions during kraft cooking (Sjöström et al. 1965). Acetyl groups can form strong hydrogen bonds with carboxyl groups, so deacetylation also increases the paths available for diffusion (Sjöström et al. 1965; Zanuttini et al. 1998; Inalbon and Zanuttini 2008; Montagna et al. 2013). For fibre liberation, it would be desired if the lignin located in the middle lamellae would be specially targeted for degradation reactions and dissolution. However, as pointed out earlier, cooking chemicals have a complicated path to travel before reaching the middle lamellae either by liquor flow through the pits or by diffusion through the fibre wall (Montagna et al., 2013).

Nowadays, researchers focus on modelling of delignification in kraft process on a chip level. This scale modelling enables to predict a delignification based on chip information. Studies by Bijok et al. was introduced in 2022 and 2023, where such model was introduced. Instead of describing the mass transfer as mathematically three-dimensional (Grénman et al., 2010, Liu et al., 2014) or using an average concentration profile over one dimension (Simão et al., 2008), the mass transfer is described by a one-dimensional finite volume discretisation along several volume compartments depicted in Figure 8 (Bijok et al, 2023).

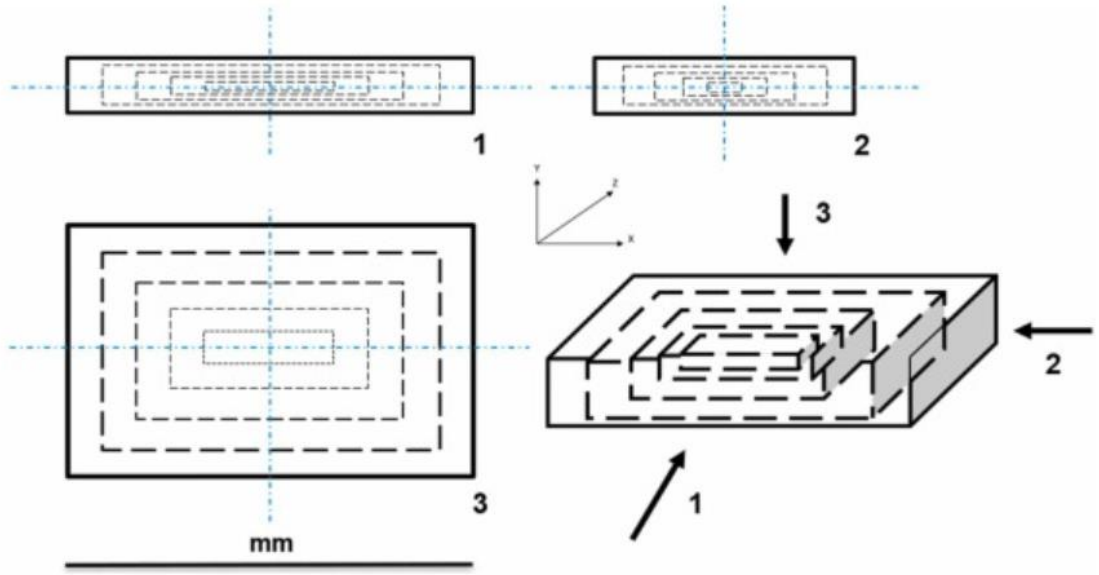


Figure 8: Schematic illustration of the wood chip discretisation into volume compartments discretised into equal-sized distances

Source: Bijok et al. (2023) [cit. 2024-02-18]

The model distinguishes between earlywood (low-density region) and latewood (high-density region) fibres with different initial fundamental chemical component concentration distributions (Bijok, 2023). No distinction between the different fibre layers has been made yet. The model operates under the assumption that the amount of fiber remains consistent during cooking, and that the wood chips are uniformly cut without any fissures or cracks typically caused by industrial chipping methods (Bijok, 2023). Unlike in previous model such Grénman´s et al. (2010), Liu´s et al. (2014) or Simão´s et al. (2008) the current model extended the view of the biomass feedstock by distinguishing the wood chips into early- and latewood regions with differences in their distribution and average values of the fundamental chemical component concentrations (Bijok, 2023) with a quite relatively high accuracy of prediction (Figure 9).

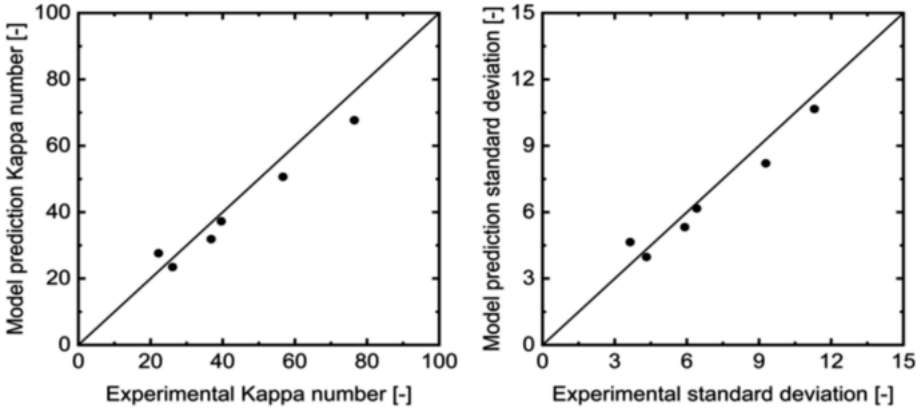


Figure 9: Model predictions against experimental data for the average kappa number (left) and the standard deviation (right) of the fibre kappa number distribution for uniform pulping conditions

Source: Bijok, 2022 [cit. 2024-02-18]

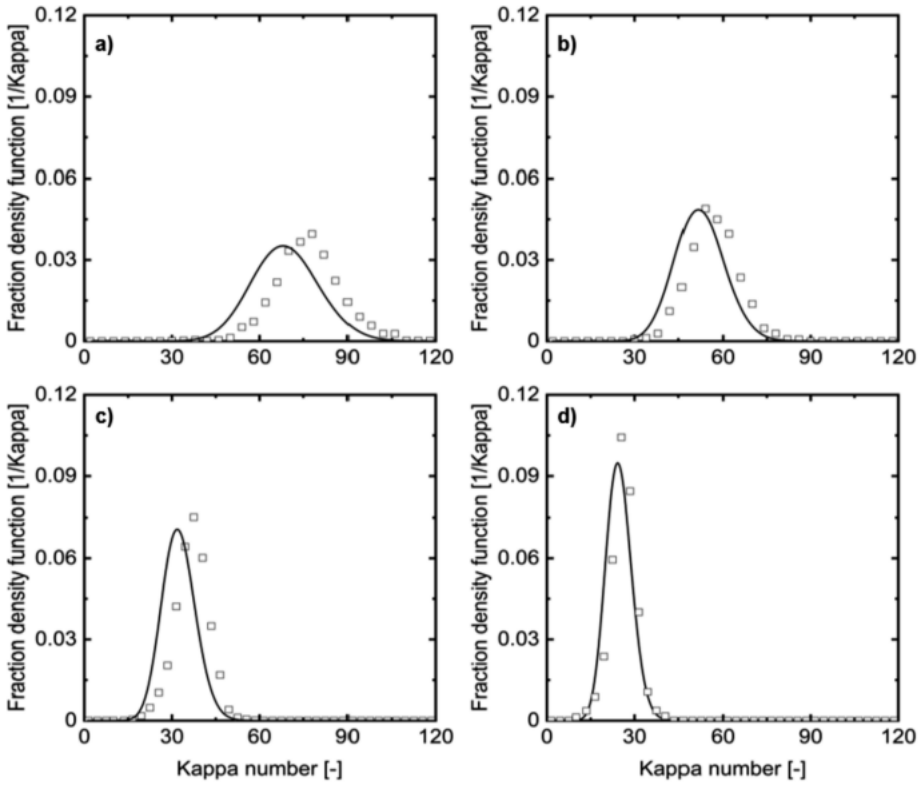


Figure 10: Model predicted (solid) and experimental (scatter) fibre kappa number distribution for the cooking experiments using initial effective alkali concentration

Source: Bijok, 2022 [cit. 2024-02-18]

3.2.4 Pre-treatment of chips

In softwood kraft pulping for bleachable paper grades, the pulp yield typically stands at around 47%, a figure that has remained constant over the years (Bolam, 1970; Kangas et al. 2014). Although softwoods contain approximately 70% polysaccharides, suggesting the potential for higher yields, the delignification process is non-selective, causing lignin and polysaccharides to dissolve simultaneously (Brännvall and Bäckström, 2016). Initially, during pulping, the alkali concentration drops sharply due to the splitting of acetyl groups, with resulting acetic acid being neutralized by alkali. This creates concentration gradients, with higher alkali concentrations at the interface between the liquor and the chip, gradually decreasing towards the chip's interior (Zanuttini et al. 1998). If the temperature is raised before the chips are uniformly impregnated, delignification begins on the surface of the chips while the centre lacks active cooking chemicals (Malkov et al., 2002). Consequently, higher residual lignin content in chip cores leads to the formation of shives, which must be removed, contributing to yield losses (Brännvall and Bäckström, 2016). Additionally, this process compromises the selectivity of

delignification on the surfaces, resulting in more dissolution of polysaccharides compared to the chip centre (Brännvall and Bäckström, 2016).

One of the pre-treatment methods used in Kraft pulping processes is an autohydrolysis. It is an environmentally friendly method for extracting hemicelluloses from wood. It involves dissolving a portion of hemicelluloses and lignin, reducing cellulose crystallinity, and increasing wood chip porosity (Mosier et al., 2005; Lu et al., 2012; Chen et al., 2017). These changes make subsequent pulping more efficient by improving chemical mass transfer and reducing energy consumption (Gorski et al., 2010; Liu et al., 2015). Recent study has shown that autohydrolysis affects fibre and wood morphology, leading to cellulose degradation, exposing less-ordered cellulose, and increasing the hydrophilicity of wood chips (Guo et al., 2021).

Autohydrolysis is used as a pre-treatment before kraft pulping process to extract hemicelluloses and produce pulp fibres according to Fišerová et al. (2014). Mild pre-hydrolysis conditions are preferred as they make wood chips more suitable for pulping and bleaching (Fišerová et al., 2014). Under milder conditions, carbohydrates and acid-soluble lignin are removed, reducing the time needed to reach the desired Kappa number in the final pulp (Chen et al., 2017; Guo et al., 2021). Severe pre-hydrolysis conditions can lead to lignin crosslinking and lignin agglomeration, which hinders pulping and results in longer processing times (Chen et al., 2022)

Chen et al. (2022) revealed that pre-hydrolysis significantly reduces the H-factor needed to reach a specific Kappa number by removing hemicelluloses and enhancing delignification. It has been found that pre-hydrolysis pulping preserved fibre properties, causing only a limited reduction in fibre dimensions and an increase in kinks. Wood chip thickness plays a crucial role in pre-hydrolysis, delignification, and pulping. Chen et al. (2022) also investigated the impact of chip thickness on pre-hydrolysis and kraft pulping. Chips under 2 mm thickness facilitated hemicelluloses oligomer removal, while chips with thickness between 2 and 6 mm achieved adequate delignification and desired fibre properties. However, chips thicker than 6 mm were not sufficiently delignified. The research (Chen et al., 2022) highlights the potential benefits of integrating mild pre-hydrolysis into kraft pulping processes and underscores the importance of chip thickness in optimizing the results. Muzamal et al. (2017) found that steam explosion pretreatment results in the formation of microcracks in the cell walls of wood.

3.2.5 Cooking process

The main objective of cooking stage is to liberate the fibres in the biomass with special focus on delignification because lignin is unwanted substance in final product – pulp, which should consist mostly of cellulose (Rance, 1980). Chemical degrading is needed to dissolve components in the middle lamellae, on account of the content of lignin in middle lamellae is approximately 50 % reaching as much as 90 % in the intersection between four tracheids, while the secondary wall has a lignin concentration of approximately 25 % (Fengel and Wegener, 1984; Brännvall, 2017). Via pulping process is necessary to remove as much as 80 % of lignin originally present in wood for required characteristics of final pulp (Rance, 1980). In process is to be distinguished between delignification on macroscopic level (wood chips) and microscopic level (wood fibres) (Brännvall, 2017).

The main active chemicals in the kraft process are hydroxide and hydrosulphide anions that are present in the kraft solution, an aqueous solution of carbon disulphide CS_2 and sulphate sodium hydroxide NaOH and sodium sulphide Na_2S, referred to as white liquor (Biermann, 1996; Sixta, 2006; Brännvall, 2017). The hydrosulphide ion plays an important role in kraft pulping by accelerating delignification and rendering nonselective soda cooking into a selective delignifying process (Rance, 1980). Delignification can be divided into three phases, namely the initial, bulk, and residual phases (Sixta, 2006). In the initial phase, delignification is caused by the cleavage of α-aryl and β-aryl ether bonds in the phenolic units of lignin which accounts for approximately 15–25 % of native lignin (Hagiopol and Johnston, 2011). In this stage, the predominant part of the total carbohydrate losses can be observed. In the bulk delignification phase the main part of the lignin is removed while at the same time only minor carbohydrate losses occur. The cleavage of β-aryl bonds in nonphenolic units of lignin is assumed to be the main delignification reaction (Hagiopol and Johnston, 2011). In the residual delignification phase, only approximately 10–15 % of the native lignin is removed (Hagiopol and Johnston, 2011). However, with continuous delignification, the dissolution of carbohydrates extensively increases. In order to maintain high yields and to preserve a sufficiently high quality of the pulp, delignification is limited to a certain degree of delignification, targeting kappa numbers of about 25–30 for softwood kraft pulps (Sixta, 2006).

Main chemicals in kraft process, also known as sulphate process (Sixta, 2006), are sodium hydroxide NaOH and sodium sulphate Na_2S. Combined with water they create a white liquor which is in conditions of high temperatures and low-pressure impregnating wood chips and thus breaking the bonds linking lignin hemicelluloses and cellulose.

3.2.6 Recovery process

As the kraft cooking process in closed loop, recovering of the cooking chemicals plays important role. Chemical, which are being recovered from black liquor are sodium hydroxide (NaOH) and sodium sulphide (Na_2S) for reuse in subsequent cooks (Bajpai, 2010). First step is black liquor evaporation which comes right after delignification of wood chips in the digester (Sixta, 2006; Bajpai, 2010). Multiple-effect evaporators progressively concentrate the black liquor by utilizing steam to remove water. This reduces the volume of the liquor and increases its solids content, making it more energy-efficient for subsequent processing stages (Smook, 2012). In some mills, the concentrated black liquor undergoes oxidation with air or oxygen-enriched air. This step promotes the conversion of odorous sulphur compounds (mainly methanethiol) into less odorous disulfides, reducing emissions and improving air quality (Gierer, 1985). The concentrated black liquor is then mixed with recovered chemicals from later stages and sprayed into a recovery furnace (Bajpai, 2010). High temperatures (around 850°C) in the furnace cause combustion of the organic materials in the black liquor, generating heat and releasing inorganic chemicals as flue gas (Sixta, 2006). The molten inorganic fraction from the furnace, primarily a mixture of sodium carbonate (Na_2CO_3) and sodium sulphide (Na_2S), is known as smelt. The smelt is dissolved in weak white liquor to form green liquor, a solution containing sodium sulphide and sodium carbonate (Sixta, 2006). Green liquor is causticized with slaked lime $Ca(OH)_2$ in a causticizing plant. This reaction converts most of the sodium carbonate in green liquor back to sodium hydroxide (NaOH). The insoluble calcium carbonate precipitate is separated from the causticized liquor (white liquor) through a series of settling and washing tanks. The final product, clarified white liquor containing NaOH and Na_2S, is then sent back to the digester for another round of pulping, effectively completing the closed-loop recovery process (Sixta, 2006).

3.3 Wood chip cracks

In the past, several studies have been conducted to investigate the process of liquid penetration into wood (i.e. Malkov, 2002). Researchers have been able to obtain direct

and indirect quantitative information on the amount of liquid penetrated and the rate of penetration, as well as qualitative information about the modes of liquid penetration into wood resulting in several concepts and theories related to transport of liquid in wood (Malkov, 2002). These findings help to understand some of the phenomena that take place during the flow of liquid into the wood matrix, the factors affecting the penetration of the liquid and its permeation into the wood. However, many aspects remain unclear and further research is needed to achieve a better understanding of the liquid penetration process. The need for new reliable and accurate methods capable of providing direct and continuous data on the process of liquid penetration also must be emphasised (Malkov, 2002).

While in general cracks and their detection and quantification is being researched in many studies (Hjortsberg et al., 2013; Cho et al., 2018; Li et al., 2023), wood chips and their cracks brings new challenges. For example, study by Li et al. (2023) proposes a new method for detecting cracks in materials using a combination of two techniques: GM (Grey Model) and ResNet (Residual Neural Network). The authors believe this method is more intelligent than previous methods. Their Grey Model (GM) component is likely a time series forecasting model, while the ResNet is a type of deep neural network architecture (Li et al., 2023).

As for the wood chip cracks, the need for the new methodologies for measuring, analysis, quantification and evaluation has upraised in wood processing companies. If believed, that cracks in the wood chips may influence potentially yield, it is important for such methodologies to be developed. Thus, in collaboration with Mondi, it is aim of this thesis to develop laboratory method for such purpose and compare its results to pulp yield to ensure its relevancy. Two different types of wood chip cracks are distinguished in this thesis – macrocracks and microcracks. Macrocracks stands for cracks which are visible by eye – defined and analysed under optical microscope. Microcracks are cracks on the cell level of wood anatomy which are to be observed under the electron microscope (Muzamal et al., 2017) or indirectly described by other crack evaluation methods regarding to impregnation theory.

3.3.1 Over-thick chip phenomenon

While many studies historically have been done on cooking processes of wood chips, which was the main focus in development of modern pulping machines and

procedures, an attention of scientist is now turning to input material – chips. Their consistency plays a crucial role. Wood chips produced vary in size, which affects how well they can be turned into pulp. Rajesh et al. (2010) studied how thicker chips affects different parts of the process, not just making pulp but also things like losing chemicals and how clean the water is when it's released. They have found, that surprisingly, better quality chips seem to have positive effects on different parts of the process when tested on a real scale. Table 1 show the results from the study (Rajesh et al., 2010), removal of over thick from whole chips results in screened pulp yield increase by 2%, with lower unbleached kappa number and higher unbleached pulp brightness.

Sl no	Parameters	Unit	Whole chips	Overthick +8mm	Accepts +7mm	Pin chips +3mm
1	Chemicals	%	17	17	17	17
2	Total yield	%	44.0	45.1	44.6	43.0
3	**Sc rejects**	**%**	**1.9**	**4.0**	**0.5**	**0.1**
4	**Screened Yield**	**%**	**42.1**	**41.1**	**44.1**	**42.9**
5	Kappa number		22.4	25.5	21.7	21.0
6	Brightness	% ISO	27.5	28.6	30.8	30.2

Constant pulping conditions: Cooking Temperature 164°C
Cooking Time 90 min
H factor 1000

Table 1: Pulping of chip fractions Euca hybrid

Source: Rajesh et al., 2010 [cit. 2024-02-17]

Study by Rajesh et al. (2010) has also defined the influence of over thick chips of Active alkali (Figure 11), kappa number and Total dissolved solids.

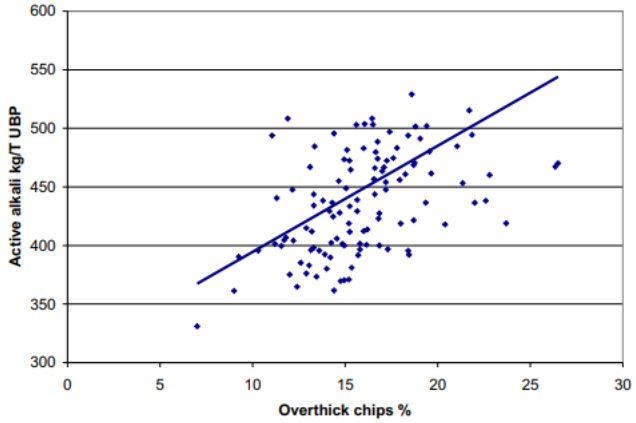


Figure 11: Impact of overthick chips of Active alkali

Source: Rajesh et al., 2010 [cit. 2024-02-17]

Rajesh et al. (2010) concludes that the over-thick chips, if ignored, result in higher rejects percentage and lower yield. They warrant usage of higher active alkali to control

the rejects which to reduced pollution load and thereby better environment (the environmental impacts of the paper industry are significantly emphasized by Smith (1997) and the individual aspects are thoroughly analysed in his book).

The underlying idea of what is behind the over-thick chips effect may cohere with chip structure and liquid penetration of wood chips, where wood cracks might play a significant role.

3.3.2 Wood chip cracks analysis methodologies

As many studies are focused on cracks and their analysis in different materials, only few studies are concentrated on different approaches in microscopical analysis of wood. On of the studies used in the creation of methodology used was “Ultra-high-resolution reflected-light imaging for dendrochronology” by Rydval et al. (2024). The study discusses the use of ultra-high-resolution imaging techniques for dendrochronology. The process involves developing large mosaic images of samples through automated stitching and focus optimization. Images are then processed to create binary representations of anatomical structures, allowing for the measurement of Latewood Surface Intensity (LWSI) (Rydval et al., 2024). The study compares Latewood Blue Intensity (LWBI) and LWSI, highlighting the advantages of the latter, such as colour-free parameters and higher resolution (Rydval et al., 2024). The technique offers a more accurate representation of narrow rings and requires fewer samples to develop robust chronologies. Overall, the document emphasizes the benefits of using ultra-high-resolution images for dendrochronological research, providing a detailed insight into the methodology and results of the study. Some of these findings were also utilized in the development of the laboratory method used. As the cornerstone of methodology was used idea of creating contrast between anatomical structures reflected on 2D surfaces with zero depth and surfaces with larger depth (lumens, cracks, etc.). For reaching this contrast, black base colour was applied on prepared surface of wood chip and afterwards white chalk was used to fill surface depths. Wood cracks are usually longer element then lumen, thus using correct length filter it was intended to develop automatic image analysis method.

4 Materials and methods

In practical part of this thesis, two different methodologies were used for testing same material, thus those methodologies are described in detail below. The first method was image analysis of wood chip surfaces in laboratory conditions used as direct wood chip cracks measurement with outcome focused on development of such laboratory method, the second one stands for indirect measurement of cracks impact on pulp produced from those cracks. Afterwards, results obtained from different methodologies were compared to declare efficiency of developed laboratory method.

4.1 Materials

For initial testing a different type of chips regarding to different chipping conditions were used. Chips from Mondi Štětí plant were used for conducting experiments. Chips were processed by gravity feed disk chipper and transported directly from the mill to the lab, where they were dried out at laboratory conditions (t = 20 ± 0,5°C; RH ~ 65 %) for one week. Two main types of raw chips from Mondi Štětí, sieve 3 (common accept) were examined. Table 2 and Table 3describe main characteristics of wood chip samples. The description is followed by Chart 1 and Chart 2 depicting share of individual fractions of samples.

Figure 12: Materials, sampling

Source: own [cit. 2023-09-20]

Sample 1

Origin:	Mondi Štětí
Date of extraction:	18.09.2023
Wood type:	softwood
Sieve:	3

Knife:	sharp
Knife monitoring:	8,0%
Running time:	2:57:42
Chipping time:	2:06:47
Effective:	71,0%
Time left:	35:20:28
Knife reset:	5

Sieve	**mass [g]**
Sieve 1	118,5
Sieve 2	263,4
Sieve 3	3113,8
Sieve 4	545,1
Sieve 5	50,4
Sieve 6	12,5
Total	4103,7

Table 2: Sample 1

Source: Mondi Štětí a.s.; edited [cit. 2023-09-20]

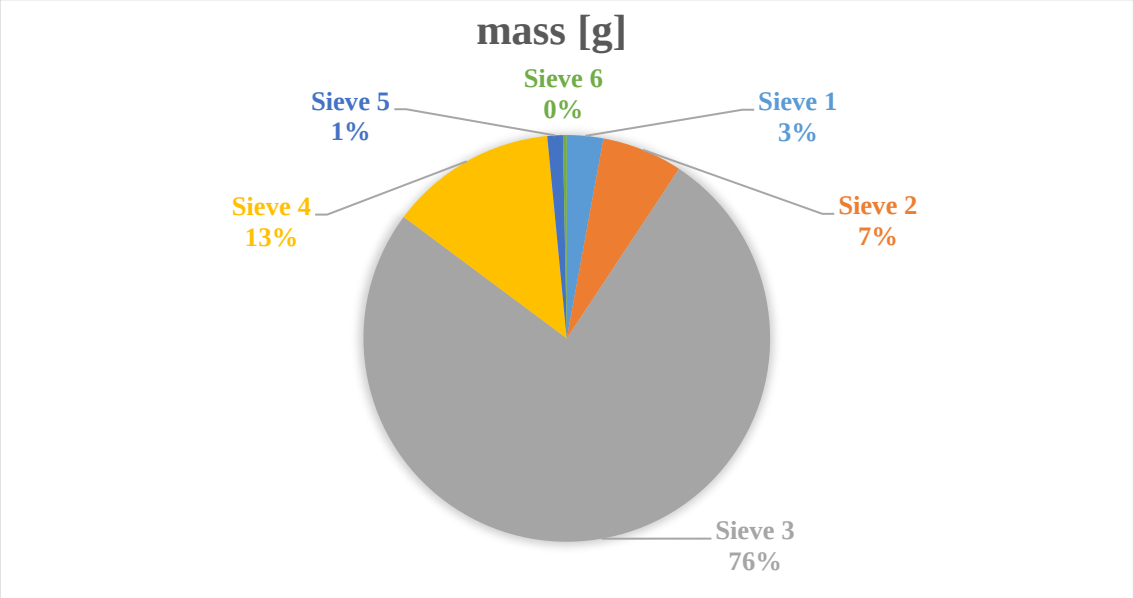


Chart 1: Fraction analysis; sharp knife sample

Source: own [cit. 2023-09-20]

Sample 2

Origin:	Mondi Štětí
Date of extraction:	19.09.2023
Wood type:	softwood
Sieve:	3

Knife:	dull
Knife monitoring:	64,0%
Running time:	3:50:13
Chipping time:	20:07:13
Effective:	72,3%
Time left:	16:54:51
Knife reset:	8

Sieve	**mass [g]**
Sieve 1	129,2
Sieve 2	288,7
Sieve 3	2777,9
Sieve 4	595,3
Sieve 5	281,9
Sieve 6	47,3
Total	4120,3

Table 3: Sample 2

Source: Mondi Štětí a.s., edited [cit. 2023-09-20]

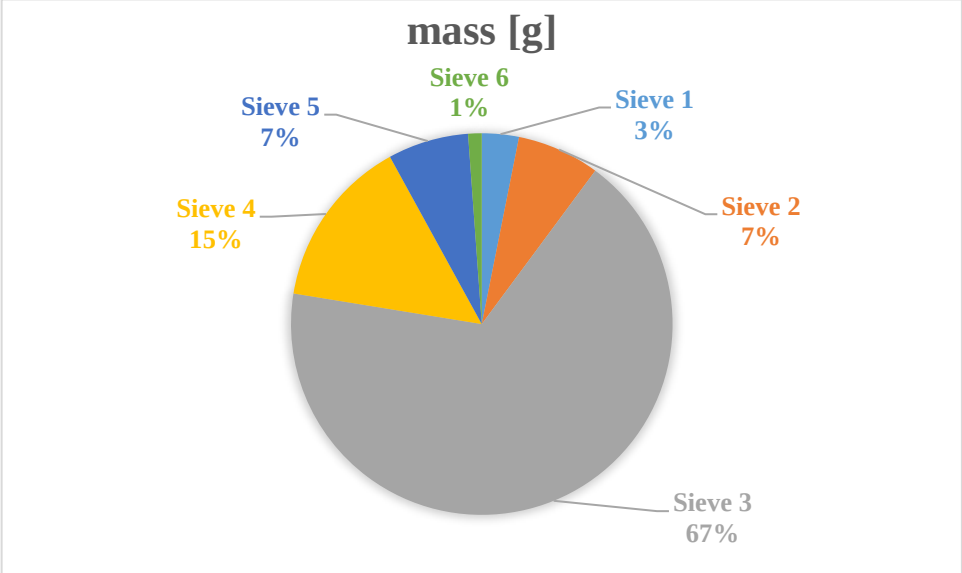


Table 4: Fraction analysis; dull knife sample

Source: own [cit. 2023-09-20]

Complete conditions of knifes during sample´s extraction and before were taken from knife monitoring software and are introduced in Table 5.

Date:	18.09.2023	18.09.2023	18.09.2023	19.09.2023
Description:	Before change	After change	After 2h chipping	After 20h chipping
Sample	-	-	1st sample	2nd sample
State:	stopped	stopped	chipping	chipping
Instant Hit:	okay	okay	okay	okay
Hit points:	okay	okay	okay	okay
Wear points:	alarm	okay	okay	okay
Knife monitoring:	91,0%	0,0%	8,0%	64,0%
Running time:	43:56:12	0:00:00	2:57:42	27:50:13
Chipping time:	35:38:32	0:00:00	2:06:47	20:07:36
Effective:	81,1%	0,0%	71,0%	72,3%
Time left:	15:13:50	0:00:00	35:20:28	16:54:51
Knife reset:	2	2	5	8

Table 5: Knife cutting conditions

Source: Mondi Štětí a.s., edited [cit. 2023-09-20]

4.2 Image analysis methodology

For a wood chip crack analysis, a chalking method with image analysis was developed. The method is a direct characterisation of a wood chip cracks of an individual chips. From chip fractions available, only chips from sieve 3 (common accept chips) were chosen for examination, as this fraction represents a majority of chips sorted and has ideal dimensions for testing via this methodology. Chalking method with image analysis follows methodical steps described below.

Chip selection and preparation

Chips form sorting sieve nr. 3 (common chip accept fraction) were chosen for testing. Firstly, chips were soaked in distilled water for 24 hours for reaching fibre saturation point. The soaking precedes microtone cutting for reaching smooth surface while cutting which has 2 effects. Ad 1 it does not create additional cracks which would depreciate gained results, ad 2 when having smooth cut image analysis is to have more precise results when evaluating lines and curves.

Microtone cutting

After soaking, chip was cut on linear microtome (WSL Serie nr.8078 Schenkung Dapples) for creating flat surface as a prerequisite for image taking of chip surface. Chip

was cut approx. in the middle and perpendicularly to the wood elements (fibres, tracheids…). The hight – distance from the bottom to the cut surface – of a cut chip could not exceed approx. 1 cm due to used microscope limitations (while having a bigger height microscope could not be maximally focused on evaluated surface).

Drying

For further procedure chips were dried out in climatization chamber at a temperature of 20°C and relative humidity of 65 % for 24 hours.

Base colour application

Base colour was applied on the flat surface of the chip. Black permanent marker was used for applying base colour. Base colour ensures adequate contrast between the base and chalk layer for easier image analysis and possible automation of analysis.

Chalk application

On the base colour a chalk layer was applied using calcium carbonate chalk, fraction 44 μm. Chalk is filling gaps and pores of transverse cut and thus creating contrast between base colour and chalk layer applied. Figure 13 gives an illustrative outlook of chip preparation procedure.

Figure 13: Chip preparation procedure

Source: own [cit. 2024-02-13]

Microscope photography

Optical microscope with camera was used to make images of prepared surface of chip (Figure 14). It was important to know the resolution of images enabling recalculation from pixels to micrometres during analysis. Length of each chip was measured with digital sliding caliper for scaling recalculation from pixels to micrometres. As wood chip

is usually longer then wider, several images of chip surface were taken to ensure higher resolution and optimal focus of microscope lens.

Figure 14: Microscope photography

Source: own [cit. 2024-02-13]

Image editing

Before the analysis itself, images had to be edited in two steps. Ad 1 choosing the best images from images taken, ad 2 merging images into panorama (Figure 15) – as chip usually has a bigger length than width, whole chip was photographed divided into few sections enabling higher resolution of images taken.

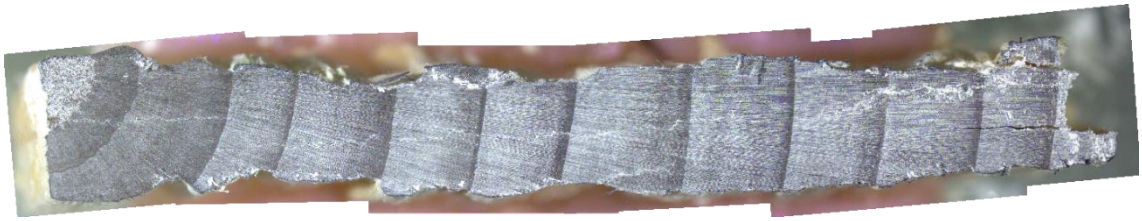


Figure 15: Image merging

Source: own [cit. 2024-02-13]

Image analysis

Finally, image analysis has been done using NIS elements software (Figure 16). For each chip parameters were defined:

- chip area,
- maximum and minimum chip projection for length,
- maximum and minimum chip projection for width,
- single crack area,

- single crack maximum and minimum projection for length,
- single crack maximum and minimum projection for width.

Those parameters were calculated both in pixels [px] and recalculated to micrometres [μm] to avoid recalculation deviation and for comparing both results. Statistical evaluation of data gained has been carried out using mainly Mann-Whitney nonparametric U-test based on stating null and alternative hypothesis and is fully described in chapter results of the thesis.

Figure 16: Image analysis

Source: own [cit. 2024-02-13]

4.3 Pulp production methodology

Pulp was produced from different chip samples at the controlled environment at the University of Pardubice (UPCE) lab with defined kappa number target $\kappa = 55$ under the supervision of Ing. Jan Gojný, Ph.D and Ing. Břetislav Češek, CSc. Cooking conditions were simulating conditions of Kamyr-line continuous production line at Mondi Štětí pulp mill, which was requested by the supervisor of the thesis topic Dr. Johannes Leitner. Base white liquor was originally got from Mondi Štětí pulp mill with defined chemical characteristics as substance content of Na_2S and NaOH. Even though characteristics of white liquor was predefined, control measurement of substances content was conducted according to ISO 23774:2023 Pulps — Kraft liquor — Determination of total, active and effective alkali using potentiometric titration in UPCE lab under the supervision of Ing. Jan Gojný, Ph.D. Following Table 6 represents both input parameters of white liquor and main setting of cooking parameters used for pulp production.

nr. of cooking	2	3
place	UPCE	UPCE
date	22.02.2024	12.03.2024
samples	6	6
active alkali [g Na2O/l]	89,07	89,07
Na_2S	29,76	29,76
NaOH	59,31	59,31
sulphidity	33,40%	33,40%
hydromodulus	2,9	2,9
mwood w [g]	80	80
dry content	88,50%	88,50%
mwood dry [g]	70,8	70,8
mwater [g]	9,2	9,2
active alkali [%]	14,50%	14,50%
active alkali [g Na2O/l]	10,27	10,27
cooking liquor [ml]	205,4	205,4
white liquor [ml]	115,3	115,3
water [ml]	80,9	80,9

Table 6: cooking parameters

Source: own [cit. 2024-02-29]

Before experiment, chips were conditioned for approx. w ~ 12 % (t = 20°C; RH = 65 %; t = 24h) and precise moisture content was calculated using formula 4.5 to ensure precise dry mass input of wood chips.

$$MC = \left(\frac{m_{wet} - m_{dry}}{m_{dry}}\right) * 100\ [\%] \tag{4.5}$$

Hot oil rotating pulp cooker VŠ-01/84 (produced by VÚPC Bratislava) available at University of Pardubice lab was used for the experiment with external dimensions of the casing were 610 × 1050 × 1030 mm. The cooker consists of 6 cooking vessels, the bath, the wire construction, and the control panel. The description of hot oil rotating pulp cooker VŠ-01/84 is based on previous dissertation thesis study by Hájková (2019).

Six cylindrical pressure vessels, so-called Hägglund bombs (Figure 17), consists of a steel shell with a convex bottom terminated by a flange. There are six boiling vessels. These vessels include steel lids, which are attached to the shell flange with six screws. A 5 mm thick Teflon gasket is inserted between the vessel flange and its lid. The inner diameter of the pressure vessels, each with a volume of 0,75 l, is 50 mm and their height is 380 mm (Hájková, 2019). The wall thickness of the vessels is 5 mm. Due to the thickness and thermal conductivity of the material (steel with a thermal conductivity of

50 W/m*K) of the pressure vessels, it was assumed that the temperature difference outside and inside these vessels was negligible (Hájková, 2019).

Figure 17: Cooking pressure vessels, Hägglung bombs

Source: own [cit. 2024-02-29]

The volume of the bath is 55 Liters. The bath, which is partially filled with silicone oil, was heated by three heating elements with a total power of 7,8 kW (Hájková, 2019). The temperature of the oil bath is automatically regulated by a thermostat.

This cylindrical structure is located in the bath, partially immersed in the oil bath, and perform a rotating motion around its horizontal axis. The rotation frequency is 2 min^{-1}. A 1,1 kW electric motor is used for the drive (Hájková, 2019). In this structure, pressure vessels are placed horizontally and immersed in the oil bath during half of the rotation.

The operation of the cooker is controlled by control panel. The desired temperature is set using the temperature scale. The temperature sensor output is also located here and shows the current temperature of the silicone bath on the scale. This is also measured by a mercury thermometer, the value of which was recorded as a control. On the front of the panel there is the main switch, the heating element switch and the switch for rotating the wire frame with the inserted pressure vessels.

Six small pulp vessels (Figure 18) were used for the experiment (each for ~ 50-100g of raw wood chips, depending on its fraction). 3 vessels were filled with the same sample (2 samples = 6 vessels) at the same time with 3 different times of exposition for each sample. This procedure was repeated 2 times. Cooking liquor from Mondi Štětí were used for pulp production and is described in Table 6. After cooking, washing and defibration of pulp, samples were conditioned and examined for:

1. H-factor,
2. unscreened yield of pulp,
3. shive’s content,
2. screen yield of pulp,
4. kappa number,
5. zero-span fibre strength
6. and residual alkali in black liquor.

Figure 18: Hot oil pulp cooker VŠ-01/84

Source: own [cit. 2024-02-29]

Cooking process is depicted in following charts. Chart 2 and Chart 3 represents corelation of time and temperature, so called “cooking curve”, in cooking experiments. Cooking process was modified regarding a capabilities of cooking device. In period 1 of cooking process temperature was constantly increasing until 115 – 120°C, after reaching desired temperature period 2 was initiated maintaining temperature constant for 20 minutes. After that, temperature was again constantly increasing until 154°C described as period 3. In period 4 temperature remained constant until reaching desired H-factor. Aim was to reaching area close to Kamyr-line usual kappa number = 55. Having wider area of H-factor (multipole samples with various cooking stages) enables plot correlation – function of different pulp parameters. Temperature was recorded every 3 minutes.

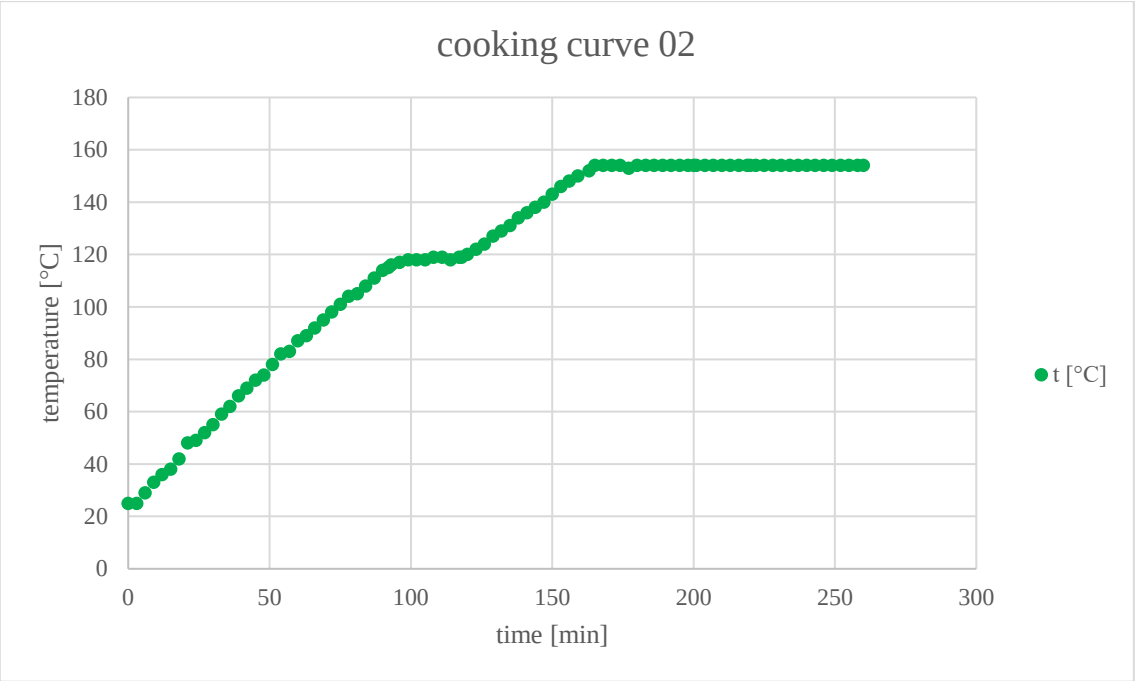


Chart 2: Cooking curve 02

Source: own [cit. 2024-02-13]

In cooking 02 in was reached H-factor of 198, 278 and 437. For each H-factor there was 2 samples – one representing "sharp" knife cutting conditions and second representing "dull" knife cutting conditions. In cooking 03 H-factor 452, 500 and 548 was reached resulting in variety of 6 different cooking stages.

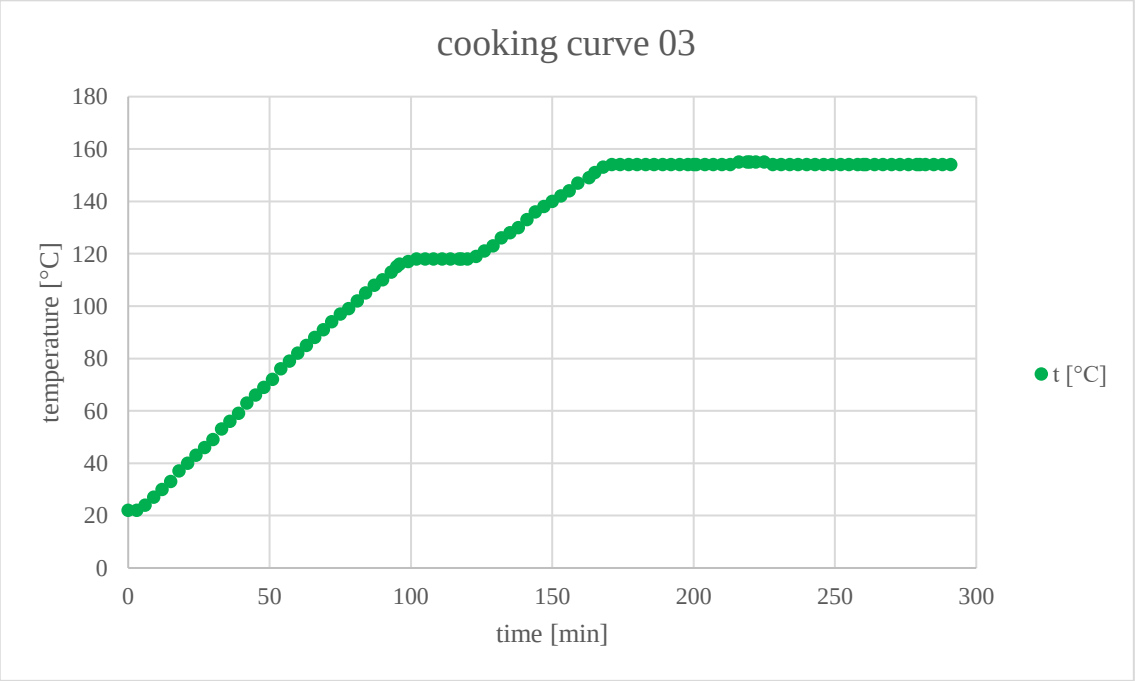


Chart 3: cooking curve 03

Source: own [cit. 2024-02-13]

Based on cooking curve, H-factor was calculated is MS Excel for every sample using trapezoidal rule (following equation 4.6).

$$\int_a^b f(x)\,dx. \tag{4.6}$$

Pulp produced by cooking procedure is depicted in Figure 20 and Figure 21. After cooking black liquor was separated and pulp was disintegrated (in standardized disintegrator machine Figure 19) and washed until reaching neutral pH value, pH = 7 ± 0,2. Pulp was collected and dried in lab conditions (RH = 65 %, t = 20 °C) for 96 hours. Afterwards, dry content was measured for calculation of unscreened yield and sampling preparation for kappa number analysis, hand sheets production, zero-span strength testing.

Figure 19: Standardized disintegrator

Source: own [cit. 2024-02-13]

Kappa number determination was conducted in accordance with international standard TAPPI T 236 Kappa number of pulp. Determination of kappa number and hand sheets production was realised at University of Pardubice lab under the supervision of Ing. Jan Gojný, Ph.D. Residual alkali value was determined according to international standard ISO 23772: Pulps — Kraft liquor — Determination of residual alkali using potentiometric titration. Experiment was done by trained personal at the Mondi Štětí lab under the supervision of head of the laboratory Miloslav Černý.

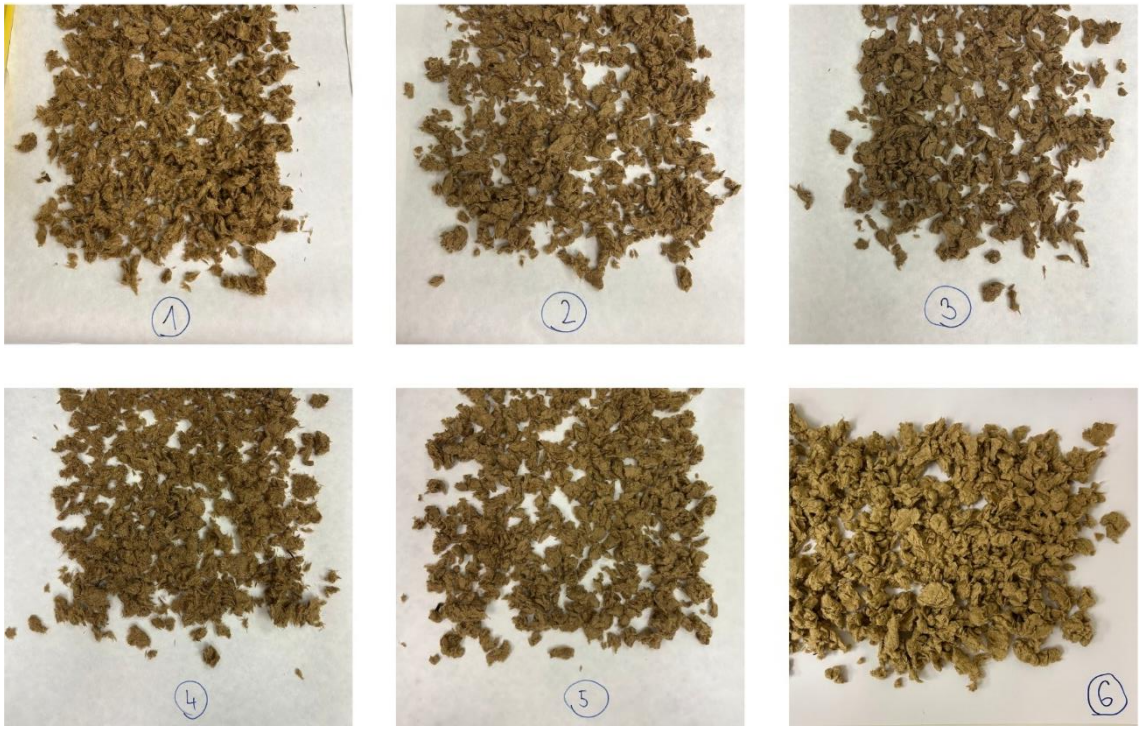


Figure 20: Pulp produced, cooking 02

Source: own [cit. 2024-02-29]

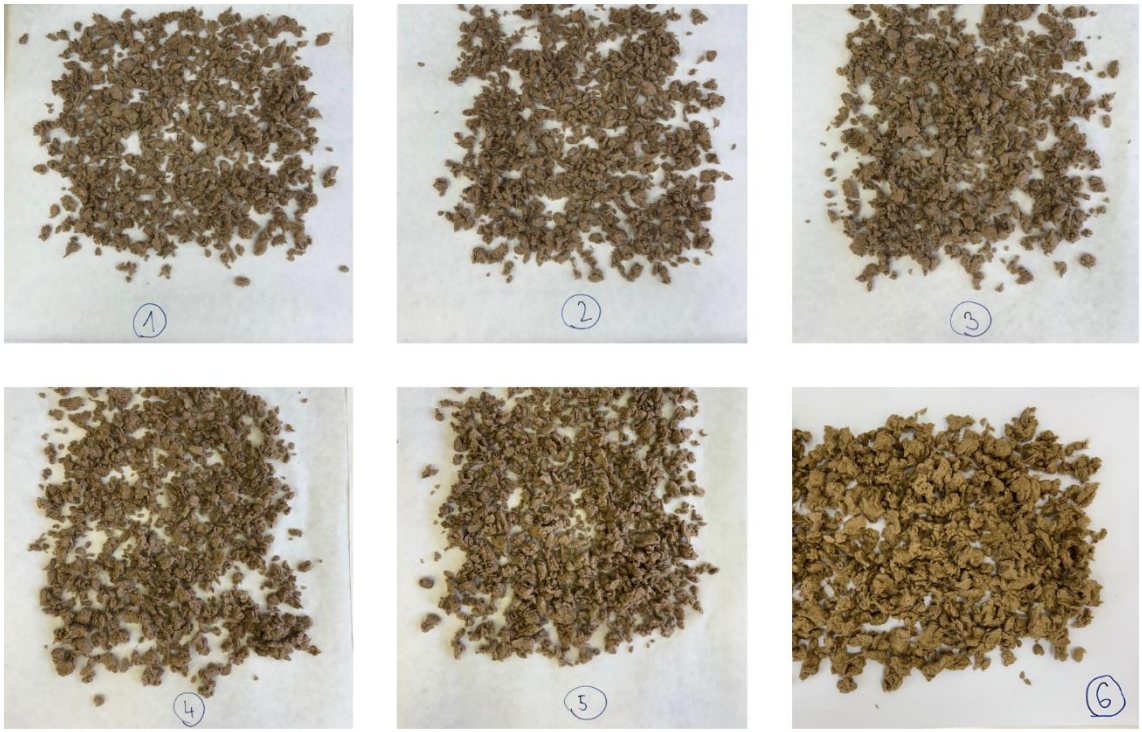


Figure 21: Pulp produced, cooking 03

Source: own [cit. 2024-02-29]

Further, standardised testing hand sheets from unscreened pulp (Figure 23, Figure 24) were formed on laboratory machine (Figure 22) according to TAPPI T 205 Forming hand sheets for physical tests of pulp. Following figures shows samples from same cooking procedures. High resolutions images of those hand sheet were taken using Canon Pixma scanner.

Figure 22: Standardised hand sheet production machine

Source: own [cit. 2024-02-13]

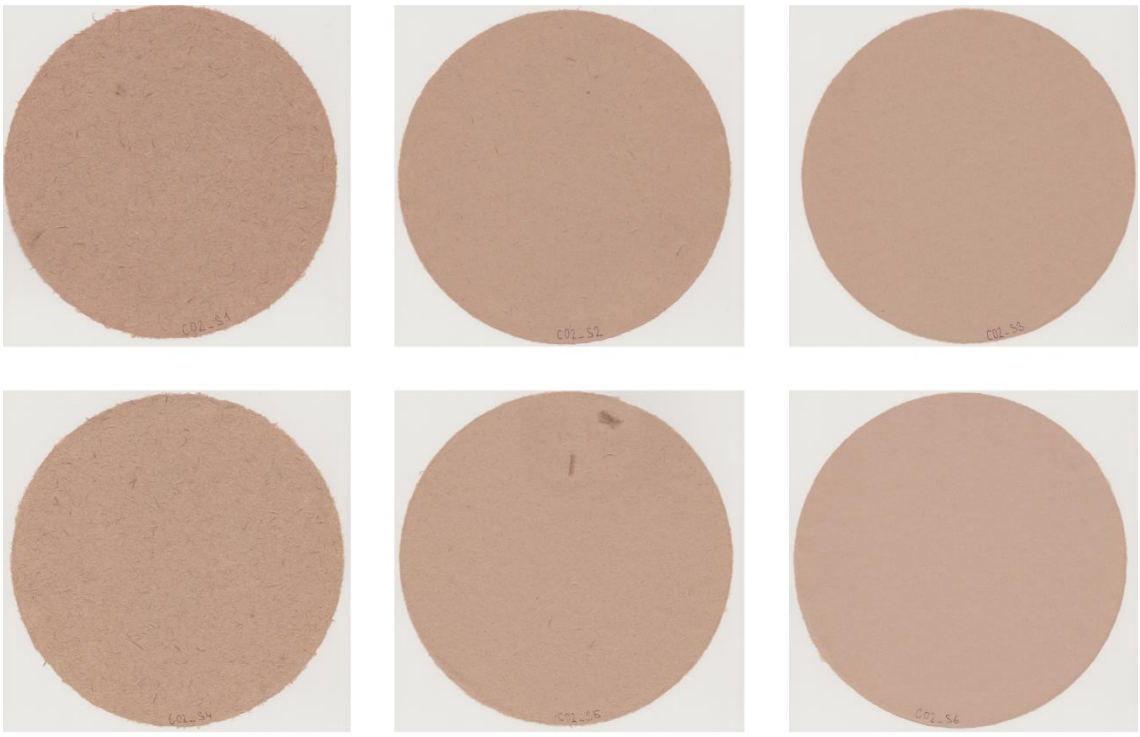


Figure 23: Shive´s content, cooking 02

Source: own [cit. 2024-02-13]

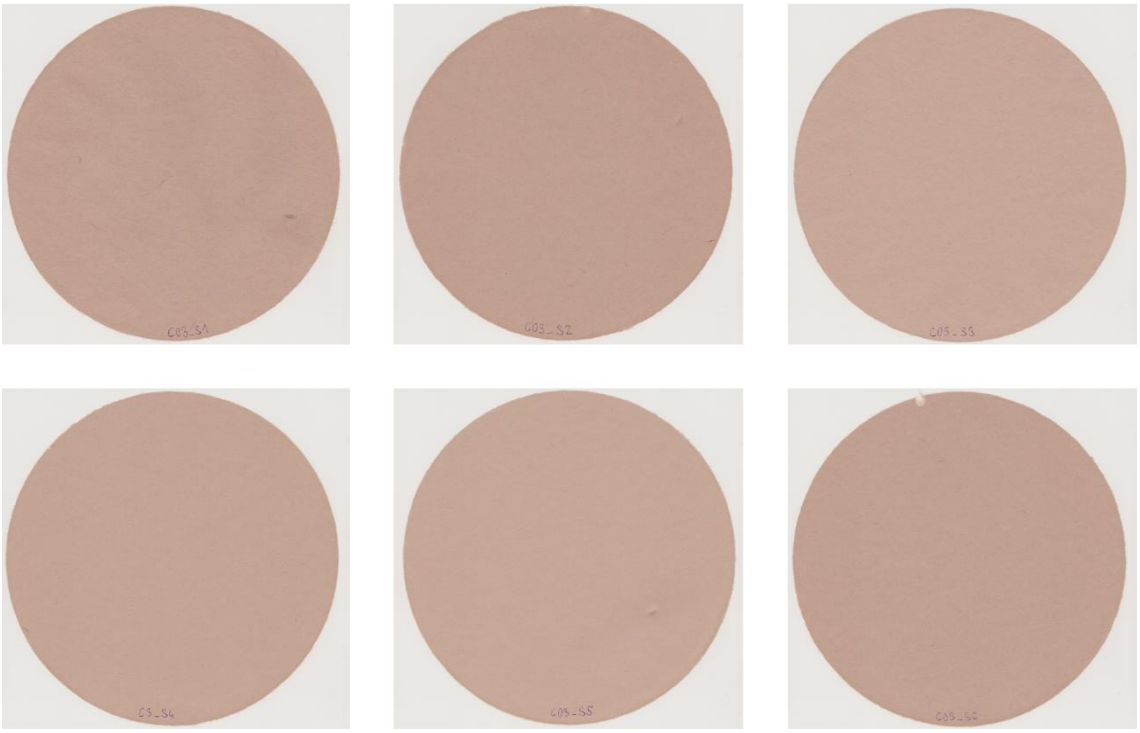


Figure 24: Shive´s content, cooking 03

Source: own [cit. 2024-02-13]

Moreover, produced pulp samples (25 g of absolute dry samples; conditioned at laboratory climate t = 20 °C; RH = 65 %) were taken to Mondi Štětí lab, where pulp was screened at the SOMERVILLE, typ K 134 device according to standard Tappi T 275. Additionally, shives content was defined by Tappi um 242 and based on the shives content of pulp screened yield of pulp was calculated.

Comparison of defined characteristics was conducted between examined samples and statistically evaluated as an "indirect" method of examination of a wood chip cracks impact on produced pulp. Finally, comparation to "optical method" introduced in Practical part I was managed.

5 Results

5.1 Image analysis

Results show increase of average crack area ratio by 0,79 % in dull knife cutting conditions unlike in sharp knife cutting. For statistical analysis Mann-Whitney non-parametric U-test was used. Hypothesis H_0 was stated: There is no difference between the "dull" knife cut sample and the "sharp knife" cut sample crack surface area with respect to the dependent variable value. And alternative hypotheses H_1 was stated: There is a difference between the "dull" knife cut sample and the "sharp knife" cut sample crack surface area with respect to the dependent variable value. Firstly, data were tested on a significance level $\alpha = 0,05$, where the H_0 hypothesis cannot be rejected with p-value = 0.05148. For rejecting H_0 significance level must be decreased for $\alpha = 0,06$. At this significance level H_0 is rejected and data obtained could be considered as statically significant at 94 % region of acceptance. It is assumed that 94 % region of acceptance is reliable enough as only small differences between the chip crack surface area in both samples were expected in regard to the initial hypothesis of the paper.

Furthermore, results show decrease of average crack length by -3,89 % with calculation in pixels or -4,65 % using recalculation to micrometres in sharp knife cutting conditions unlike in dull knife cutting (Table 7).

For the decrease of average crack length by -3,89 % calculation in pixels, statistical analysis the Mann-Whitney non-parametric U-test was used. H_0 was stated: There is no difference between the "dull" knife cut sample and the "sharp knife" cut sample average crack length with respect to the dependent variable value. And alternative hypotheses H_1 was stated: There is a difference between the "dull" knife cut sample and the "sharp knife" cut sample average crack length with respect to the dependent variable value. Data was tested within the significance level $\alpha = 0,05$. The p-value = 0,578 and thus hypothesis H_0 cannot be rejected with respect to the potential chance of type I error 57,8 % and it has to be assumed that data obtained are not statistically significant.

For the decrease of average crack length by -4,65 % recalculation to micrometres, statistical analysis the Mann-Whitney non-parametric U-test was used. H_0 was stated: There is no difference between the "dull" knife cut sample and the "sharp knife" cut sample average crack length with respect to the dependent variable value. And alternative hypotheses H_1 was stated: There is a difference between the "dull" knife cut sample and

the “sharp knife” cut sample average crack length with respect to the dependent variable value. Data was tested within the significance level $\alpha = 0,05$. The p-value = 0,8551 and thus hypothesis H_0 cannot be rejected with respect to the potential chance of type I error 85,51 % and it has to be assumed that data obtained are not statistically significant. The higher p-value and thus higher chance of statistical error in results recalculated to micrometres compared to calculation in pixel has been caused by recalculation itself increasing standard deviation by lowering precision in every value recalculated.

However, there is limitation to the statistical results regarding comparison of average chip crack length, too. It is expected to get large variability of data as chip cracks do not oscillate around the average value, so regardless the denial of statistical significance of data, it is still important to not avoiding interpretation of average chip crack length results obtained.

average length projection ("dimension")			
	dull knife	**sharp knife**	**difference**
pixel [px]	2828,31	2722,54	-3,89 %
real [µm]	4561,94	4359,35	-4,65 %

Table 7: Comparation of length projection of cracks

Source: own [cit. 2024-02-13]

Nevertheless, results show increase of average crack width by 6,96 % (calculated in pixels) and 6,57 % (calculated in micrometres) in sharp knife cutting conditions unlike in dull knife cutting (Table 8).

For the increase of average crack width by 6,96 % calculation in pixels, statistical analysis the Mann-Whitney non-parametric U-test was used. H_0 was stated: There is no difference between the “dull” knife cut sample and the “sharp knife” cut sample average crack width with respect to the dependent variable value. And alternative hypotheses H_1 was stated: There is a difference between the “dull” knife cut sample and the “sharp knife” cut sample average crack width with respect to the dependent variable value. Data was tested within the significance level $\alpha = 0,05$. The p-value = 0,7054 and thus hypothesis H_0 cannot be rejected with respect to the potential chance of type I error 70,54 % and it has to be assumed that data obtained are not statistically significant.

For the increase of average crack width by 6,57 % recalculation in micrometres, statistical analysis the Mann-Whitney non-parametric U-test was used. H_0 was stated: There is no difference between the “dull” knife cut sample and the “sharp knife” cut sample average crack width with respect to the dependent variable value. And alternative hypotheses H_1 was stated: There is a difference between the “dull” knife cut sample and the “sharp knife” cut sample average crack width with respect to the dependent variable value. Data was tested within the significance level $\alpha = 0,05$. The p-value = 0,5862 and thus hypothesis H_0 cannot be rejected with respect to the potential chance of type I error 58,62 % and it has to be assumed that data obtained are not statistically significant.

Additionally, possible deviation caused by manual image analysis regarding the crack characteristics (length of crack is much larger than its width) leads to the conclusion, that average crack width cannot be used as reliable indicator in relation to initial hypothesis of the paper or its interpretation.

average width projection ("dimension")			
	dull knife	**sharp knife**	**difference**
pixel [px]	351,44	377,72	6,96 %
real [µm]	560,73	600,15	6,57 %

Table 8: Comparation of width projection of cracks

Source: own [cit. 2024-02-13]

5.2 Pulp production

Chart 4 depicts correlation of kappa number on H-factor comparing “sharp” and “dull” knife cut samples. Curve was plotted using nonlinear regression based on the set of 5 data points for both samples as extreme values were terminated. Correlation coefficient for “sharp” knife cut sample was $c_{S2} = -0,996$ and for dull knife cut sample $c_{S3} = -0,998$ resulting in very strong correlation of data with respect to Evans (1996). Results show the difference between both samples, greater in low H-factor narrowing with higher H-factor. Dull knife cut positively influenced cooking procedure resulting in lower kappa number (more lignin was cooked out) at the similar kappa number. While in lower H-factor are differences greater, in higher H-factor are differences are blur. It could be

presumed, that differences were caused by better penetration of cooking liquor inside the chip through chip cracks, which correlates with hypothesis.

When comparing targeted value of kappa number 55, a straight horizontal line was fitted to the graph at y value 55 (kappa number target) and x value (H-factor) was calculated using exponential curve equations for both “sharp” and “dull” knife cut sample correlations. It could be concluded that in the matter of correlation of kappa number and H-factor, pulping process of “dull” knife cut chips was more efficient by 7,55 % in comparison to pulping process of “sharp” knife cut sample. The efficiency of the process means that same kappa number target κ = 55 was reached at lower H-factor, thus less energy and time for pulping process was needed for pulping “dull” knife cut sample then “sharp” knife cut sample.

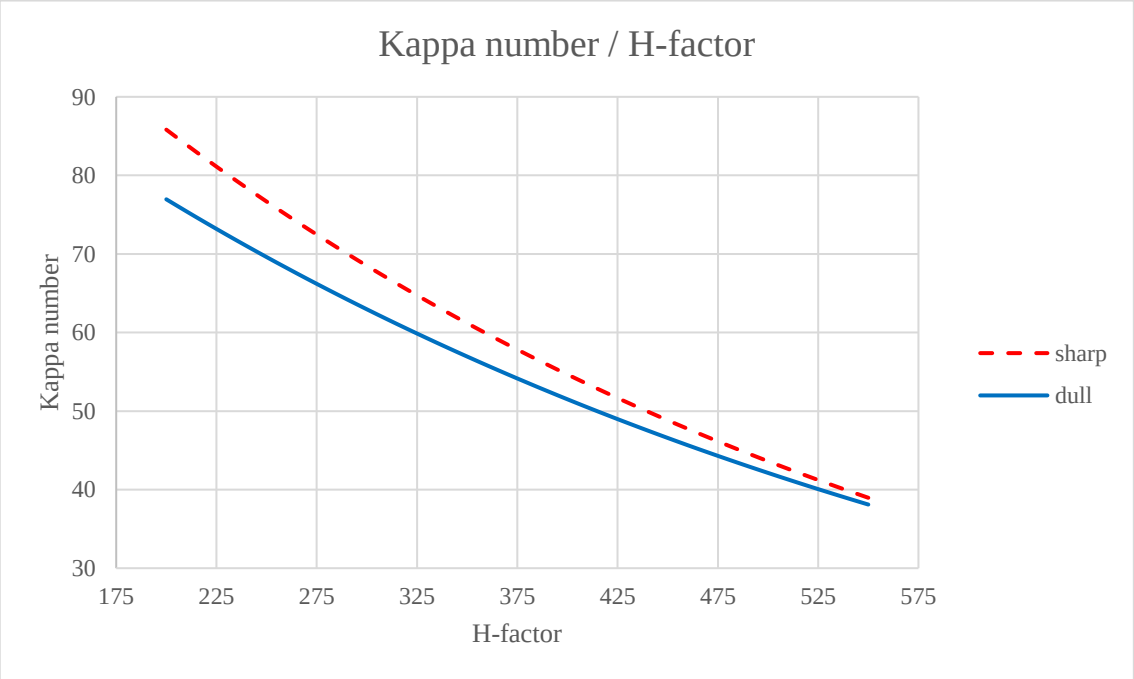


Chart 4: Blades comparison - Kappa number x H-factor

Source: own [cit. 2024-02-13]

Chart 5 compares samples correlation of unscreened yield and H-factor using nonlinear regression. It could be seen, that “dull” knife cut sample was easier to cook, which slightly negatively influenced unscreened yield compared to “sharp” knife cut sample with difference being negligible in low H-factor and greater with higher H-factor. Correlation coefficient for “sharp” knife cut sample was c_{S2} = -0,984 and for dull knife cut sample c_{S3} = -0,980 resulting in very strong correlation of data with respect to Evans

(1996). Trend of obtained results of unscreened yield on H-factor correlates with hypothesis that wood chip cracks enable faster penetration of cooking liquor and thus accelerates cooking process resulting lower unscreened yield for “dull” knife cut sample compared to “sharp” knife cut sample at the same H-factor.

For comparison the H-factor = 382 was used, which approximates with kappa number target κ = 55. The H-factor comparison target value was calculated as simple average of H-factor´s obtained by nonlinear regression analysis at kappa target κ = 55 for both “sharp” knife cut and “dull” knife cut samples (depicted in Chart 4). A straight vertical line was fitted to the graph at x-value 382 (H-factor target) and y-value (Unscreened yield of pulp) was calculated using exponential curve equations for both “sharp” and “dull” knife cut sample correlations. With respect to H-factor target (H-factor = 382) unscreened yield of “dull” knife cut sample was ΔUY = -1,54 % in comparison to “sharp” knife cut sample.

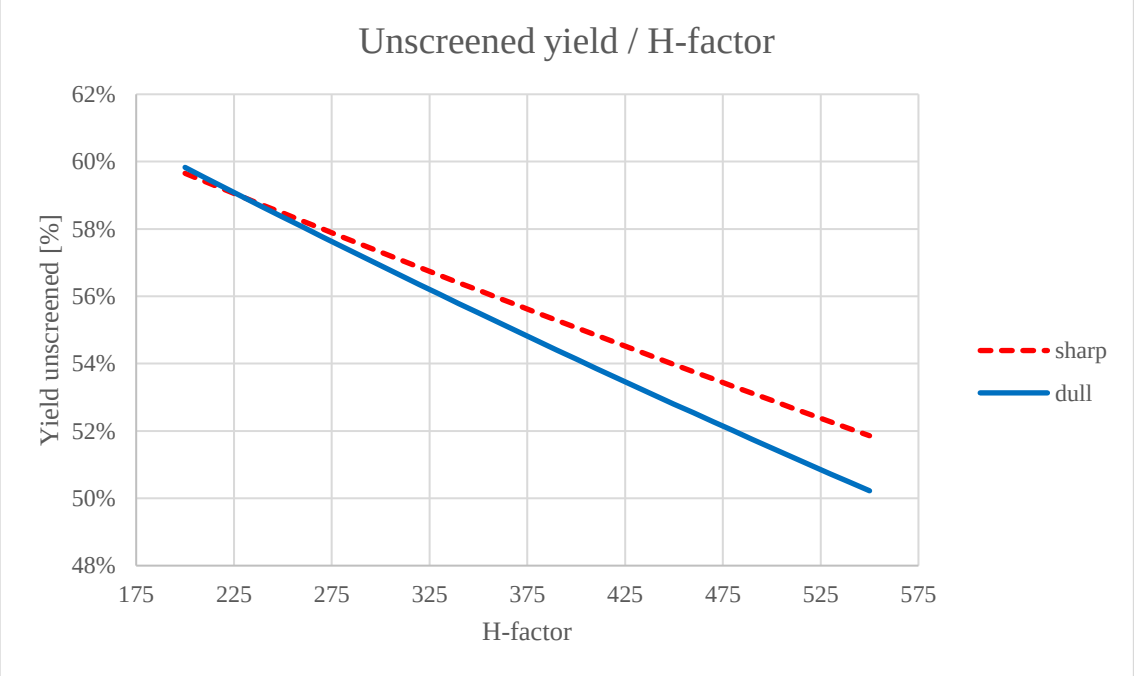


Chart 5: Blades comparison - Unscreened yield x H-factor

Source: own [cit. 2024-02-13]

Correlation of unscreened yield and kappa number is provided by Chart 6. Results show higher yield with “dull” knife cut sample compared to “sharp” knife cut sample at the same kappa number. Results are considered to prove very strong correlation of data with respect to Evans (1996) as correlation coefficients were c_{S2} = 0,990 and c_{S3} = 0,970.

As in the first two cases, a comparison of kappa number target κ = 55 was conducted fitting a straight vertical line to the graph at x-value 55 (kappa number target) and y-value (unscreened yield of pulp) was calculated using exponential curve equations for both "sharp" and "dull" knife cut sample correlations. When comparing desired kappa target κ = 55, unscreened yield is positive for "dull" knife cut sample by ΔUY = 1,50 %.

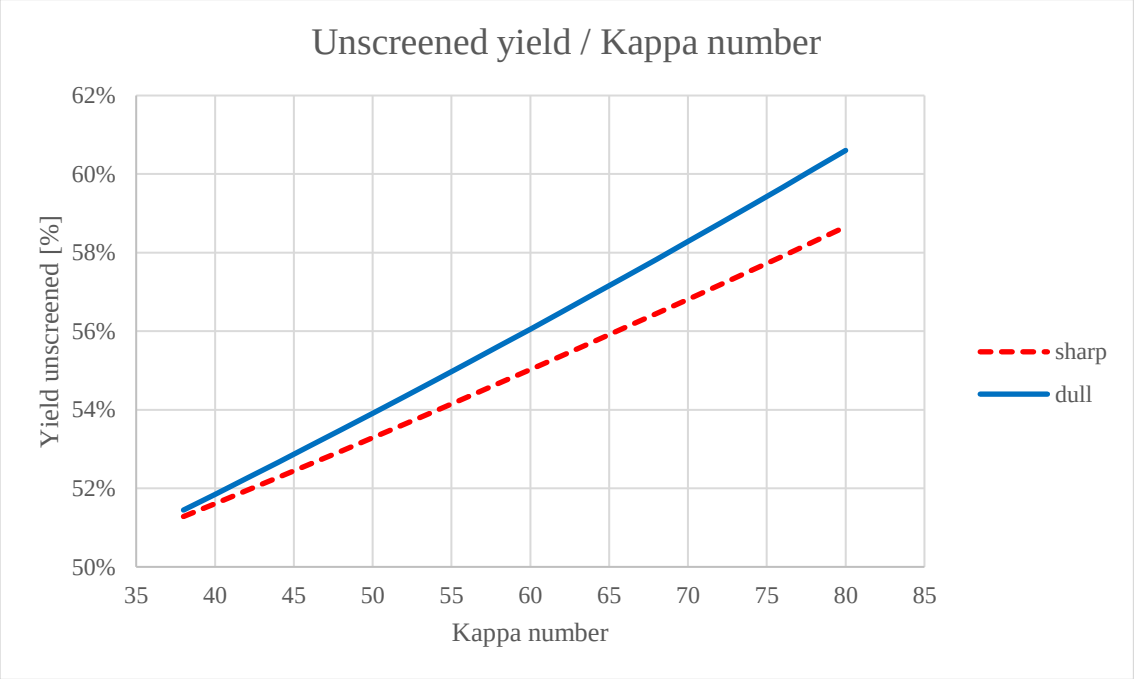


Chart 6: Blades comparison - Unscreened yield x Kappa number

Source: own [cit. 2024-02-13]

Following Chart 7 represents residual alkali in black liquor with correlation on H-factor. Residual alkali was determined by international standard ISO 23772: Pulps — Kraft liquor — Determination of residual alkali using potentiometric titration. The results indicate correlation among variables across the two examined datasets. The correlation is at the limit of "strong" correlation (in consideration of terminology of Evans 1996) regarding the correlation coefficients for "sharp" knife cut dataset c_{S2} = -0,90 and for "dull" knife cut dataset c_{S3} = -0,80. Regarding the correlation coefficients, comparison cannot but use as direct prove of difference between both datasets, as both sample groups are intermingling with respect to correlation deviation. Nevertheless, if willing to compare, results could be interpreted in the term that more cracks in "dull" knife resulted in lower residual alkali in black liquor at the same desired kappa number κ = 55 (equivalent of H-factor = 382, respectively) by ΔRA = -4,33 % unlike in "sharp" knife cut sample.

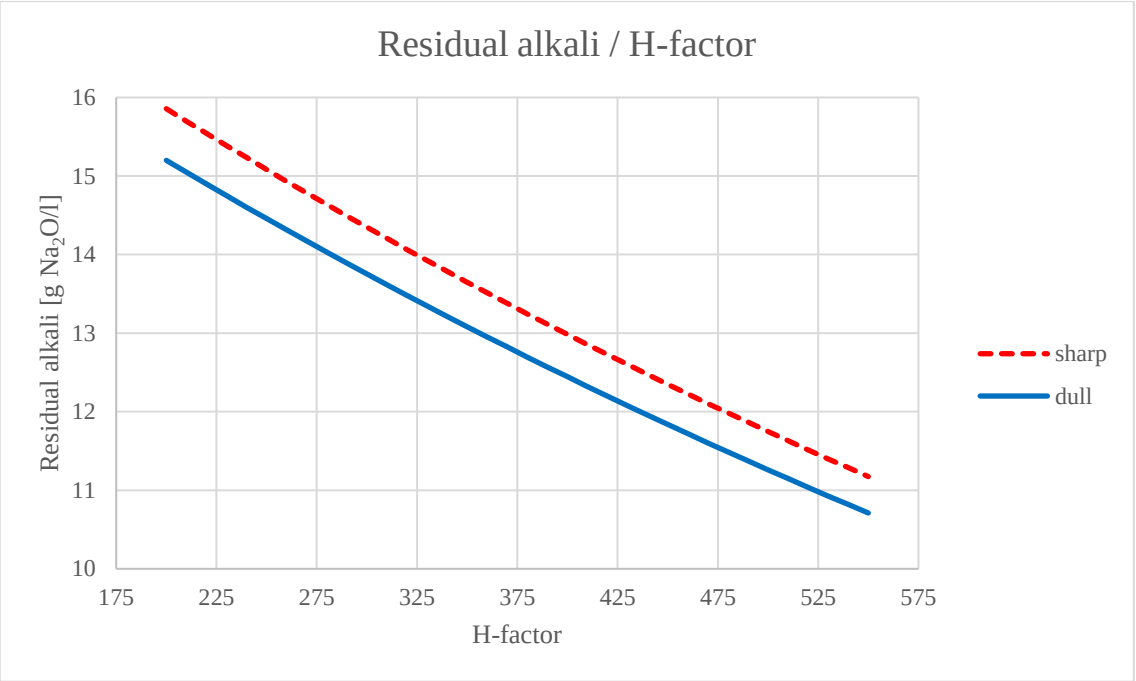


Chart 7: Blades comparison - Residual alkali x H-factor

Source: own [cit. 2024-04-03]

Connected to Chart 7 is Chart 8 representing residual alkali in black liquor with correlation on Kappa number. It could be seen that lower residual alkali was achieved in "dull" knife cut sample with more wood chip cracks with respect to corelation coefficients for "sharp" knife cut dataset c_{S2} = 0,90 and for "dull" knife cut dataset c_{S3} = 0,86. In addition, for the same desired kappa number target κ = 55 residual alkali was lower in "dull" knife cut sample by ΔRA = -14,79 % unlike in "sharp" knife cut sample. This value cannot be used as direct prove of the difference between both datasets with respect to potential intermingling of both datasets in reference to correlation coefficients.

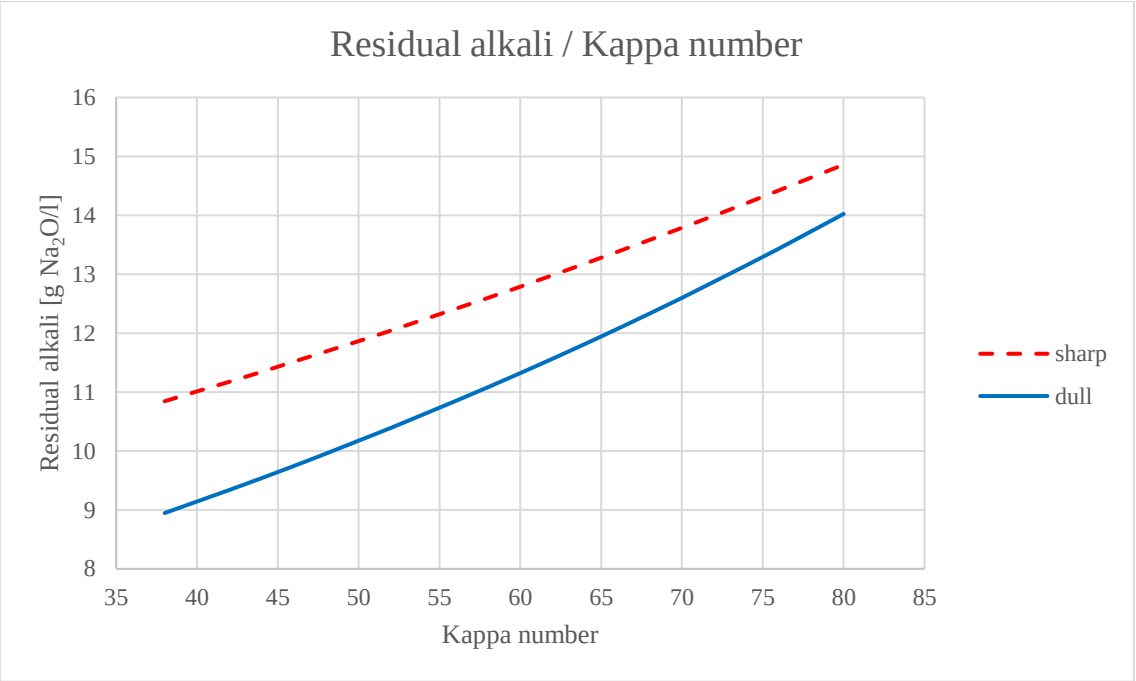


Chart 8: Blades comparison - Residual alkali x Kappa number

Source: own [cit. 2024-04-03]

Zero-span fibre strength was tested in Mondi Štětí lab for comparing fibre strength of both samples. Zero-span test results are in "fibre strength" units [FS] explained by following equation (5.7) according to PULMAC device manual (Mondi Štětí, 2024).

$$Fibre\ Strength\ Number = Avg.of\ Wet\ Zero\ Spans * \frac{Actual\ Basis\ Weight}{Target\ Basis\ Weight} = \frac{N}{CM} @\ 60\ grams/m^2 \quad (5.7)$$

The trends of correlation of Zero-span fibre strength and H-factor is depicted in Chart 9. Even though the trend show difference in between both datasets, results cannot be considered significant according to high deviation expressed by correlation coefficients for "sharp" knife cut dataset c_{S2} = 0,64 and for "dull" knife cut dataset c_{S3} = 0,40. Assumption could be made, that higher presence of wood chip cracks results in lower Zero-span fibre strength. If comparing same desired kappa number κ = 55 (equivalent of H-factor = 382, respectively) the "dull" knife cut sample Zero-fibre strength was lower by ΔFS = -3,49 % in comparison to "sharp" knife cut sample, but therefore underlining the aspect of statistical insignificance of this result.

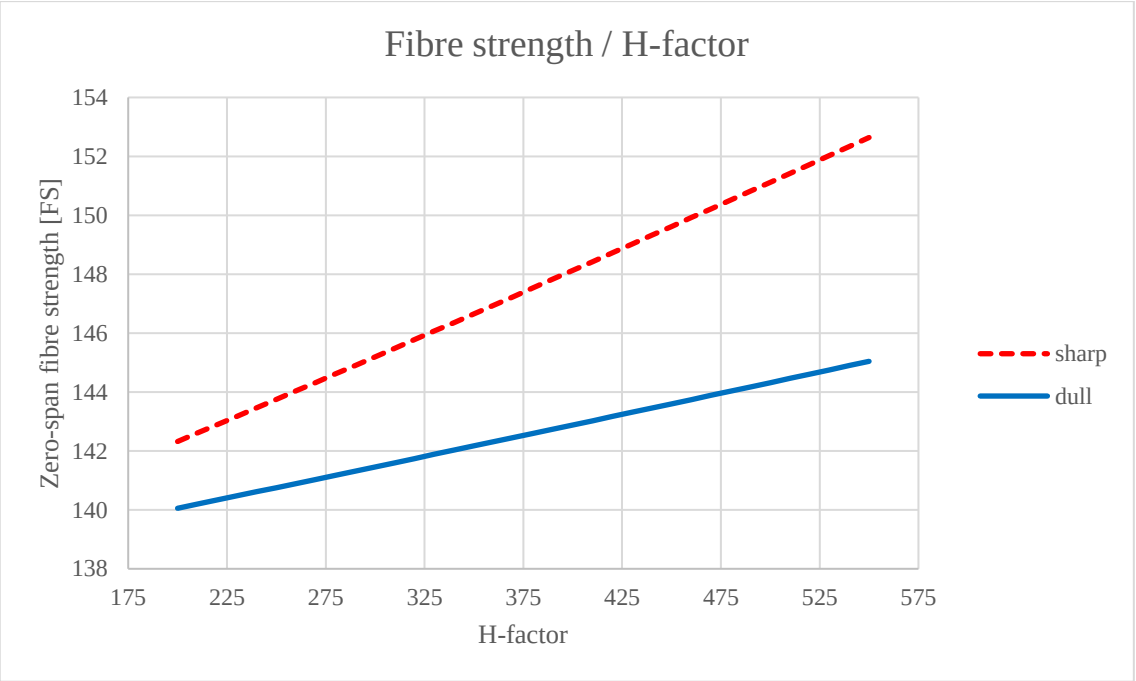


Chart 9: Blades comparison - Fibre strength x H-factor

Source: own [cit. 2024-04-03]

Comparative results of correlation of Zero-span fibre strength and Kappa number are plotted in Chart 10. Results obtained are limited by correlation coefficients for "sharp" knife cut dataset c_{S2} = -0,64 and for "dull" knife cut dataset c_{S3} = -0,42. Due to middling correlation (Evans, 1996) no precise conclusions could be made of those results. Trend show slightly higher fibre strength in "sharp" knife cut sample compared to "dull" knife cut sample. If comparing same desired kappa number $\kappa = 55$, the "dull" knife cut sample Zero-fibre strength was lower by ΔFS = -1,71 % in comparison to "sharp" knife cut sample, but therefore underlining the aspect of statistical insignificance of this result.

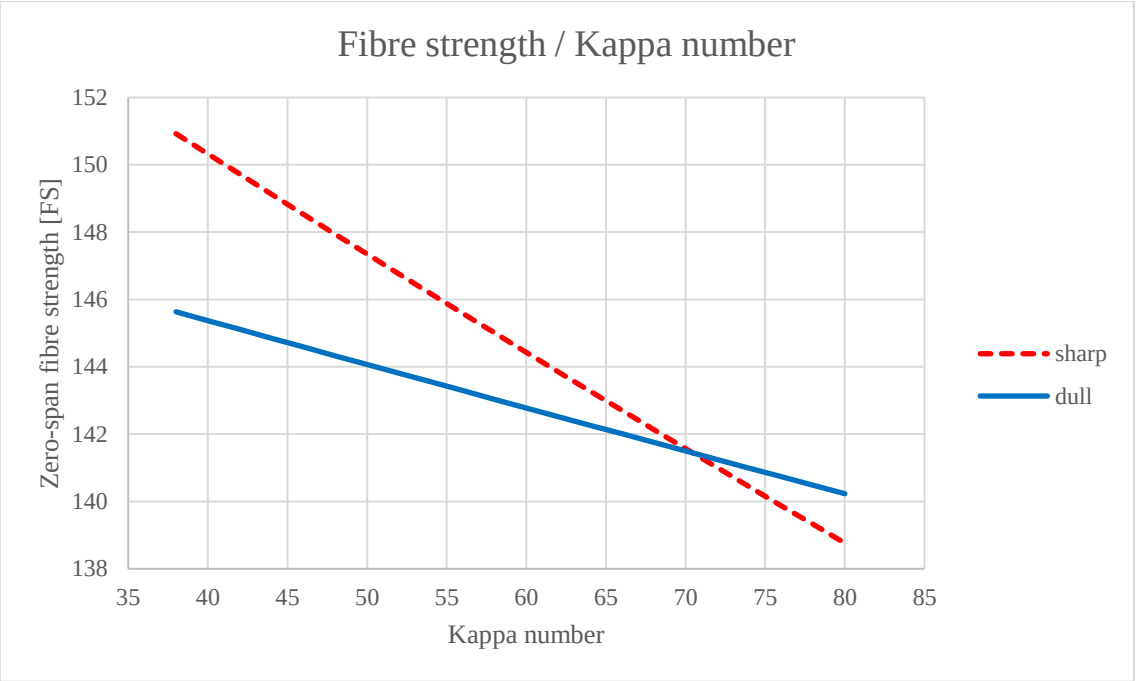


Chart 10: Blades comparison - Fibre strength x Kappa number

Source: own [cit. 2024-04-03]

Corelation of H-factor and shive´s content is represented by Chart 11. Results obtained are limited by correlation coefficients for "sharp" knife cut dataset c_{S2} = -0,83 and for "dull" knife cut dataset c_{S3} = -0,86. Correlation is not strong enough to make precise conclusions. It could be assumed, that in general trend there is higher percentage of shive´s in "dull" knife cut sample compared to "sharp" knife cut sample, but without statistical significance of this assumption. If comparing same desired kappa number κ = 55 (equivalent of H-factor = 382, respectively), the "dull" knife cut sample shive´s content was higher by ΔSC = 9,27 % in comparison to "sharp" knife cut sample, but therefore underlining the aspect of statistical insignificance of this result.

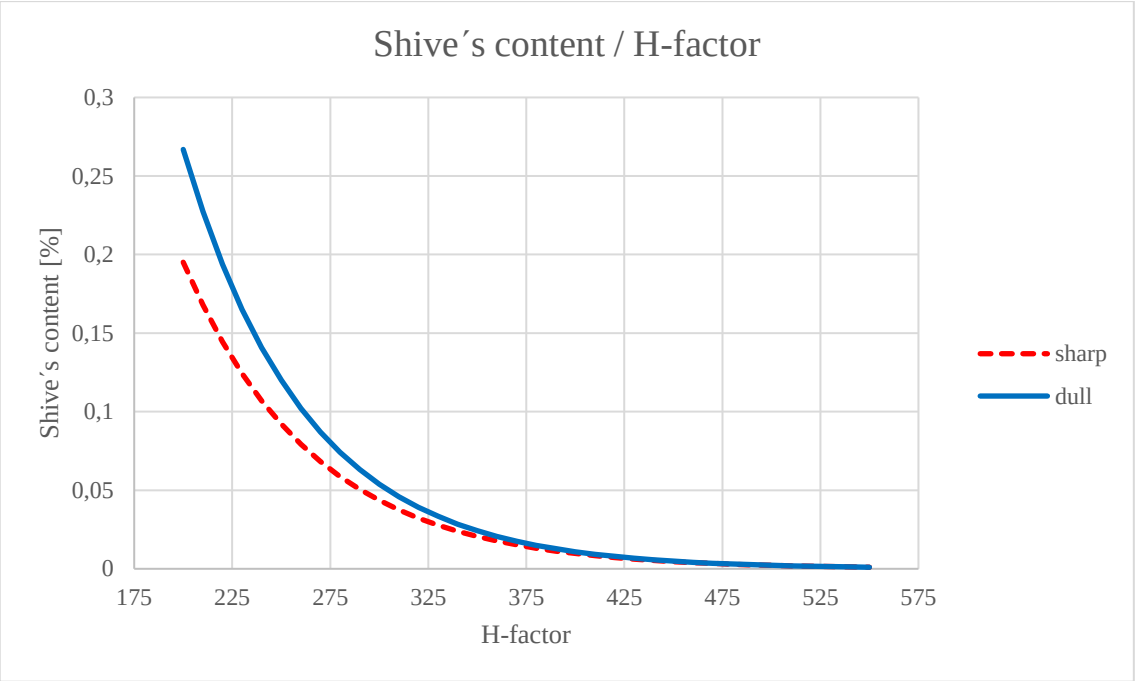


Chart 11: Blades comparison – Shive's content x H-factor

Source: own [cit. 2024-04-04]

Corelation of H-factor and screened yield is depicted by Chart 12. Results obtained are limited by correlation coefficients for "sharp" knife cut dataset c_{S2} = 0,63 and for "dull" knife cut dataset c_{S3} = 0,69. Correlation is not strong enough to make precise conclusions. If comparing same desired kappa number κ = 55 (equivalent of H-factor = 382, respectively), the "dull" knife cut sample screened yield was higher by ΔSY = 1,19 % in comparison to "sharp" knife cut sample, but therefore underlining the aspect of statistical insignificance of this result.

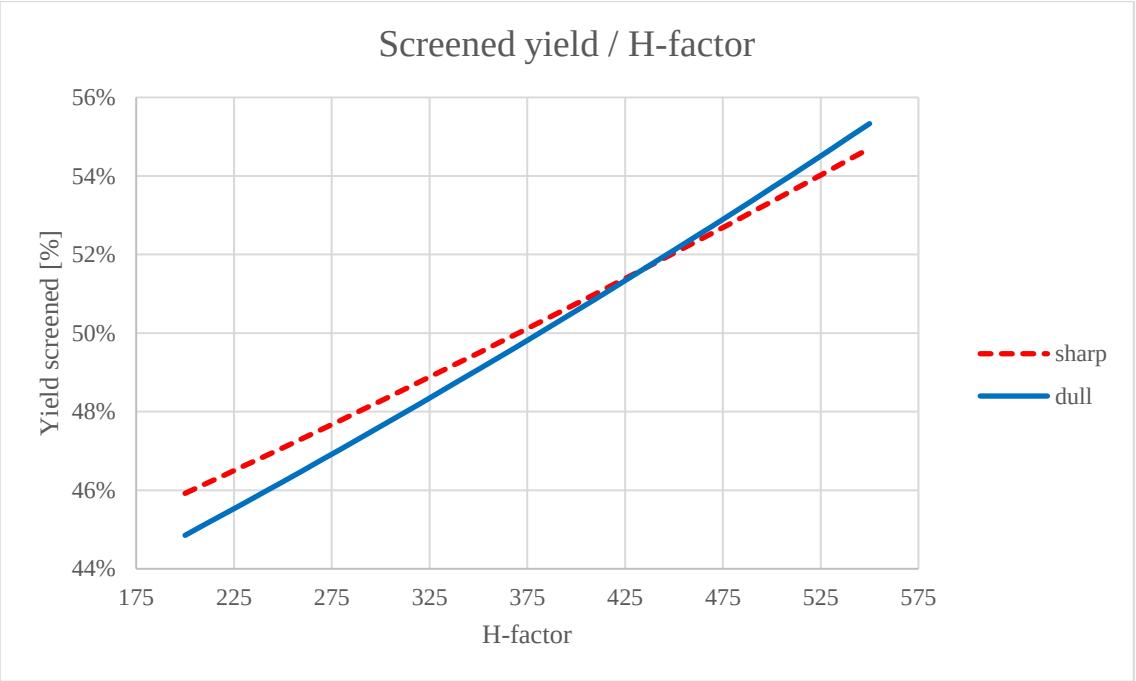


Chart 12: Blades comparison – Screened yield x H-factor

Source: own [cit. 2024-04-04]

On contrary, higher screened yield was reached in “dull” knife cut sample at the same kappa number target unlike in “sharp” knife cut sample (depicted in Chart 13). Results obtained are limited by correlation coefficients for “sharp” knife cut dataset c_{S2} = -0,67 and for “dull” knife cut dataset c_{S3} = -0,66. Correlation is not strong enough to make precise conclusions. If willing to compare same desired kappa number $\kappa = 55$, the “dull” knife cut sample screened yield was higher by $\Delta SY = 3{,}33$ % in comparison to “sharp” knife cut sample. The similar trend is to be seen in those results as if compared to the correlation of unscreened yield and kappa number (Chart 6). Nevertheless, statistical insignificance of this results must be considered regarding unreliable correlation coefficients.

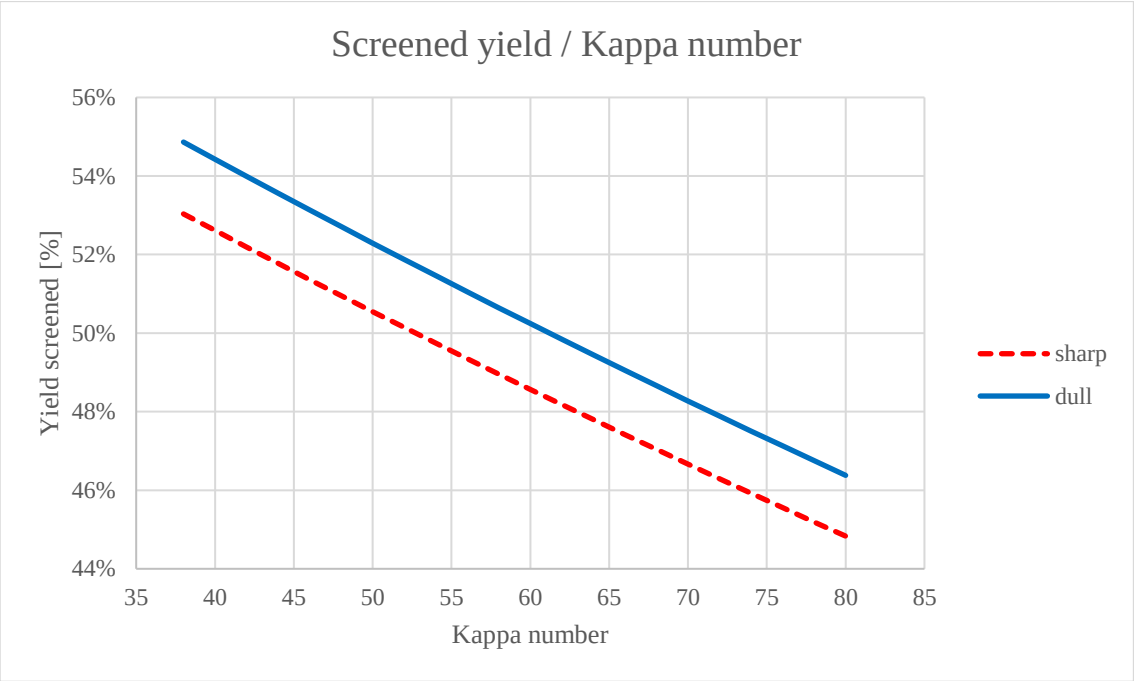


Chart 13: Blades comparison – Screened yield x kappa number

Source: own [cit. 2024-04-04]

To sum up the results, Table 9 shows comparison of the key values obtained by pulp production methodology and mainly differences between “dull” and “sharp” knife cut samples expressed in difference percentage at target value corresponding to Mondi Štětí Kamyr-line production, kappa number κ = 55 or equivalent as H-factor = 382. Approaches leading to general comparison in Table 9 are fully described above following description of each chart in pulp production methodology chapter. It was found that kappa number target κ = 55 was reached in lower H-factor by -8,16 % in “dull” knife cut sample unlike in sharp knife cut sample, which supports the hypothesis, that wood chip cracks (represented by “dull” knife cut sample) enable faster penetration of cooking liquor and thus fasten a pulping process. This is confirmed by other finding of unscreened yield at the H-factor target (H-factor = 382), corresponding to kappa number target κ = 55 (described above in explanation related to Chart 5). Unscreened yield was reduced by - 1,54 % in “dull” knife cut sample unlike in “sharp” knife cut sample, which is assumed to be caused by better cooking (“delignification”) stage. What is more, it was proven that unscreened yield at the desired kappa number target κ = 55 was positively influenced by increase of 1,50 % in “dull” knife cut sample unlike in “sharp” knife cut sample, leading to conclusion, that “dull” knife cut sample representing more cracks in wood chips as input pulp mill material has positive impact on pulping process efficiency in the matter

of yield. Other results obtained are limited by low correlation of variables for comparison of both samples, thus no conclusions were made from these results, only assumptions for possible interpretation of results (preceding charts with results) with respect to low correlation. Those assumptions and possible interpretation of results were not included in conclusion of the thesis.

variables of correlation based on chart			target comparison value		dull knife sample value		correl. coef. CS3	sharp knife sample value		correl. coef. CS2	Δ sharp / dull [%]
Chart 4	H-F	κ	κ	55	H-f	367,25	-0,99	H-f	397,23	-0,98	-8,16
Chart 5	H-F	UY	H-F	380	UY [%]	54,62	-0,98	UY [%]	55,46	-0,98	-1,54
Chart 6	κ	UY	κ	55	UY [%]	54,97	0,97	UY [%]	54,15	0,99	1,50
Chart 7	H-F	RA	H-F	380	RA [g Na2O/l]	12,67	-0,80	RA [g Na2O/l]	13,22	-0,90	-4,33
Chart 8	κ	RA	κ	55	RA [g Na2O/l]	10,73	0,86	RA [g Na2O/l]	12,32	0,90	-14,79
Chart 9	H-F	FS	H-F	380	FS [FS]	142,63	0,40	FS [FS]	147,60	0,64	-3,49
Chart 10	κ	FS	κ	55	FS [FS]	143,42	-0,42	FS [FS]	145,88	-0,64	-1,71
Chart 11	H-F	SC	H-F	380	SC [%]	1,47	-0,86	SC [%]	1,34	-0,84	9,27
Chart 12	H-F	SY	H-F	380	SY [%]	50,03	0,69	SY [%]	50,29	0,62	-0,53
Chart 13	κ	SY	κ	55	SY [%]	51,26	-0,66	SY [%]	49,55	-0,67	3,33

Table 9: Pulp production, sum of results

Source: own [cit. 2024-02-13]

Explanations: H-F = H-factor; κ = kappa number; UY = unscreened yield; RA = residual alkali; FS = zero-span fibre strength [FS]; SC = shives content; SY = screened yield

6 Discussion

When comparing results of methodologies used, both methodologies obtained results tend to correspond with initial hypothesis and relates to each other. In the image analysis methodology, the sample cut by “dull” knife was identified as samples having larger cracks in wood chips and by results obtained throughout pulp production methodology it was confirmed that those cracks have an impact on both kappa number and unscreened yield of the pulp. Other results regarding residual alkali, shive´s content, zero-span fibre strength and screened yield are not confirming nor denying the hypothesis stated. Those results have unreliable strength of correlation which is respected in conclusion. Based on results obtained altogether it could be concluded that image analysis methodology for evaluation of wood chip cracks leads to relevant results. Based on proving relevancy of this methodology, a lab method instruction sheet was developed for potential use of this method in future and is available as an Attachment nr.1 of the master thesis. Introduced laboratory method follows basics introduced by Rydval et al. (2024) and build upon them.

Obtained results as H-factor, kappa number and unscreened yield corresponds with liquid penetration theory of wood chips introduced Biermann (1996) described by Malkov (2002) a mentioned by and Sixta (2006). Results regarding residual alkali, zero-span fibre strength, shive´s content and screened yield tend to correspond with wood chip liquid penetration theory (Biermann, 1996; Malkov, 2002; Sixta, 2006), but are limited by unreliable correlation, thus no conclusion in both confirming nor declining hypothesis could be made. As the initial point of the thesis the hypothesis was stated by Dr. Johannes Leitner (Mondi AG), that wood chip cracks have positive impact on cooking liquor penetration of the chip resulting more efficient cooking procedure. Results partially agree with the hypothesis.

Nevertheless, it must be considered, that only small and specific sample was studied in the experimental part of the thesis and further studies have to be conducted to confirm results obtained by introduced methodologies. Introduced laboratory method (Attachment nr. 1) may serve as the basis for following research and for potential comparison of future results of studies focusing on cracks in wood chips. Methodology used in the thesis have other limitations and concerns introduced in following sub-chapter. Opportunities and potential new approaches are also mentioned in this sub-chapter.

6.1 Limitations and opportunities

Limitations regarding the methodology used to obtain the results as well as opportunities of further development of the method are expressed in this chapter. Following concerns may have influenced results and must be considered in results discussion.

Length of a chip limitation

During chip preparation, the length of the chip could not exceed 1 cm, due to the microscope used for an image photography as microscope was unable to focus transverse section with larger length dimension. Thus, not all chips could have been cut in the middle of their length resulting in slight differentiation of analysed position.

Image editing

For an image analysis images had to be merged into a panorama (using Microsoft Image Composite Editor), therefore both results in pixels and in micrometres (after recalculation) was gathered to compare their accuracy and thus validity of results obtained. It is also important to preserve input parameters when taking images for comparison validity.

Image analysis method automation

For future use of image analysis method, it is important to optimize analytical part of this method to be able to evaluate each chip automatically which will need further development of chip preparation, especially chalk application. It was found that used chalk did not fill all cracks homogenously and thus automatic evaluation could not be applied, and manual evaluation was used for image analysis method. Possible optimization could in using more different fractions of chalk and its application from the smallest to the largest fraction to ensure filling all the cracks homogenously and thus enabling automatic evaluation in NIS elements software.

Chip length recalculation

For maximal chip length and width projections recalculation from the pixels to micrometres was used based on manual measurements of chip length using digital sliding caliper with resolution of 0,01 mm which resulted in standard deviation of data set std = ± 3,51 %. This deviation could be reduced using more precise optical method on chip length measurement in future experiments.

Innovative methodology approaches

Other innovative methods and approaches could be used in further development of direct wood chip cracks analysis as ultrasonic testing or X-ray tomography which could enable evaluation of not only 2D surface of chip, but rather 3D modelling of whole chip and therefore a progression of cracks. It is important to ensure that used devices have desired resolution as wood chips are rather small inhomogeneous object with dimensions variability. Electron microscopy was found as not favourable method for purpose as wood is an organic material and hence an electron flow could damage an analysed surface of chip.

Pulping limitations

Pulping process was conducted in laboratory conditions. Nevertheless, some aspects of pulping might have influenced results as i.e. washing of pulp, when pulp was washed through screens and even with precise procedure, some fibres might have been lost throughout this process, potentially influencing unscreened and screened yield of pulp. Next limitation was caused by lab method of kappa number measurement resulting in standard deviation at "sharp" knife cut sample SD_{S2} = 2,82 % and for "dull" knife cut sample SD_{S3} = 3,63 % lowering accuracy of measurements and thus results.

7 Conclusion

To conclude, in was proven, that wood chip cracks influence pulping process and thus have an impact on properties of pulp produced as kappa number and unscreened yield of pulp. The increased presence of cracks in wood chips formed by cutting with a dull knife had a positive effect on selected pulp characteristics as kappa number and unscreened yield, resulting in more efficient pulping. Namely, for reaching similar kappa number target $\kappa = 55$, a lower H-factor by -8,16 % in "dull" knife cut sample unlike in sharp knife cut sample was needed, which supports the hypothesis, that wood chip cracks (represented by "dull" knife cut sample) enable faster penetration of cooking liquor and thus fasten up a pulping process. This is confirmed by other finding of unscreened yield at the H-factor target (H-factor = 382), corresponding to kappa number target $\kappa = 55$ (described in explanation related to Chart 5). Unscreened yield was reduced by -1,54 % in "dull" knife cut sample unlike in "sharp" knife cut sample, which is assumed to be caused by better cooking ("delignification") stage. What is more, it was proven that unscreened yield at the desired kappa number target $\kappa = 55$ was positively influenced by increase of 1,50 % in "dull" knife cut sample unlike in "sharp" knife cut sample, leading to conclusion, that "dull" knife cut sample, representing more cracks in wood chips which are an input pulp mill material, has positive impact on pulping process efficiency in the matter of unscreened yield.

A laboratory method for the image analysis of wood chip cracks, which is attached to this study, was developed and it´s relevancy was tested by comparing this method to experimental pulping procedure outputs. Throughout the method it was found that there was a difference in average crack area ratio of "dull" knife cut sample and "sharp" knife cut sample with average crack area ratio being higher in "dull" knife cut sample by 0,79 % and the data was proven statistically significant at a significance level $\alpha = 0,06$. Average length of wood chip cracks was also examined resulting in larger average crack length in "dull" knife cut sample by 3,89 % compared to "sharp" knife cut sample, but result cannot be considered statistically significant at significance level $\alpha = 0,05$. The developed laboratory method is an instruction sheet with precise procedure for potential future use of studying wood chip cracks for both academic and professional interest.

In the new perspective of pulp and paper industry development focusing on maximizing efficiency of pulping process and saving material for reaching economic and ecological targets, understanding of role that cracks in a wood chips play may be crucial

of fine-tuning pulping procedures. This master thesis is just the first step into this problematics and further research in this area needs to be conducted.

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9 List of charts

10 List of tables

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12 List of abbreviations and symbols

AA	active alkali
$Ca(OH)_2$	calcium hydroxide
CS_2	carbon disulphide
CZU	Czech University of Life Sciences
FS	zero-span fibre strength
EA	effective alkali
FSC	Forest Stewardship Council
GM	Grey Model
H-factor	pulp cooking function of time and temperature
HS^-	hydrosulphide anion
ISO	International Organization for Standardization
LWBI	Latewood Blue Intensity
LWSI	Latewood Surface Intensity
NaOH	sodium hydroxide
Na_2O	sodium oxide
Na_2S	sodium sulphate
Na_2CO_3	sodium carbonate
PEFC	Programme for the Endorsement of Forest Certification
pH	potential of hydrogen
px	pixel
RA	residual alkali
ResNet	Residual Neutral Network
RH	relative humidity
SC	shive´s content
SY	screened yield of pulp
TA	Total alkali
TAPPI	Technical Association of the Pulp and Paper Industry
UPCE	University of Pardubice
UY	unscreened yield of pulp
μm	micrometre
°C	degree of Celsius