



On estimating local defects of softwood kraft fibers stained with congo red and assessed with a novel fiber analyzer



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ABSTRACT

The fiber deformations of once-dried, bleached and never-dried unbleached kraft pulps were studied with respect to their behavior in high- and low-consistency refining. The pulps were stained with congo red to experimentally highlight areas where the arrangement of the fibrils was altered by refining such as dislocated zones or slip planes. The stained fibers were analyzed with conventional Metso Fiberlab but also with a novel prototype measurement device utilizing a color imaging setup. The local intensity of the stain in the fiber was expressed as degree of overall damage (Overall fiber damage index, OFDI). The rewetted zero span tensile index (RWZSTI) was used to verify the OFDI with respect to the pulp strength. High consistency refining resulted in a clear increase in the number of kinks which negatively influenced the pulp strength. The OFDI which was used to detect the intensity of local fiber defects also responded accordingly. A higher OFDI resulted in a lower pulp strength. Low consistency refining removed a significant amount of kinks and resulted in an increase in fiber swelling. A slight increase in fibrillation and a significant increase in flake-like fines were also observed. The OFDI, however, was not reduced in low consistency refining as it would be expected by the removal of less severe dislocations. One reason proposed here is that low consistency refining created new fiber pores that allowed the dye to penetrate into the fiber wall similarly as it does in the zones of the dislocations.

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1. Introduction

The mechanical properties of paper are determined by the chemical and the physical properties of the pulp such as the average fiber length, the degree of external fibrillation or the shape of the treated fibers. For example, the digester discharge (Knutsson and Stockman, 1958), the medium consistency mixing (Ellis et al., 1997), the storage at elevated temperatures (Leitner et al., 2013) or the refining (Sjöberg and Höglund, 2005) of pulp introduces local fiber damage and, thus, alters the shape of the fibers and, in turn, the mechanical paper properties. Several authors (Seth, 2006; Mohlin et al., 1996) lately discussed the effects of kinked and curled fibers on the tensile stretch and tensile strength, but little attention was given to the importance of fiber dislocations on the physical pulp and paper properties.

Nyholm et al. (2001) have reviewed the large variety of axially compressed zones in the fiber wall, which are also named as dislocations, microcompressions, slip planes or minute compression failure. Slip planes and minute compression failures were described as small local failures of the fiber wall whereas dislocations are larger zones where the fiber is locally compressed in a range of several micrometers. According to the review of Nyholm et al. (2001), such axial compressed zones were more pronounced and more frequently found in thick-walled latewood tracheids. These local defects originate from the axial compression in the living tree, the axial compression during chipping, the digester discharge, the medium consistency mixing or from the high consistency refining of mechanical and chemical pulps (Nyholm et al., 2001).

Numerous researchers reported the positive effects of fiber dislocations on physical fiber and paper properties such as the tensile stretch (De Grace and Page, 1976; Page, 1966), the tear resistance (Page and Seth, 1980) or the fiber flexibility (Hartler, 1995; Page and Seth, 1980; Hartler and Nyren, 1968). Furthermore dislocations were also considered as favorable for the chemical reactivity of pulp (Hartler, 1995; Page and de Grace, 1967) in operations such as pulp

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washing, bleaching or enzymatic treatment. However, tracheids with large severe dislocations tend to rupture preferentially at the site of the wall failures during low consistency beating (Forgacs, 1961) but less severe dislocations are removed (Kibblewhite, 1976; Zeng et al., 2012).

Mechanical pulp treatments under papermaking conditions also affect the in-plane fiber properties. Many experimental strategies for the measurement of the strength of pulp fibers have been suggested. Among the most important methods are testing of single fibers, and zero span tensile tests.

An extensive amount of single fiber tests with a strong focus on wooden tissue were performed between 1960 and the early 1980's. Although the presence of defects such as natural fiber pits (Page et al., 1972) and dislocations (Hartler and Nyren, 1968) were investigated, back then research was mainly focused on the effect of the microfibril angle (El-Hosseiny and Page, 1975) and the radial position of the fibers in the stem (Mott et al., 2001) or in the year ring (Schniwind, 1966). Single fiber tests are generally very important to discover small-scale defects. However, they are very tedious, expensive and often lack statistical significance.

Dry zero span and especially rewetted zero span tensile tests are considered alternative tools for quantifying significant changes in the strength properties of pulp fibers. However, these methods also do have some drawbacks. The dry zero span tensile strength, for instance, was found to be only marginally affected by the fiber–fiber bonding and the fiber length, and hence may not yield meaningful results in many cases. Limitations of measuring the rewetted zero span tensile index (RWZSTI), on the other hand, include load transfer through the clamps, effects of lateral contraction and load transfer of single fibers (Wathen, 2006). Other limits of the RWZSTI originate from the straightness of the pulp fibers (Mohlin et al., 1996) or from the actual distance of the clamps which was estimated to be 0.1 mm (El-Hosseiny and Bennet, 1985). Despite the numerous difficulties of the zero span technique, the method is widely used in many pulp laboratories for estimating the morphological changes induced by, for example, pulp refining. Since the RWZSTI is considered a good measure for the pulp strength (Mohlin et al., 1996), it was extensively used to study the effects of local fiber defects.

Ellis et al. (1995, 1997) showed that the dislocations in kraft pulp introduced during medium consistency refining in the fiber line had a significantly negative influence on the RWZSTI of paper. Zeng et al. (2012) recently investigated the role of high- and low-consistency refining on the pulp strength. They found that the mechanical treatment resulted in fiber deformations which were reflected in their zero span strength values. Several researchers (Mohlin et al., 1996; Seth and Chan, 1999) also pointed out, that the RWZSTI was to a great extent influenced by the straightness of the fibers. However, results in contradiction with these findings were also published. For instance, it was shown that the curled and straight fibers have the same RWZSTI (Wathen, 2006) and that the structural arrangement of the fibrils as the structural elements of the fibers also affects the fiber properties (Joutsimo, 2004). Due to these ambiguities in the scientific literature the present study was intended to broaden the experimental data basis available for discussing the effect of mechanical treatments on fiber quality and to resolve some of the mentioned contradictions.

Besides the single fiber and zero span tensile tests that provide an indirect measure for the effects of fiber dislocations, several other approaches have been reported in the literature to directly analyze the number and/or the size of dislocations in pulp fibers: Polarized light microscopy (Hakanen and Hartler, 1995), optical light microscopy (Wardrop and Dadswell, 1947), balloon swelling (Ander and Daniel, 2007), enzymatic and acid cleaving (Suchy et al., 2009; Ander and Daniel, 2007) and differential staining (Simons, 1950; Yu et al., 1995). Polarized light microscopy uses the fact that

the locally different fibril angle in the dislocation results in a light-pink and dark red color when the cross polarizer filter is turned from the maximum light absorption of the fiber. Balloon swelling, acid and enzymatic cleaving and differential staining techniques are based on the locally higher chemical reactivity of the cell wall defects.

However, the microscopic techniques are tedious and usually only a comparatively small number of fibers can be studied which poses the potential problems of lack in representativeness of the analyzed sample and sometimes poor reproducibility. Measurements based on differences in the chemical reactivity on the other hand do not always provide the full picture in terms of number and size of the dislocations that would be required for a thorough discussion of the observed effects.

The analysis of dislocations are often indirect, tedious and time consuming measures of great designed studies which have to be made very accurately in order to get a reliable result. Hence such studies are typically rather performed by scientific institutions than by the pulp and paper industry during its daily business. Furthermore, Zeng et al. (2012) recently pointed out that there is yet no reliable method available which quantitatively determines the degree of dislocations. Some attempts to overcome these drawbacks by establishing an automated method to detect the number and size of dislocations have been made by Frölander et al. (1969); Nyström (1995); Thygesen and Ander (2005). However their attempts were not commercialized yet.

In the present contribution, it was focused on the use of stains to evaluate the effects of refining on the sum of the fiber wall defects. The most common approach, the differential Simons staining technique was developed to investigate the nature of refining of chemical pulps (Simons, 1950). The principle of this technique is based on the competitive adsorption of a high-molecular weight dye with a low affinity to cellulose and a second dye with a high affinity to cellulose of low-molecular weight (Yu et al., 1995). The low-molecular weight dye readily penetrates into all native and generated fiber pores by refining whereas the high molecular weight dye is only adsorbed at locally restricted regions of the fiber that are accessible for it. In regions where both large and small pores occur a mixed coloration of the fiber is typically observed. Jayme and Harders-Steinhäuser (1955) proved that the presence of hemi-celluloses on the fiber surface does not influence the staining. Dye adsorption was solely influenced by processing steps, that altered the packing density of the fibers such as chemical pulp refining or drying. Simons' staining was also used to evaluate the homogeneity of the fiber treatment in conventional TMP refining (Fernando and Daniel, 2010), in biomechanical pulping (Blanchette et al., 1992; Behrendt and Blanchette, 1997) or in enzymatic treatment of ligno-cellulosic substrates (Esteghlalian et al., 2001; Chandra et al., 2008). Dadswell and Wardrope (1946) showed that even lignin selective stains colored the dislocations of textile fibers which naturally contain very little lignin. They concluded that the staining is most likely not due to any chemical differences but rather to a physical difference. Other research groups proved that staining of TMP pulp fibers with only Rhodamine (Heinemann, 2008) or staining of softwood kraft fibers with only methylene blue (Zeng et al., 2012) are also feasible approaches to highlight wall disruptions such as dislocations. Green (1962) postulated that staining of bleached pulp fibers with congo red should result in a preferential coloration of the regions which contain cell wall defects. However, this hypothesis was never experimentally properly verified.

In the present study unrefined and industrially beaten, bleached and once-dried softwood kraft pulp and never-dried and unbleached softwood kraft pulp were colored with congo red-stain to study the influence of the sum of the small and the larger cell wall disruptions such as slip planes and dislocations on the pulp strength (RWZSTI). The novelty of the present study is that we have used a

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