Changing Quality of Recycled Fiber Material – Part II. Characterization of the Strength Potential with Fiber Integrity Value and its Relationship with the Strength Properties of Paper

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A novel method was introduced to evaluate the quality of fiber material from paper for recycling. The new concept, fiber integrity value, its components, and its relationship with paper strength properties were examined in more detail. The effect of deinking and screening on fiber integrity value, and its component parameters was shown. The fiber integrity value is closely connected to the strength potential of the pulp. It was shown that when the bonding degree was also considered, there was very good correlation with the tensile strength, tensile stiffness, and compressive strength (SCT) values. The fiber integrity value concept can be determined based on data from in-line analyzers.

Keywords: Recycling; Fiber; Fiber properties; Integrity; Strength; Bonding

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INTRODUCTION

The quality of paper for recycling is changing, and paper mills are often unsatisfied with the reduced quality of recycled fibers. However, recycling and papermaking processes have less effect on the fiber properties than often considered. The changes that individual fibers undergo due to recycling are very modest and mainly related to their bonding ability (Howard 1991; Nguyen 2001). Fibers could tolerate more recycling than what is usual today. For example, the recent statistics from the Confederation of European Paper Industries (CEPI 2016) show that in 2015 the amount of recycled material was 47.7 Mt, and the amount of virgin material was 55.5 Mt. This suggests that on average, approximately 46% of materials used for paper and board production originates from recycling, and the annual input of virgin material is greater than the use of recycled material. Modelling studies to find out how many times the fibers circulate has been performed, but the results largely depend on the assumptions made, *i.e.* the paper grade, the geographical area, exported amounts, and the reliability of the collected data (Schabel and Putz 2005; Keränen and Ervasti 2014, Meinl *et al.* 2016).

It was argued in the previous article (Part 1) that the reduced quality of the fiber material used for recycling is not mainly caused by the degradation of individual fibers in the recycling loop, but due to the inherent blending process, in which different fiber grades and non-fiber components are blended in a non-optimal way. Therefore, the quality of the fiber material should not be assessed based solely on the original paper grade, *i.e.*, by using the list of standard grades of paper for recycling (CEPI 2013), but also by measuring the fiber level properties of the material.

A new concept, 'fiber integrity', was introduced for the evaluation of the fiber material quality in paper for recycling (Keränen and Retulainen 2016). The concept is based on the idea that the strength potential is a fundamental characteristic of any solid material, including paper. Although other characteristics may, in practical applications, be considered more valuable, the ease with which the required strength level is obtained (and being proportional to the strength potential) gives more freedom for optimization of other desired properties once the sufficient strength level is obtained. The fiber integrity value was defined in the previous article according to Eq. 1,

Fiber Integrity Value =
$$(1 - \alpha Filler) * (1 - \beta Fines) * (1 - \gamma Lignin) * L * L/c$$
(1)

where α , β , and γ are the constants, "Filler" is the content of inorganic material (g/g), "Fines" is the fine material content in the cellulosic substance (g/g), lignin is the lignin content of the fibers (g/g), *c* is the fiber coarseness (mg/m), and *L* is the length-weighted projected fiber length (mm).

The value is a relative number and has a unit of m^3/kg . It can mainly be considered as a predictor of the strength potential that is reached when the bonding level of the fiber material is high. Therefore, it can be deduced that the fiber integrity value is connected with the strength of the paper as follows,

$$Strength = (Fiber integrity value) * (Bonding factor)$$
(2)

The objective of this paper is to apply the 'fiber integrity value' concept to actual recycled pulps, and show how the value changes in deinking or screening, and how the value is related to the strength properties of paper made from these pulps.

EXPERIMENTAL

Materials

Recycled paper and pulp were collected from several sources in Europe (Holmen Paper, Madrid, Spain, Papierfabrik Utzenstorf, Utzenstorf, Switzerland, S.C. Vrancart S.A., Adjud, Romania and old newspaper "Keskisuomalainen" collected in Jyväskylä, Finland), and short fiber reference material was obtained from (Metsä Board, Tampere and Powerflute, Kuopio) Finland. To see the changes that take place in the mills, the recycled material was also obtained before and after two-stage flotation deinking, and from the board mill before and after screening. The samples used for testing the fiber integrity method are shown in Table 1.

Methods

Measurement of filler content

The filler content of the pulps or sheets was measured at 525 °C. To evaluate the kaolin/carbonate ratio, the ignition losses at 900 °C were also used. From the ignition losses, an average kaolin/carbonate ratio was evaluated as 47%/53%. This ratio was used only for the correction of measured lignin contents.

Measurement of fiber length, fines content, and coarseness

The fiber length, fines, and coarseness measurements were conducted using the L&W STFI FiberMaster device (Lorentzen & Wettre, Kista, Sweden,). The coarseness of

the pulps was measured using material that remained on 100-mesh wire when wet, and thin 5 g/m^2 to 6 g/m^2 handsheets were formed. The amount of fines was estimated based on the length-weighted value, which is known to overestimate the gravimetric amount of fines.

Table 1. Materials Used in the Experiments

- 1. Recycled pulp from newsprint mill I, before deinking
- 2. Recycled pulp from newsprint mill I, after deinking
- 3. Recycled pulp from newsprint mill II, before deinking
- 4. Recycled pulp from newsprint mill II, after deinking
- 5. Recycled pulp from a board mill, before 1st stage fine screening
- 6. Recycled pulp from a board mill, after fine screening
- 7. ONP (old newspaper ; "Keskisuomalainen" = name of publication),
- 8. Disintegrated birch fluting
- 9. Board grade chemi-thermomechanical pulp (CTMP)

Measurement of lignin content

The pulp samples were air-dried and ground using a Fritsch Pulverisette 14 mill (Fritsch GmbH, Kastl, Germany). The samples were hydrolyzed with sulfuric acid. The Klason lignin content, *i.e.* the insoluble residue from the hydrolysis, was determined gravimetrically. Acid soluble lignin in the hydrolysate was detected at 215 nm and 280 nm using the equation described by Goldschmid (1971). Only 0.2% of the pulp was classified as soluble lignin. The pulps contained undissolved inorganic material, and the Klason lignin fraction also contained filler. Additional tests with kaolin and calcium carbonate fillers revealed that under the same acidic conditions, 89.9% of kaolin and only 1.2% of calcium carbonate remained unsolved. Based on this result, an additional correction was made to the lignin values. The laboratory scale lignin measurement is time-consuming and for intended applications in the mill environment faster methods are needed.

Handsheet making, testing, and data analysis

Handsheets were made according to ISO 5269-1 using conventional rectangular KCL type sheet mold and 350 kPa wet pressing pressure. Methods used in the testing of the pulp and handsheets are listed in Table 2.

Property	Method
Basis Weight	ISO 536 (2012)
Thickness, Density	ISO 534 (2011)
Tensile Strength	ISO 5270 (2012) (at 12 mm/min)
Tear Index	ISO 5270 (2012)
Compressive strength	ISO 9895 (2008)
Ash Content	TAPPI T211 (2002), TAPPI T413 (2011)
Lignin Content	TAPPI T222 (2002)
Fiber Composition	ISO 9184 (1990); Ilvessalo-Pfäffi (1995)

Table 2. Test Methods Used

Standard 60 g/m² handsheets were made from the pulps. In addition to the standard handsheets, some sheets were also pressed at 1800 kPa pressure to give them a higher bonding level.

Microsoft Excel 2010 program (version 14.0.7177.5000 32-bit, Redmond, WA) and its solver was used in calculating, testing and optimizing the parameters in Eq. 1 using GRC non-linear solving method.

RESULTS AND DISCUSSION

Fiber Composition in Certain Pulps

The composition of the recovered paper that mills are using to make new paper can vary considerably. Table 3 shows the fiber analysis results from three mills using paper for recycling. The mechanical pulp content was low (39%) and the chemical pulp content was very high (61%) in the newsprint mill 1, and the chemical pulp fraction contained a high proportion of hardwood fibers. This is far from the mechanical pulp content in the virgin fiber-based newsprint, which is approximately 90% to 100%. This result indicates that the fiber material composition can vary considerably in the incoming material, and it will vary in the newsprint made from it as well. This variation can be high when different geographical areas are compared or samples are taken from the same location during a different season or year. This implies that the paper grade based classification does not properly describe the fiber material content and that there is need for fiber-level characterization. In addition, the fiber material that came to the paperboard mill had a considerable amount of chemical pulp fibers, although semi-chemical fibers had the highest proportion.

Sample	Chemical Hardwood (%)	Chemical Softwood (%)	Mechanical Softwood (%)	Semi- Chemical Hardwood, (%)	Semi- Chemical Softwood, (%)
Newsprint mill 1 after deinking	34	27	39	-	-
Newsprint mill 2 after deinking	23	21	56	-	-
Paperboard mill	13	17	10	11	49

Table 3. Pulp Composition

Applying Integrity Value for Pulps; Effect of Deinking and Screening

The fiber integrity value was calculated for the nine pulps shown in Table 1. Given that constant parameter values (α , β , and γ) are needed for the equation, rough estimates for the parameters were obtained from previous literature. The values were based on how the variables (filler, fines, and lignin content) affect the tensile strength. The first approximations for the constants were: $\alpha = 1.9$, $\beta = 1.4$, and $\gamma = 1.2$. These

values were obtained from the experimental data in the literature (Page *et al.* 1985; Rundlöf 2002; Velho 2002).

The variables were assumed to linearly affect the relative value of the tensile strength, which was initially set to one. Linear equations were constructed and the constant values were obtained from the slope of the line.

The calculated fiber integrity values ranged from 1.5 to 3.5 and are shown in Fig. 1. Birch fluting and board-grade CTMP were almost on the same level as paperboard mill pulp before screening, with a value of 3. Screening increased the fiber integrity value by 20% for the paperboard mill to 3.6. In newsprint mill 1, deinking increased the fiber integrity value by 59%, from a relatively low level of 1.6 to 2.6. In newsprint mill 2, the starting level was higher, and the increase was more modest, 14% from 2.1 to 2.4. In practical applications, the exact numeric value may not be so important; it is more important to focus on the changes that occur during deinking, and to look at the integrity values changes in reject and accept fractions.



Fig. 1. Fiber integrity values for different pulps

When closely examining which factors were behind the observed effects of deinking and screening, consider Table 4. The fiber integrity value increased as substances such as filler were removed. There was very little change in the lignin content, which suggested that the ratio between the chemical and mechanical pulp fibers did not change. The amount of fines was reduced, as well as the coarseness of the fiber. This may have been an indication that the fiber coarseness was reduced due to fines creation. Deinking, or actually the kneading unit in the deinking plant, was the probable cause for increasing the curl (McKinney and Roberts 1997). The curl decreased the effective fiber length.

Fiber Integrity Value and Strength Properties

The fiber integrity value itself is not useful unless it can also be linked to the properties of the end-product. It seemed that the value alone does not necessarily correlate with the paper tensile strength, as shown in Fig. 2. To see the connection, as shown in Eq. 2, an estimate of the bonding factor is needed.

Pulp/Mill	Filler (%)	Lignin (%)	Fines (%)	Fiber Length (mm)	Coarseness (mg/m)	Curl (%)
Newsprint mill 1, before deinking	38.6	14.8	22.8	1.17	0.24	15.5
Newsprint mill 1, after deinking	18.0	15.3	19.2	1.14	0.19	16.1
Newsprint mill 2, before deinking	26.1	13.1	19.0	1.10	0.21	14.6
Newsprint mill 2, after deinking	15.1	14.5	19.2	1.10	0.19	17.8
Paperboard mill, before screening	17.4	12.0	15.7	1.18	0.19	13.8
Paperboard mill, after screening	13.2	11.2	14.8	1.23	0.19	13.8

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Fig. 2. Correlation between the fiber integrity value and tensile strength

It will be assumed here that the specific bond strength of recycled fiber material is roughly constant and that the bonding is mainly dependent upon the relative bonded area of the fibers (RBA). From previous literature (Retulainen and Ebeling 1993), the authors could assume that the bonded area is a linear function of the density of the fiber network, at least in the density range relevant to paper applications. An estimate for RBA can be obtained from Eq. 3,

$$RBA = k \left(\rho_{\rm f} - 0.3 \right) \tag{3}$$

where k is constant (cm³/g), and ρ_f is the apparent density of the fiber network excluding the filler (g/cm³). When plotting the strength values as a function of the product of the fiber integrity value and RBA, reasonable correlations were obtained. The correlations with the strength properties are shown in Fig. 3.

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Fig. 3. Correlation of certain strength values (tensile, SCT, and tear indexes) and the fiber integrity value (multiplied by a term describing the bonding); Constants used were $\alpha = 1.9$; $\beta = 1.4$, and $\gamma = 1.2$.



Fig. 4. Correlation between measured strength values and the fiber integrity value multiplied by a bonding factor; the fitted values used for the parameters were $\alpha = 0.90$, $\beta = 1.59$, and $\gamma = 0$.

There seemed to be a good correlation with the tensile strength and compression strength, and a clearly lower correlation with the tear strength. The constant parameters that were estimated from the previous literature data, presumably were not exactly correct, because the correct parameter values also depend on the strength property, experimental conditions, and the methods used in determining the data for the variables. An optimization process was performed using Excel solver to find the best parameter values. The result obtained when the best explanation percentage for tensile strength was sought also clearly gave a higher coefficient of determination for SCT and tensile stiffness, but not for the tear strength (Fig. 4.). The optimized positive parameter values obtained were ($\alpha = 0.90$, $\beta = 1.59$, and $\gamma = 0$). The optimised parameter values would depend on the property predicted but here values were optimised only regarding tensile strength. The parameter obtained for lignin ($\gamma = 0$) suggested that the role of the lignin in this experimental material was not important for the fiber integrity value, and the role of lignin came through the bonding factor (fiber flexibility and bonding ability that affect the RBA). This further indicated that the lignin content may not necessarily have to be determined to obtain a relevant fiber integrity value, which would make the practical use of the equation easier.

Estimating the Explanation Potential of the Integrity Equation and its Components

The role of individual factors was examined more closely by examining the different terms that explain certain strength properties (Table 5. The sheet density alone already gave a high positive correlation with the tensile strength ($R^2 = 0.66$), which was largely due to the effect of the bonded area. Clearly a better prediction ($R^2 = 0.77$) was obtained when the density value used was the apparent fiber network density value, in which the effect of the filler was subtracted. This suggests that the role of the fiber contact areas dominate the bonding effect, and there is a rather limited variation in the specific bond strength.

By adding fiber length and coarseness to the explanatory terms, the coefficient of determination (\mathbb{R}^2) for tensile strength increased to 0.83. Also, the correlations with SCT and especially with the tensile stiffness were increased, but the tear strength was not explained so well ($\mathbb{R}^2 = 0.43$). When the parameters α , β , and γ were fitted with regards to the tensile strength, the \mathbb{R}^2 for tensile and SCT strength increased to 0.88 and 0.85, respectively. However, the correlation for tear strength decreased ($\mathbb{R}^2 = 0.33$), which implied that the relationship was not linear. The fitting gave the following values for α , β , and γ : 0.90, 1.59, and 0, respectively. This result suggests that the role of the lignin content was considered by the bonding factor, whereas the fines and filler content both had a major contribution to the fiber integrity value. This simplifies the measurements needed at the mill, because of the lack of an established, reliable inline method for monitoring the lignin content.

If the effect of fibers was not assumed to be proportional to an area (L^2) , but only to length (L) of fibers, somewhat better R² values were obtained for tensile and SCT, when same constant values were used as earlier. By fitting the parameters to the tensile strength data, the correlations were actually not improved, except for tensile stiffness and the tear strength. The R² values for tensile, tensile stiffness, SCT, and tear strengths were 0.91, 0.69, 0.88, and 0.27, respectively, when the fitted parameters α , β , and γ were 0.07, 1.79, and 0, respectively. This suggests that the prediction of tensile strength, tensile stiffness, and compression strength is quite straight-forward. The energy-based values are more complex, *e.g.* tear (or probably also fracture strength), since they are known to exhibit an optimum as a function of RBA as shown in Part 1. A low value for the filler parameter ($\alpha = 0.07$) also suggests that the role of filler in the network strength aspect may have already been partly considered by the fines term or bonding term ($\rho_f - 0.3$). Generally, this material suggests that the use of the term 'L' instead of 'L²' may in certain cases be justified.

	Tensile	Tensile	SCT	Tear	
	Index	Stiff. Index	Index	Index	
Explanatory term	R ²	R ²	R ²	R ²	
ρ	0.66	0.45	0.58	0.33	
ρ _f - 0.3	0.77	0.48	0.71	0.31	
L ² /c *(<i>p</i> _f - 0.3)	0.83	0.63	0.77	0.43	
(1-0.90 Filler)(1-1.59 Fines) *L ² /c *(p _f - 0.3)	0.88	0.69	0.85	0.33	
L/c *(p _f -0.3)	0.86	0.61	0.81	0.35	
(1-0.9 Filler)(1-1.59 Fines) *L/c *(p _f - 0.3)	0.91	0.66	0.88	0.26	
(1-0.07 Filler)(1-1.79 Fines) *L/c *(p _f - 0.3)	0.91	0.69	0.88	0.27	
$ ho$ sheet density, $ ho_{ m f}$ apparent fiber network density without filler					

Table 5. Coefficients of Determination	(R ²) for Different Components	of Fiber
Integrity Value		

How the Information can be Utilized

The fiber integrity value and the proposed approach offers a tool to understand the strength potential, and separate the effects of fiber dimensions, fines, filler, and bonding ability (density) on the selected strength properties. This has not been done to this extent previously. It offers opportunities to evaluate the quality of paper for recycling, to characterize the changes in the quality of incoming materials at the paper mill, and it can be used for evaluating the effect of mill processes on the quality. The sensitivity of the fiber integrity value could also be tuned for different end-product properties. The approach also offers new insights into how the overall fiber recycling and fiber material use should be re-evaluated.

The approach showed that the integrity value had an important role in the strength potential of the furnishes. However, the role of the strength potential of fibers may not always be recognized so well because a lower strength potential in many cases is compensated by increased sheet density. The ongoing trends to reduce the grammage or increase the bulk implies the need for decreasing the sheet density and simultaneously maintaining or increasing the network strength and stiffness. This is possible only when the fiber integrity value is high.

Because the fiber integrity value is reduced with recycling, it can be deduced that the raw material with a high fiber integrity value should first be used for making bulkier products. As the fibers will be recycled more and the strength potential and integrity value decreased, the fibers could be used in applications in which the reduced strength potential can be compensated by making denser structures.

The developed fiber integrity value for recycled fiber material was constructed using only a few variables. Additionally, in certain applications, the equation may be possible to simplify further. Values for the needed variables can basically be obtained from fiber analyzers or other existing inline analyzers. If the lignin/cellulose content of the material is also determined, it possibly offers additional information, but reasonable results may be obtained without lignin determination.

The fiber integrity value seemed to reasonably reflect changes in the quality of the pulps. The fiber integrity value of pulps was not dependent on the bonding ability of fibers (and resulting bonded area of the sheets). The fiber integrity values measured showed that deinking increased the value. Fiber integrity was also greater when using fresh, non-recycled pulps. However, the integrity concept is targeted for the recycled material, although with adjusting the constants, its application range could be widened, but it has not been studied yet.

The fiber integrity concept is planned to reflect the strength potential of the pulp, and it alone did not directly predict the paper properties. However, fiber integrity value was shown to correlate well with some strength properties measured from the handsheets when the value was combined with a fiber network density that represented the bonding ability of the pulp. Therefore, the fiber integrity value could also have potential as a continuous tool to monitor the incoming recycled material at the mill.

The results (Table 5) showed that the fiber network density, *i.e.*, bonded area, explained several strength properties. Of course, the specific bond strength also had a role, but here in this material, and perhaps it is characteristic for recycled fiber material, the variation in bond strength per area seemed to be of lower importance. This may be because in recycling the fiber surfaces become hornified, collect impurities and lose their bonding ability on molecular level.

The fiber integrity value concept does not contain variables, such as stickies or ink content, although they can have a considerable effect on the usability of the paper for recycling. Additionally, because the value is focused on the strength potential, it does not have any connection with the optical properties of the pulps. Other factors are needed for predicting them.

Application of Integrity Value with Side Streams

In addition to the characterization of the fiber material for papermaking, the fiber integrity value could also be utilized for side streams, which gave an additional option to evaluate the side stream potential in a fast and cost-efficient way.

When the potential of reject fractions was considered, the use of fiber integrity value became more important, due to necessary process optimization to prevent unnecessary loss of fibrous material in the main stream. For side streams, the initial potential shown by the fiber integrity value should be low, but it may also have been optimized when reject fractions were used for novel materials. In some cases, loss of certain fiber fractions in the main stream may have been justified if the side stream could then be utilized in another product with sufficient economic value.

CONCLUSIONS

1. A new concept, fiber integrity value, proposed in the Part 1 was here tested for the evaluation of the quality of recycled fiber material.

- 2. The fiber integrity value offered information on the strength potential of the fiber material. When combined with fiber network density or RBA, it correlated well with several strength properties of paper.
- 3. Fiber integrity value was improved by deinking and screening.
- 4. Fiber integrity value can potentially be measured using in-line analyzers and used for evaluating the quality of recycled paper, material entering the mill, and for monitoring and controlling mill processes.
- 5. The concept when differentiating the strength potential and fiber network density can give new insights how the paper cycle, and the recycling steps of fiber- material should be optimized.
- 6. Fiber integrity concept can also give guidance for side stream management. Fiber use in the side streams product could provide additional value for the mill if only a fraction of fibrous material is needed in side stream products.

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