

High-Performance Sustainable Tissue Paper from Agricultural Residue: A Case Study on Fique Fibers from Colombia

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Abstract

Global sustainability megatrends are promoting the utilization of sustainably perceived fibers such as recycled and agricultural residue fibers in hygiene tissue applications. Tissue paper products advertised as sustainable have higher prices and inferior performance than conventional products manufactured from virgin wood fibers. This work demonstrates the feasibility of using agricultural residues from fique plantations (*Furcraea microphylla* genus) as an alternative to Northern Bleached Softwood Fibers (NBSK) in high-performance hygiene tissue applications. For our study, fiber residues were mechanically cleaned and upgraded to a tissue pulp using a simple pulping and bleaching process. A complete characterization of tissue paper properties (bulk, softness, water absorbency, tensile strength) was performed and compared against the NBSK market pulp. Additionally, fique residue pulp was blended with Bleached Eucalyptus Kraft (BEK) to match the performance of a selected benchmark consisting of 70% BEK and 30% NBSK. Results indicate fique residue bleached pulp has similar fiber morphology and comparable strength properties in terms of the tensile strength (+6%) and tear strength (+10%), but superior bulk (+12%), water absorbency (+28%), and softness (-29 % TS7 values) than NBSK pulp. A fiber blend of 70% BEK and 30% fique residue showed superior tensile strength (+21%), tear strength (+54%), bulk (+ 5.5%), water absorbency (+1.5%), and softness (-8.7% TS7 values) over a similar fiber blend of BEK and NBSK. Our findings demonstrate that fibers from fique residue can substitute NBSK in hygiene tissue applications. Upgrading residues from fique fibers as raw materials for the tissue industry can bridge the gap between sustainability and product performance, simultaneously opening the possibility of new revenue streams for millions of small farmers in the producing countries.

1. Introduction

Global paper production has been steadily increasing over the years and reached approximately 425 million tons per year in 2020 (FAO - Forestry Production and Trade 2020). Hygiene tissue papers are one of the fastest-growing categories of all paper products witnessing a CAGR (compound annual growth rate) of 2.0% between the years 2015-20 (FAO - Forestry Production and Trade 2020). Within the broad spectrum of hygiene tissue paper, a wide range of products such as bath tissue, kitchen towels, wipes, facial tissue, napkins are designed to serve the specific needs of consumers. For example, kitchen towels have excellent strength and water absorbency, while bath/facial tissue should have softness and strength. Overall, water absorbency, softness, and strength are the most critical properties of tissue paper products (Wrap 2005; Gigac and Fišerová 2008). By choosing a suitable combination of fiber mix, paper machine technology, and chemical additives, tissue papers can be produced to achieve the desired performance as required for any specific application (de Assis et al. 2019; Zambrano et al. 2020).

Fiber morphology plays an important role in tissue paper performance. High-value premium tissue papers are predominantly manufactured using a fiber mix from virgin wood fibers of softwoods and hardwoods. Tissue products manufactured from virgin wood fibers account for more than 60% of the total market share, while products made from recycled fibers and other non-wood alternative fibers constituent the remaining fractions (Fisher International 2021). NBSK (Northern bleached softwood kraft) and BEK

(Bleached eucalyptus kraft) are the two most important raw materials for the tissue industry, representing approximately 45% of the total market share of pulps (Fisher International 2021). Long and thin fibers of northern softwoods are added to provide superior strength properties, whereas shorter, low coarseness fibers of BEK provide good softness properties (Gonzalez et al. 2020). Thus, an optimum mix of NBSK and BEK provides tissue paper with a superior combination of softness and water absorbency at a given strength (De Assis et al. 2018; de Assis et al. 2019).

However, the exclusive use of virgin wood fibers for making premium tissue products puts tremendous strains on already scarce natural forests. Substantial environmental concerns have been raised over the logging of NBSK from Canada, a key raw material in tissue furnish (Skene and Vinyard 2019; Vinyard and Skene 2020). Global sustainability megatrends are forcing manufacturers to look for alternatives beyond virgin wood fibers (Thomas and Liu 2013). This has motivated tissue makers to utilize sustainably perceived fibers such as post-consumer recycled fibers and agricultural residue fibers (Vinyard and Skene 2020). Post-consumer recycled paper as a raw material for tissue paper is certainly a sustainable option as it diverts the waste from entering landfills and the manufacturing process has lower environmental footprints than using virgin fibers (Wrap 2005; Skene and Vinyard 2019; Vinyard and Skene 2020). However, the high content of short fibers, fines, and other foreign matters (fillers, ink, and stickies) typically present in recycled pulp may have a detrimental effect on tissue properties (Hubbe et al. 2007). In addition, recycled fibers are stiffer with limited fiber swellability and wet flexibility (Hubbe et al. 2007). Hence, tissue papers produced with higher content of recycled fibers are weaker, stiffer, denser with lower softness and water absorbency (Wrap 2005; De Assis et al. 2018; de Assis et al. 2019). Shortcomings associated with recycled fibers limit their use to mainly economy and value-grade tissue products and are rarely used in premium or ultra-premium tissue products (Wrap 2005; De Assis et al. 2018).

In addition to recycled fibers, several non-wood alternative fibers have been researched as a source of raw material to produce tissue papers (Byrd and Hurter 2013; de Assis et al. 2019). Typically, non-wood fibers can be classified into agricultural residue and fiber crops (Hurter 2001; Byrd and Hurter 2013). Agricultural residue fibers such as wheat straw, corn stalks, and sugar cane bagasse present an interesting case from an economic and sustainability standpoint. Primary crops can account for most of the cost and environmental burden associated with the cultivation and harvesting of agricultural residue fibers (Byrd and Hurter 2013). Additionally, the utilization of agricultural residue in the paper industry offers an economical solution to the waste disposal problem as most of the leftover straw residues are burnt off in the field in the absence of any suitable disposal solutions (Byrd and Hurter 2013; Skene and Vinyard 2019). However, prior efforts to utilize agricultural residue fibers in tissue paper have yielded limited results in terms of product performance. De Assis et al. (2019) compared the performance of bleached and semi-bleached wheat straw soda pulp with commonly used virgin wood pulps in tissue paper manufacturing and concluded that wheat straw pulps had remarkably higher fines content, which resulted in lower freeness of the pulp and increased densification of the tissue handsheets made thereof (de Assis et al. 2019). Increased sheet density had detrimental effects on important tissue paper properties. Wheat straw pulp had the worst combination of softness, water absorbency, and tensile

strength among all market pulps evaluated in this study and was found only suitable for making economy-grade tissue products (de Assis et al. 2019).

On the other hand, fiber crops such as cotton, flax, hemp, jute, kenaf, abaca, sisal, and fique are cultivated specifically to yield fibers. Papermaking pulps obtained from these fibers have extremely high tear and high tensile strength beyond what can be achieved with premium wood pulps (Judt 1993). However, pulps from these fibers are mainly utilized for making specialty paper products that command a premium in the market over commodity paper products (Judt 1993; Atchinsons 1998). Some limited efforts to utilize these fibers in hygiene tissue application have given positive results in the past. For example, patent US5320710 assigned to James River Corporation describes utilizing low coarseness and longer fiber length of chemically pulped hesperaloe fibers to produce tissue papers with improved bulk, water absorbency, and softness compared to those resulting from similar fiber blends of softwood and hardwood pulps (Reeves and Plantikow 1994). Similarly, Hermans et al. (1997) from Kimberly-Clark Corporation claims to use low coarseness fibers of abaca, paper mulberry, or pineapple leaf fibers to improve the softness of tissue paper products by introducing them in fiber blends of softwood and hardwood fibers (Hermans and Sauer 1997). Nevertheless, the high cost of market pulps from these non-wood fibers prohibits their utilization in tissue paper applications.

The standard processing technique employed to extract fibers from the fiber crops also produces a significant quantity of by-products or waste along with the primary fibers. These by-products are trimmed waste, which has similar characteristics as primary fibers but is mixed with foreign particles, soils, parenchyma cells, and inorganic impurities (Ovalle-Serrano et al. 2018b; Lee et al. 2020). For example, the processing of bast fibers such as flax, hemp, and jute produces a series of by-products such as shives and tows along with the primary bast fibers (Michael Carus 2017). Similarly, mechanical decortication of leaf-based fibers such as abaca, sisal, henequen, and fique generates short and entangled tow fibers as by-products (Ovalle-Serrano et al. 2018a). Such residues are either sold as low-value products or left on the field without any market value (Michael Carus 2017; Ovalle-Serrano et al. 2018b). Considering these by-products comprise the major yield of fiber processing, these residues present an interesting case as low-cost biomass for valorization into value-added products.

In this context, the objective of this work was to study the feasibility of upgrading residues from the processing of fiber crops as a substitute fiber to Northern Bleached Softwood Fibers (NBSK) for high-performance hygiene tissue applications. Future forecasts point to uncertainties over the long-term supply and prices of NBSK fibers and require the tissue industry to explore developing alternative fiber sources to complement the currently used fiber mix (Thomas and Liu 2013). To achieve this objective, the residue obtained from the decortication process of the fique fiber was selected as a raw material for our study. Fique is a leaf-based natural fiber similar to abaca and sisal, which is primarily utilized for the fabrication of coffee sacks in Colombia. Fique residues are a by-product of the decortication process that produces 4-5% primary long fibers (used for making coffee sacks) and 8-10% short tow fibers as a residue (Ovalle-Serrano et al. 2018a). These residues are left on the field without any market value. To the best of the author's knowledge, no prior work on upgrading residue from the processing of bast and leaf-

based fibers for hygiene tissue applications has been reported in the literature. Considering that these residues are currently left on the field without any market value, their upgradation into an alternative raw material for tissue manufacturing offers a unique opportunity to utilize these fibers into value-added products. This has the potential of opening new revenue streams for millions of small farmers in the producing countries, simultaneously easing the supply of imported virgin softwood fibers into the country.

2. Materials And Methods

2.1. Materials

Compania de Empaques S.A., Medellin, Colombia, provided cleaned and uncleaned samples of fique residue fibers. Uncleaned fique residue is a decortication waste and contains a mixture of short fibers, parenchyma tissue, and other inorganic impurities such as soil, dirt, and sand. Uncleaned residue from the field was mechanically cleaned using a fiber carding line and slotted screens to separate the fibers from the parenchyma tissues and other remnants. Market pulps of northern bleached softwood kraft (NBSK) and bleached eucalyptus kraft (BEK) were sourced from pulp manufacturers in a dry sheet form. Chemical reagents used for pulping, bleaching, and pulp characterization were purchased from Sigma-Aldrich.

2.2. Methods

2.2.1. Pulping and bleaching process

Soda pulping of cleaned and uncleaned fique residue fibers was carried out in an air-heated tumbling bomb digester. Following fiber loading, the bomb was charged with cooking liquor made of sodium hydroxide (NaOH) and water to obtain a liquor to wood ratio of 10:1. Bombs were loaded into the slots of the tumbling digester preheated to a pulping temperature of 170°C. The temperature inside the bomb was raised to 170°C in the first 75 minutes, following which cooking was carried out for further 60 minutes. After cooking, bombs were cooled by immersing into a cold-water reservoir for 15-20 minutes. Pressure in the bomb was relieved, and pulped fibers were transferred into a perforated basket for washing with tap water. Following washing, the fiber sample was passed through a laboratory disc refiner for complete disintegration before passing through a 0.15 mm slotted laboratory screen to remove uncooked fibers as rejects. Subsequently, the screened pulp was centrifuged, fluffed, and stored for further processing.

Three-stage elemental chlorine free (ECF) bleaching sequence ($D_0E_pD_1$) of the unbleached soda pulp was performed as per the process described by Danielewicz and Surma-Slusarska (2017) for hemp bast fibers (Danielewicz and Surma-ślusarska 2017). First, 120 o.d. (oven-dried) grams of unbleached pulp was placed inside a polyethylene bag and charged with a ClO_2 (chlorine dioxide) solution and deionized water to obtain a consistency of 10%. Kappa factor of 0.2 (% chlorine per unit of kappa number per o.d. pulp) and oxidizing equivalent ratio of 2.63 were used to calculate the total amount of ClO_2 required in the

bleaching process. 65% of the total calculated ClO_2 was charged into the D_0 stage, and the remaining 35% was charged into the D_1 stage. No pH adjustment was made in the D_1 stage, while NaOH was added at the D_1 stage to obtain the final pH in the range of 4 to 5. In between the D_0 and D_1 stages, the pulp was treated in a hydrogen peroxide-reinforced alkali extraction stage (E_p). After charging with bleaching reagents, the polyethylene bag was sealed, kneaded with hands, preheated in a microwave for one minute, and placed in a water bath set at a constant temperature of 70°C. Retention time at D_0 , E_p , and D_1 stages was 60, 90, and 60 minutes respectively. The resulting properties of the final fique residue bleached pulp have been summarized in Table 1. This pulp was used for making tissue handsheets, as described below.

2.2.2. Characterization of pulp samples

Following soda pulping of the fibrous raw material, pulp samples were characterized in terms of the pulping yield (percentage), kappa number (Tappi T 236 om-99 2006), cupriethylenediamine (CED) pulp viscosity (Tappi T 230 om-08 2008), ISO brightness (ISO 2470-1 2016), and pulp freeness (TAPPI T 227 om-09). Following the ECF bleaching process, pulp samples were characterized for bleaching yield (%) and ISO brightness (ISO 2470-1 2016). Pulping and bleaching yields were estimated gravimetrically by comparing samples' oven dried weight before and after the treatment process. After initial characterization of the pulps, morphological properties such as fiber length, width, coarseness, and fines content were measured using an optical fiber quality analyzer (HiRes FQA – OpTest Equipment Inc.).

2.2.3. Handsheets preparation

Tissue handsheets of basis weight 30 g/m² were prepared from unrefined and mechanically refined pulp samples. A lab-scale PFI mill refiner (PFI Mill – N°312, The Norwegian Pulp and Paper Research Institute, Oslo, Norway) was used to refine the pulp samples according to Tappi standard T248 sp-00 (2000). The refining energy applied to the pulp samples is evaluated in terms of the number of PFI revolutions, with higher PFI revolution indicating a higher refining energy. Since tissue products are produced using lightly refined fibers, only 1000 and 2000 PFI revolutions were applied to refine the pulp fibers. Following refining of the pulp, tissue handsheets were prepared using a modified version of the Tappi standard T 205 sp-02 (2006) to mimic properties of tissue papers as previously described by de Assis et al. (2019). For forming handsheets, 24 grams of oven-dried pulp was disintegrated at 1.2% consistency in a standard pulp disintegrator, and additional water was added to dilute the pulp slurry to 0.3% consistency. Freeness of the pulp (Canadian Standard Freeness) was measured according to Tappi T 227 om-99 (1999) before making the handsheets. Tissue handsheets were formed in a standard handsheet former. The wet handsheets thus formed were not subjected to any further mechanical pressing to avoid densification and loss of bulkiness. Wet handsheets were dried using a drum dryer preheated at 110°C. The dried handsheets were then conditioned according to Tappi conditioning standard Tappi T 402 sp-98 (1998) at 23°C ambient temperature and 50% relative humidity before measuring the final properties.

2.2.4. Handsheet properties measurement

Paper properties most relevant for tissue papers were measured using the conditioned handsheets. The average value of at least five measurements was taken for tensile strength, tear strength, zero span tensile strength, and softness. The average value of two measurements was taken for water absorbency, and the average value of 15 handsheets was taken for bulk/density. The basis weight, defined as mass per unit of the surface area of the handsheets, was measured according to ISO 12625-6 (2005). The bulk, which is the inverse of the apparent density, was measured following (ISO 12625-3 2014). The tensile strength was measured according to ISO standard (ISO 12625-4 2005) using a universal INSTRON tensile tester (Instron model 4443, Canton, MA.). Tear strength (Elmendorf type) was measured according to TAPPI T 414 om-98 (1998). Zero span tensile strength, which is an indicator of the strength of individual fibers, was measured according to Tappi T 231 cm-96 (1996) using a Pulmac zero span tester (Middlesex, VT). The water absorption capacity per unit mass of fiber was measured according to ISO 12625-8 (2016) using the basket immersion test method. The softness of the handsheets was measured with a Tissue Softness Analyser (TSA Softness), equipment manufactured by Emtec Electronic GmbH, Leipzig, Germany. The softness evaluation is based on measuring the surface and bulk softness independently using three basic parameters: TS7 (surface softness), TS750 (surface smoothness/roughness), and in-plane flexibility (bulk softness) (Wang et al. 2019). TS7 mimics the human perception of surface softness by rolling a rotor over the surface of tissue handsheets and measures the amplitude of vibrations generated by the rotor due to the number of free fiber ends present on the surface of tissue paper. The rotating lamella records a lower vibration amplitude if it crosses over a large number of flexible fiber ends and corresponds to a better softness. TS750 measures the vibration of tissue samples and correlates to the smoothness/roughness of the surface structure. A higher peak of TS750 indicates lower smoothness or higher surface roughness. The equipment also measures the stiffness or the in-plane flexibility, which correlates to the bulk softness, by applying a fixed load to the tissue sample in the vertical direction.

2.2.5. SEM imaging of the handsheets

Surface and cross-section micrographs of the handsheets were taken using a variable pressure scanning electron microscope (VPSEM Hitachi S3200 N, Hitachi High Technologies America, Schaumburg, IL). Handsheet samples were cooled under liquid nitrogen and cut using sharpened edge blades. The samples were sputter-coated with a thin layer of gold-palladium (~35 nm).

Comparison of fique residue bleached pulp with NBSK fibers as a potential reinforcement fiber in hygiene tissue products

In order to evaluate the suitability of fibers from the fique residue in hygiene tissue applications, a comparison was made against NBSK fibers. The comparison with the NBSK fibers was made in two stages, as shown schematically in Figure 1. In the first stage (referred to as direct fiber-to-fiber comparison), tissue handsheets were prepared using 100% bleached fique residue pulps and 100% NBSK market pulps, respectively. Physical properties of handsheets relevant to tissue papers as previously described were measured and compared against each other. In the second stage (comparison of tissue properties), unrefined bleached eucalyptus pulp (BEK) was blended with refined NBSK (refined at 1000

PFI revolutions) in different weight ratios to form the tissue handsheets similar to the traditional practice in the industry (base case). In the alternate case, NBSK was replaced with the fique residue pulp. Tissue handsheets were prepared by blending unrefined BEK with the refined bleached fique residue pulps (refined at 1000 PFI or 2000 PFI revolutions). The resulting tissue paper's properties in the alternate case were compared against the base case tissue papers made with the pulp blends of BEK and NBSK.

3. Results And Discussion

3.1. Comparison of fiber morphology

Diluted and fully disintegrated pulp samples of bleached fique residue, NBSK, and BEK were analyzed in terms of their fiber length, width, fines content, and fiber coarseness. Fiber population in terms of millions of fibers per gram of pulp was calculated from the total number of fibers and the total mass of pulp samples used in the FQA analysis. Table 2 shows fiber morphology data as obtained from FQA for all the pulp samples. Fique residue bleached pulps and NBSK market pulps had remarkably similar fiber morphology. The average fiber length of fique residue bleached pulp (~ 2.3 mm) was comparable to the NBSK market pulp (~2.4 mm). Fiber length is one of the most critical parameters for reinforcing pulps as longer fibers have the ability to form inter-fiber bonds with multiple fibers and provide superior strength to the tissue paper (De Assis et al. 2018). Moreover, fique residue bleached pulp had lower fiber width, fiber coarseness, and lesser fines content than the NBSK market pulp. These properties of fique residue bleached pulp are more conducive to developing favorable tissue paper's properties than the NBSK market pulp. Due to the combination of comparable fiber length and narrower fiber width, fibers from fique residue bleached pulps had a higher aspect ratio (length/width). Slightly smaller fiber length and lower fiber coarseness resulted in fique residue bleached having a higher fiber population (~10% higher) than NBSK market pulp.

In addition to the average values, the distribution of fiber length and width are also shown in Figure 2. Compared to NBSK market pulp which is likely manufactured from tress with different ages and species, fique residue pulp was prepared from a single plant species of similar age. As a result, fique residue bleached pulp had a narrower distribution of fiber length and width. A narrower distribution of fiber morphology improves product uniformity and gives better control on achieving final properties (de Assis et al. 2019).

3.2. Effect of refining on fiber properties

A lab-scale PFI refiner was used to refine the pulps in this study. The compressive and shear forces applied during the PFI refining process produces several changes in the fiber structure, such as external fibrillation on the fiber surface, internal fibrillation (delamination of cell wall layers and changes in the internal structure of cell wall), fiber shortening, and fines generation (Page 1989). The relative predominance of these refining effects depends on the physical and chemical properties of fibers. Thus different fibers respond differently to refining (Page 1989; Gharekhani et al. 2015). The cumulative effect of these changes in the fibers' structure can be observed by measuring changes in pulp freeness,

apparent density, light scattering coefficients, and strength properties of the final paper web (Wang et al. 2005, 2007; Kang and Paulapuro 2006). In this study, the effect of refining on fiber properties was evaluated by measuring the freeness of the pulp and bulk (inverse of the apparent density) of the final tissue handsheet. In addition, changes in the fiber's internal structure were investigated using SEM (scanning electron microscopy) micrographs.

Figure 3 shows SEM micrographs of the surface and cross-section of tissue handsheets made with unrefined and refined fibers of bleached fique residue and NBSK pulp. Lower resolution images of the surface of the tissue samples present in the first column (Figures 3a, 3d, 3g, 3j) are used to evaluate the network structure of fibers. Higher-resolution surface micrographs present in the second column (Figures 3b, 3e, 3h, 3k) are used to compare any changes in the internal structure of fibers before and after refining. Micrographs present in the third column (Figures 3c, 3f, 3i, 3l) represent the cross-section of the tissue samples and are used to evaluate the collapsibility of the fibers lumen and relative consolidation (loss of bulkiness) of the fibrous assembly with the refining process. Overall, refining consolidated the paper web structure and reduced the pore size and pore volume of both pulps. However, it can be observed that fique residue bleached pulp formed a more open, porous, and bulkier paper structure than NBSK pulp in both unrefined and refined fiber states. The cell wall of NBSK fiber completely collapsed into a flat ribbon structure with refining (Figures 3k and 3l). In contrast, some fibers in fique residue pulp can be observed to retain their original cylindrical shape and intact fiber lumen (Figure 3i). Moreover, some sporadic external fiber fibrillation was observed on the outer surface of refined fique residue pulp, while the outer surface of NBSK pulp fibers was relatively cleaner and smoother. Overall, the cell walls of NBSK pulp fibers completely collapsed and seemed to favor more internal fibrillation and negligible external fibrillation with refining. Cell walls of fique residue pulp did collapse with refining; however, they were more resistant to the internal fibrillation than NBSK pulp and favored a certain degree of sporadic external fibrillation. The milder conditions of refining conducted in this study did not generate secondary fines or reduce the fiber length significantly.

To further investigate the effect of refining on the properties of the fibers and corroborate the findings from SEM micrographs, changes in freeness and bulk of the tissue paper were measured for fique residue and NBSK pulp. Figure 4a indicates the change in pulp freeness with the number of PFI revolutions, and Figure 4b compares the change in bulk of the tissue paper at different pulp freeness values. Freeness of pulp determines its drainage behavior when used on paper machines to produce paper. As the number of PFI revolutions increased, the freeness of both pulps decreased proportionally (Figure 4a). Fique residue and NBSK almost followed a similar drainage profile with the refining; however, fique residue seems to have a faster drop in pulp freeness at higher PFI revolutions. Compared to pulp freeness, fique residue pulp displayed higher bulk than NBSK pulp for a given value of pulp freeness (Figure 4b). However, fique residue pulp had a higher reduction in bulk than NBSK pulp with increasing refining. Thus, the bulk of both pulps seems to be converging at higher refining energy (lower pulp freeness). It appears that the thin cell wall of NBSK pulp easily develops internal fibrillation, thus making densified paper sheets than fique residue pulp. On the other hand, fique residue pulp also favors a certain degree of external fibrillations apart from internal fibrillation, particularly in the later stage of refining. These observations agree with

previous studies aimed to separate the effects of external and internal fibrillation on final paper properties. Wang et al. (2007) and Kang and Paulapuro (2006) reported that internal fibrillation increases the collapsibility and flexibility of fibers and is the primary factor responsible for the densification of a paper sheet, while external fibrillation leads to an increased specific surface area of fibers and mainly correlates with the decreased freeness of pulp.

3.3. Comparison of strength properties

Tissue paper products must have sufficient strength and durability to resist tear and rupture during the manufacturing and consumer use stages (De Assis et al. 2018). In this context, tensile strength and tear strength are considered two essential indicators of tissue paper's strength properties (Shannon 2016). Page (1969) and Van den Akker (1958) showed that the strength of a lightly bonded paper structure such as tissue papers is mainly limited by the inter-fiber bonding strength between individual fibers rather than the strength of individual fibers (Van Den Akker et al. 1958; Page 1969). Mechanical refining and wet pressing can be used to improve the inter-fiber bonding in a tissue paper sheet; however, there are undesirable tradeoffs with other important tissue properties such as softness and water absorbency. NBSK fibers with long fiber length, low coarseness, and easily collapsible thin cell walls are highly desirable in the tissue industry to provide the necessary strength to the paper while balancing softness and other important tissue properties (Shannon 2016; De Assis et al. 2018). Thus, the potential of fique residue bleached pulp to act as a reinforcing fiber in tissue paper products was investigated by comparing its strength properties against the NBSK market pulp. Figure 5a shows the tensile strength of fique residue bleached pulp and NBSK pulp at different pulp freeness, and Figure 5b compares the tear strength of both pulps for a given tensile strength value. Both fibers were subjected to the same amount of refining energy (measured by the number of PFI revolutions), and their response to this refining energy was observed.

As expected, the tensile strength of both pulps increased, and freeness decreased as refining forces increased the flexibility, conformability, and total surface area of the fibers. At higher pulp freeness, when the bonding between fibers is low, the tensile strength of fique residue is higher than the NBSK market pulp. When refining is increased, NBSK pulp develops tensile strength faster than the fique residue pulp. At pulp freeness below 600 ml when interfiber bonding is relatively developed, NBSK market pulp displays superior tensile strength than the fique residue pulp. The difference in the tensile strength curve between both fibers can be understood by observing the morphology of fibers in the unrefined state and changes occurring in the structure of fibers throughout the refining process. Higher fiber population, higher aspect ratio, and lower fiber coarseness of fique residue pulp (Table 2) translate into better fiber coverage and a higher number of fiber contacts than the NBSK market pulp and contribute to its higher tensile strength in the unrefined state (Seth 1990a, b). As refining proceeds, NBSK fiber with its wider lumen and thinner cell walls collapses easily into a flat ribbon-like structure (Figure 3 and Figure 4b), thus significantly increasing the relative bonded area between the fibers. Also, at higher refining when bonding between fibers is well developed, a higher percentage of fibers are broken during the tensile failure of paper, and individual fibers' tensile strength becomes more important for the total strength of paper (Van Den Akker

et al. 1958; Page 1969). Zero span tensile strength of both pulps was measured as an indicator of tensile strength of individual fibers in that pulp (Van Den Akker et al. 1958; Seth and Chan 1999). It was observed (Table 2 and Figure A1 in the supplementary information) that NBSK pulp has approximately 20% - 35% higher zero span tensile strength than fique residue bleached pulp indicating superior tensile strength of individual fibers in the NBSK pulp. Moreover, SEM micrographs (Figure 3) show that the refining actions expose cellulosic fibrils and microfibrils on the outer cell wall layers of fique bleached residue pulp and make its fiber surfaces rougher than the refined NBSK pulp. A rougher surface hinders the contact between adjacent fibers and reduces the relative bonded area (Hubbe 2006), thus resulting in weaker inter-fiber bonding for fique residue bleached pulps than NBSK market pulp at higher refining levels.

After a direct fiber-to-fiber comparison between the fique residue pulp and the NBSK market pulp, both reinforcement pulps were separately blended with BEK market pulp at different weight proportions, and tissue handsheets were made using the resulting pulp furnish. Since fique residue bleached pulp provided better bulk properties even at higher refining, it was refined at two different pulp freeness values and separately blended with BEK pulp. Figure 6 shows the tensile strength of the resulting tissue handsheets as a function of the weight percentage of the reinforcing pulps in the pulp furnish. As the amount of reinforcement pulp increased in the furnish, the tensile strength of the final paper increased proportionally. As expected, the addition of fique residue bleached pulp refined at higher refining energy (or to a lower freeness) provided the highest improvement in the tensile strength at any given weight percentage. Despite NBSK pulp showing faster tensile strength development with refining, the addition of fique residue bleached pulp refined at similar refining energy as NBSK pulp (1000 PFI revolutions) provided either similar or better tensile strength than the NBSK market pulp. The reinforcement mechanism is based on longer fibers forming bonds with multiple fibers leading to a more efficient stress transfer between fibers when a load is applied to the fibrous assembly (Seth 1990b). Moreover, blending two different fibers in a pulp furnish can lead to either positive or negative synergy between the fibers depending on the packing structure of fibers in the fibrous network. Considering that NBSK fibers had a compact structure even at 1000 PFI refining revolutions, BEK pulp's addition might disrupt its existing compact network structure. In comparison, fique residue bleached pulp had an open network structure with lots of voids and gaps between the fibers. The addition of shorter fibers from BEK pulp might fill the open space and bridge the gap between fibers leading to better stress distribution when a load is applied.

3.4. Water absorption capacity

Hygiene tissue products are designed to absorb the maximum amount of water in the lowest possible time (Beuther et al. 2010). Hence, water absorbency properties are one of the most crucial properties for tissue paper products. Water absorption capacity determines the total amount of water that a tissue paper can retain under saturated conditions (Ko et al. 2016). From a water absorption capacity point of view, tissue paper can be considered as a two-phase system of cellulosic fibers and interfiber pores where both phases can potentially absorb water (Bristow 1986). The function of the cellulosic fibers forming the underlying tissue structure is to maximize the volume of interfiber pores and provide sufficient

hydrophilicity for additional sorption of water into fibers (Ko et al. 2016; de Assis et al. 2019; Zambrano et al. 2021a).

The primary objective of adding a reinforcement pulp is to provide strength and durability to the tissue structure. However, the addition of a reinforcing fiber might negatively impact the water absorbency properties of the tissue paper (De Assis 2018; Stankovská et al. 2020). Hence, the best performing reinforcement pulp should provide the maximum water absorbency for a given tensile strength of tissue paper. Figure 7 compares the water absorption capacity of fique residue pulp with NBSK pulp at different refining energies. Refining has been used to change the tensile index and the apparent bulk of paper made with both pulps. Figure 7a and Figure 7b show that fique residue bleached pulp provided better water absorption capacity than NBSK pulp for any given value of the tensile index. As refining progressed, the water absorption capacity of both pulps decreased proportionally; however, the same trend of fique residue pulp having higher water absorption capacity than NBSK pulp was observed at all refining levels used in this study. Previous studies have shown that the majority of water absorbed in a tissue paper sheet is located inside the pores between fibers and fiber lumens (Bristow 1986; de Assis et al. 2019; Zambrano et al. 2021b). De Assis et al. (2019) and Zambrano et al. (2021) reported a high correlation between water absorption capacity and the bulk of the tissue paper. For tissue papers made with highly bleached cellulosic fibers, when there is not much difference between the chemical composition of fibers, the difference in water absorption capacity can be explained based on the total apparent pore volume available in the paper (Zambrano et al. 2021b). Thus, fique residue bleached pulp's superior water absorption capacity can be attributed to the higher bulk of its paper structure (Figure 7b). Overall, water absorption capacity showed a 92% linear correlation with the bulk of all tissue handsheets evaluated in this study. This effect can also be visualized through the SEM micrographs in **Figure 3**, wherein higher fiber curl and tubular fiber structure of the fique residue pulp created a relatively porous and bulkier web structure than the compact and consolidated paper web structure generated by NBSK pulp.

Additionally, it is important to note that all tissue papers displayed higher water absorption capacity than the original pore volume present in the paper, which can be calculated using the apparent bulk (cm^3/g) of the paper in the dry state. The extra water being absorbed is accommodated between the plies, the pores within the fiber cell wall, and changes in the original pore volume of paper caused by the swelling of the cellulosic fibers (Bristow 1986; de Assis et al. 2019). An equal number of handsheets was used to measure the water absorption capacity for both pulps; hence, it can be assumed that a similar amount of water was present between the plies in all observations. However, it is essential to acknowledge that NBSK pulp is a once-dried pulp, and fique residue is a never-dried pulp. Drying brings some irreversible changes in the morphology of fibers (i.e., hornification) and negatively impacts their swellability with water upon rewetting (Gurnagul et al. 2001; Hubbe et al. 2007).

Figure 8 shows the water absorption capacity as a function of the tensile index of tissue handsheets prepared from pulp blends of BEK and the individual reinforcement pulps. Tissue handsheets made with 100% unrefined BEK pulp have a water absorption capacity of 7.1 grams water per gram of fiber which was in a similar range as observed for fique residue bleached pulp refined at 1000 PFI (635 CSF mL).

Hence, the addition of fique bleached pulp refined at 1000 PFI did not have a negative impact on the water absorption capacity of the tissue handsheets. Compared to that, adding NBSK pulp or fique residue pulp refined at higher refining energy (2000 PFI) reduced the water absorption capacity of the resulting tissue handsheets (Figure 8). However, in both cases (similar refining energy or higher refining energy), the fique residue pulp provided better water absorption capacity than NBSK pulp for a given value of the tensile index, which can be attributed to the bulkier fibrous structures imparted by the introduction of fique residue pulp

3.4. Softness

The perceived softness of a hygiene tissue product is a subjective impression that the human mind generates when tactile sensors present in human hands interact with the sample material (Wang et al. 2019). Previous studies have shown a high correlation between perceived softness and measurable physical properties of tissue papers such as surface roughness, stiffness, compressibility, flexibility, free fiber ends, fiber flexibility, etc. (Hollmark 1983; Hollmark and Ampulski 2004; Wang et al. 2019). Accordingly, two different aspects of softness have been proposed in terms of surface softness and bulk softness. The surface softness relates to the perception of softness when fingertips move over the surface of a tissue paper, while bulk softness indicates the softness perception when a human hand folds and crumples the tissue paper (Hollmark 1983). In this work, the softness of the tissue handsheets was measured using a tissue softness analyzer (TSA, Emtec Electronic GmbH, Germany), which measures the surface and bulk softness independently using three basic parameters: TS7 (surface softness), TS750 (surface smoothness/roughness), and in-plane flexibility (bulk softness) (Wang et al. 2019; Prinz et al. 2021). As with the water absorption capacity, the objective is to maximize the softness of tissue handsheets for a given value of the tensile index. Figure 9a and 9b compare the TS7 (surface softness) and the in-plane flexibility (bulk softness) of tissue handsheets made with 100% fique residue bleached pulps and 100% NBSK pulp without any fiber blending with BEK pulp.

The comparison among BEK, NBSK, and fique residue bleached pulp in their unrefined state showed that BEK pulp had the best softness properties in terms of surface softness (TS7), bulk softness (in-plane flexibility), and TS750 (surface smoothness/roughness). TS750 values for tissue handsheets are presented in Figures A3 and A4 and provided in the supplementary information. BEK pulp produced the lowest TS7, highest in-plane flexibility, and highest surface smoothness (lowest TS750). Fique residue bleached pulp provided better surface softness (lower TS7) and higher surface smoothness (lower TS750) than the NBSK pulp with a similar bulk softness (in-plane flexibility). As refining was used to develop the tensile strength of NBSK and fique residue pulp, it negatively impacted all aspects of softness. NBSK pulp with lower coarseness and flexible thin cell walls are desirable in hygiene tissue products over other long-length fibers such as SBSK (southern bleached softwood kraft) given their ability to impart superior surface softness property (Shannon 2016; De Assis 2018). However, fique residue bleached pulp consistently produced lower TS7 values than NBSK pulp at all refining levels which indicates its ability to provide superior surface softness when used in hygiene tissue products. It appears

that the combination of shorter fiber length, lower coarseness, low fines content, higher fiber curl, and higher fiber population contributes to the fique residue pulp having lower TS7 values than NBSK pulp (Wang et al. 2019; Prinz et al. 2021). However, it is also important to acknowledge the limitations of TSA equipment regarding its ability to accurately compare the softness of samples made from different fiber sources. Apart from flexible and free fiber ends, a more porous sample can also dampen the amplitude of vibrations more efficiently, resulting in lower TS7 values recorded in comparison with a less porous surface (Prinz et al. 2021). Fique residue pulp had a lower apparent density (higher porosity) than the NBSK pulp at all refining levels (Figure 4b), which could have also contributed to it producing lower TS7 values than NBSK pulp. The open and porous nature of tissue samples made with fique residue bleached pulp can also be visualized through the SEM micrographs presented in Figure 3.

Compared to the surface softness, it was surprising to observe both pulps showing similar in-plane flexibility or bulk softness at all refining levels despite fique residue pulp having superior bulk. To further evaluate this observation, tensile stiffness of handsheets was recorded from the tensile testing of handsheets. Figure A2 (provided in the supplementary information) indicates that both pulps have similar tensile stiffness in their unrefined state, but the NBSK pulp has significantly higher tensile stiffness (~35% higher) than the fique residue pulp at higher refining levels. The in-plane flexibility as measured by the TSA equipment is closely related to the flexural rigidity or bending stiffness of the sample (Zambrano et al. 2021a). The flexural rigidity, in turn, is a function of the tensile stiffness and the third power of the sheet thickness (Hollmark 1983). Hence, it appears that the sheet thickness is also having significant impacts on the measured in-plane flexibility of tissue samples (Hollmark 1983). This might explain why NBSK pulp with higher tensile stiffness but lower caliper has similar in-plane flexibility as fique residue pulp with lower tensile stiffness but higher caliper.

After comparing the softness properties of individual pulps, the softness of tissue handsheets prepared using pulp blends of BEK and reinforcement fibers was studied. Figure 9c and Figure 9d present the TS7 (surface softness) and in-plane flexibility (bulk softness) of tissue handsheets made with different pulp blends. Since reinforcement pulps were successively added in smaller weight percentages, it is expected that tissue properties should be mainly dominated by the BEK fibers present in the majority. It is argued that this effect should also negate the impact of porosity on softness measurement, thus providing a more accurate comparison of softness than that obtained by comparing pulps individually. Shorter fibers have a higher tendency to protrude out from the paper surface, which is augmented by the remarkably higher number of individual fibers in the pulp. On the other hand, adding refined and long-length reinforcing fibers reduces the number of free fiber ends and increases the stiffness of the paper structure by increasing the degree of bonding between the fibers. As a result, both surface softness (TS7) and in-plane flexibility got worsened as the amount of reinforcing pulp was increased in the tissue handsheet. Individually, tissue handsheets prepared with the pulp blend of BEK and fique residue refined at lower refining energy provided superior surface softness (lower TS7) than the NBSK pulp. However, when fique residue pulp was refined at higher refining energy (580 CSF), its surface softness became comparable to the NBSK pulp (Figure 9c). Similar to the trend observed when comparing individual pulps, in-plane

flexibility or the bulk softness of all pulp blends was comparable (Figure 9d), which was attributed to the combined effects of tensile stiffness and the thickness of tissue handsheets.

4. Conclusion

Pulp blends of northern bleached softwood kraft (NBSK) and bleached eucalyptus kraft (BEK) are commonly used to produce premium hygiene tissue products with best combination of water absorbency, softness, and strength properties. However, environmental concerns over logging NBSK has created uncertainty about its long-term supply. This work successfully demonstrated the feasibility of upgrading agricultural residue from the processing of fique fibers as a sustainable alternative to NBSK fibers for producing high-performance tissue products.

Fibers from fique residue can be upgraded into high-quality pulp using a simple mechanical cleaning and mild pulping and bleaching process. Fiber morphology played a critical role in developing tissue paper's properties, and a comparison of morphological properties showed that fique residue pulp has similar fiber length, lower width, coarseness, fines content, and higher fiber population than NBSK market pulp. SEM micrographs showed that the compressive forces acting during the refining process easily collapsed the thin cell walls of NBSK pulp into a flat ribbon-like structure, while fibers from the fique residue pulp were less flexible and conformable. The bulk of tissue handsheets made with fique residue bleached was consistently higher than NBSK pulps at all refining levels, which corroborate different responses of both fibers to mechanical refining. Therefore, NBSK pulp displayed higher tensile strength than fique residue pulp at higher refining levels (lower pulp freeness) but similar strength properties at higher pulp freeness. When tradeoffs between the tensile strength, water absorbency, and softness were evaluated, fique residue bleached pulp provided a superior combination of water absorption capacity and softness properties than NBSK market pulp at a given tensile strength value. However, it is important to mention that NBSK market pulp is a once-dried pulp, while fique residue pulp was produced in the laboratory and has not been dried. Drying has been shown to bring some irreversible changes in the fiber cell wall that negatively impacts the bonding ability and swellability of fibers. Hence, the results should be analyzed as demonstrative rather than representative of the pulp samples. Nevertheless, promising results obtained from this study show the potential of low-value residue from fiber crops of abaca, sisal, fique, hemp, flax to be developed into high-quality pulps for hygiene tissue applications. This may not only solve the residue handling problems but also open a new revenue stream for millions of small farmers in the producing countries while providing flexibility in fiber sourcing for the manufacturer of tissue paper products.

Declarations

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Competing Interests

The authors declare no relevant financial or non-financial interests to disclose.

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Tables

Table 1 – Results from the pulping and bleaching studies of fique residue fibers

Fiber Samples	Fique Residue
Soda pulping	
Soda charge (% fiber o.d. weight)	20
Total yield ¹ (%)	55.4
Kappa number	24
ISO brightness (%)	40
CED pulp viscosity (cP)	20
ECF Bleaching (D0-Ep-D1)	
ISO brightness (%)	84
Bleaching yield ² (%)	92
Total yield (%)	51

¹ yield includes screened yield as well rejects. Rejects during all pulping trial was less than 1% on o.d. fiber basis

² Bleaching yield also includes manual losses during the process

Table 2 – Morphological properties of unrefined bleached pulps used in this study for making tissue paper

Pulp Name	Fique Residue	NBSK market pulp	BEK market pulp
Arithmetic mean fiber length (mm) (> 0.2 mm)	1.8	1.7	0.61
Length weighted mean fiber length (mm) (> 0.2 mm)	2.3	2.4	0.71
Mean fiber width (μm) ($W = 7 - 60 \mu\text{m}$)	22.9	25.8	15.8
Fines content ¹ (%) (0.025 - 0.2 mm)	1.1	2.2	4.5
Fiber coarseness (mg/km)	125	135	73
Mean curl index	0.16	0.14	0.097
Fiber population ² (million fibers/gram)	3.5	3.2	18.8
Zero span tensile strength (km.)	10.9	14.8	10.8

¹ length weighted fines content

²Fiber population has been calculated using total number of fibers divided by the total mass of fibers used for each FQA analysis

Figures

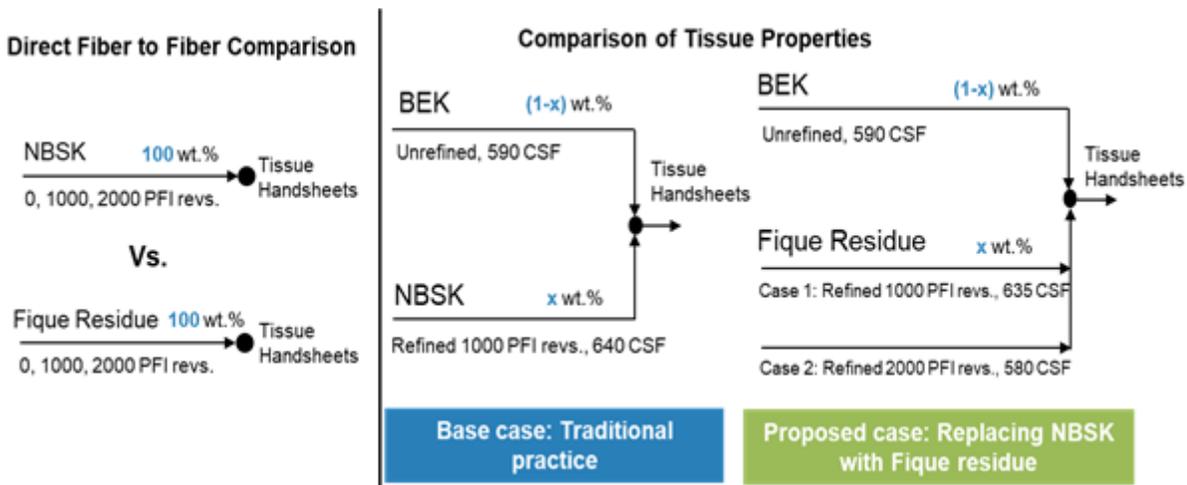


Figure 1

Work plan designed for comparing the tissue properties of fique residue bleached pulp with NBSK market pulp. PFI revolutions indicate the refining energy applied to each pulp before making the tissue handsheets. PFI revolutions indicate the refining energy applied to each pulp before making the tissue handsheets.

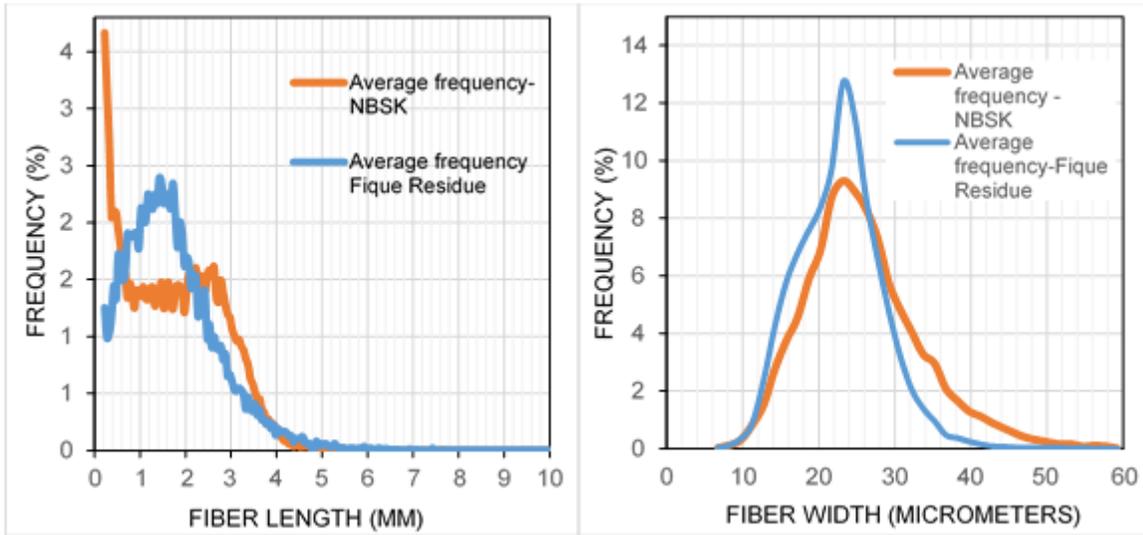


Figure 2

Comparison of (a) fiber length and (b) fiber width distribution for NBSK market pulp and fique residue bleached pulp

Figure 3

SEM micrographs of surface and cross section of unrefined and refined handsheets made with fique residue bleached pulp and NBSK market pulp. a-c unrefined fique residue (703 ml CSF); d-f unrefined NBSK (696 ml CSF); g-i refined fique residue (1000 PFI, 635 ml CSF); j-l refined NBSK (1000 PFI, 640 ml CSF)

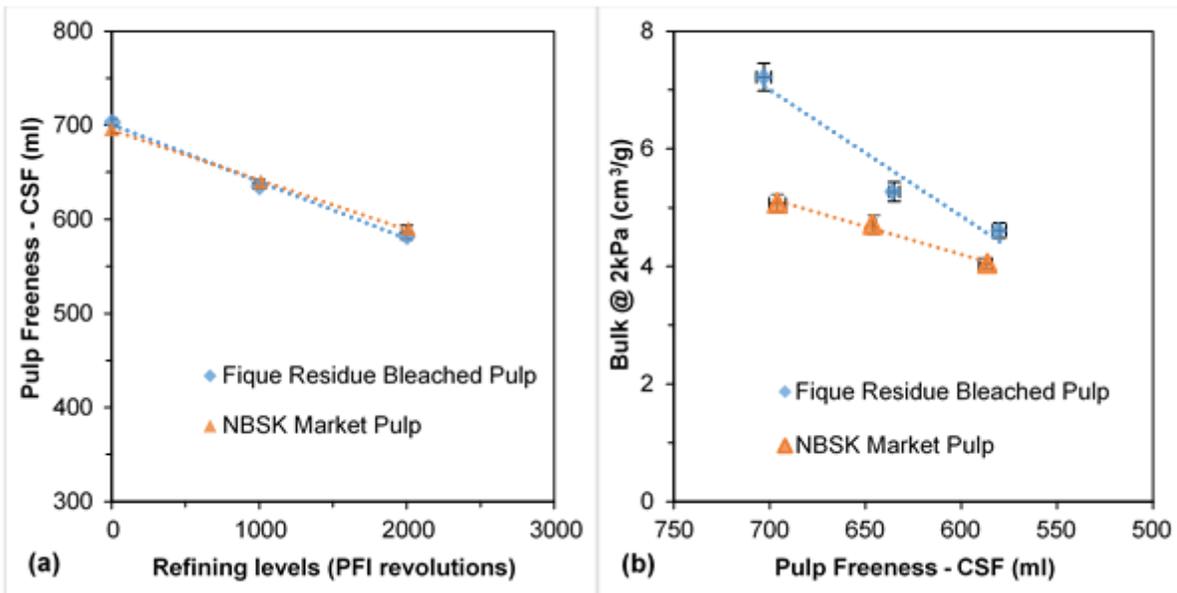


Figure 4

Effect of refining on fiber properties evaluated by measuring (a) the change in pulp freeness against the number of PFI revolutions, and (b) change in the bulk of paper by varying the freeness of pulps with refining. Error bars indicate one standard deviation.

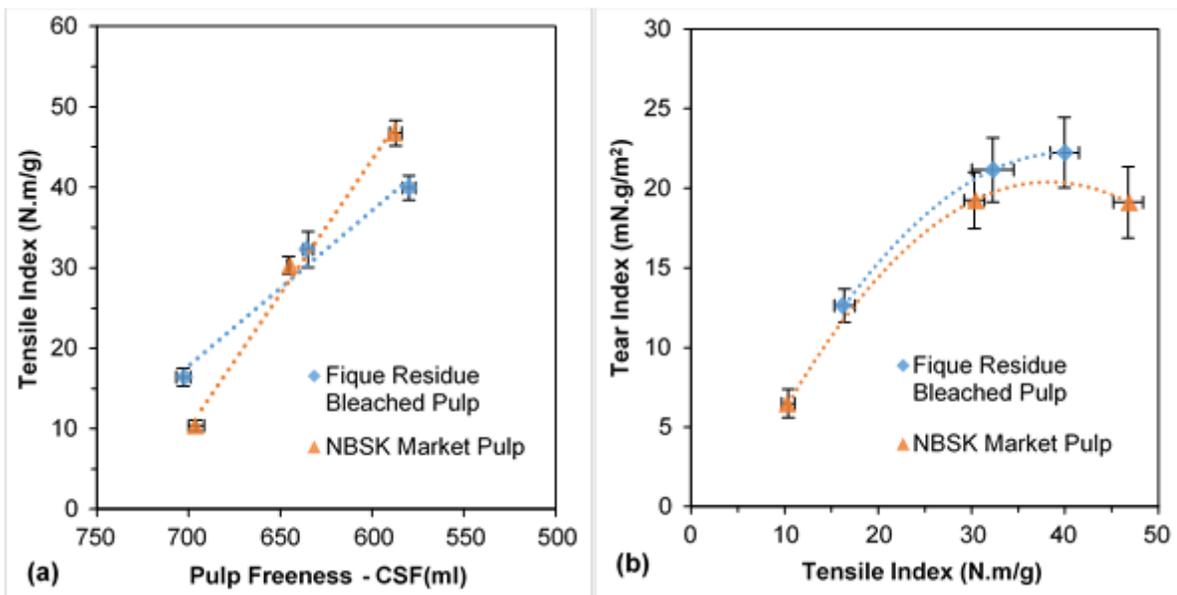


Figure 5

Comparison of strength properties of figue residue bleached pulp with NBSK market pulp in terms of (a) tensile strength as a function of pulp freeness, and (b) the relationship between tear and tensile index. Error bars indicate one standard deviation.

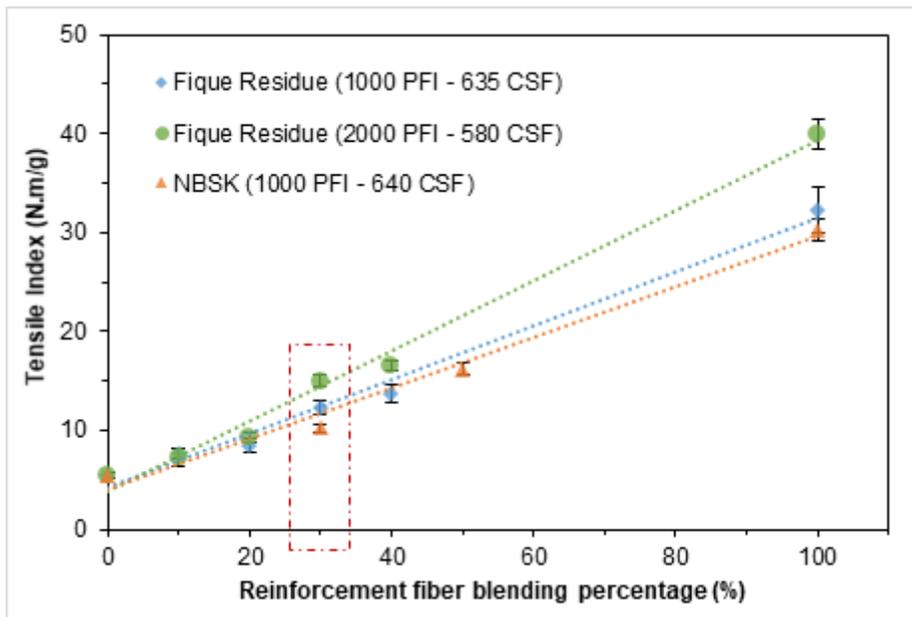


Figure 6

Tensile strength of tissue handsheets prepared by blending reinforcement pulps with bleached eucalyptus kraft pulp plotted against amount of reinforcement pulp in the pulp furnish. Zero % corresponds to pure eucalyptus pulp and 100% corresponds to only reinforcement pulps. Error bars indicate one standard deviation

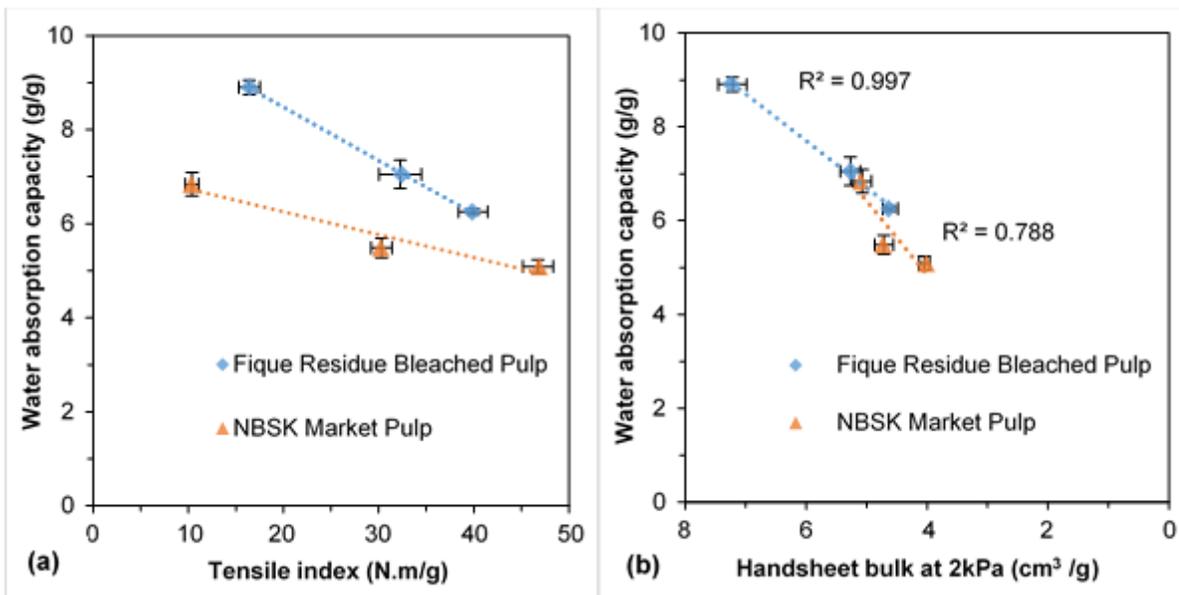


Figure 7

Comparison of water absorption capacity of fique residue bleached pulp with NBSK market pulps (a) the tradeoffs between water absorption capacity and tensile index, and (b) as a function of tissue handsheet bulk. The error bars indicate one standard deviation.

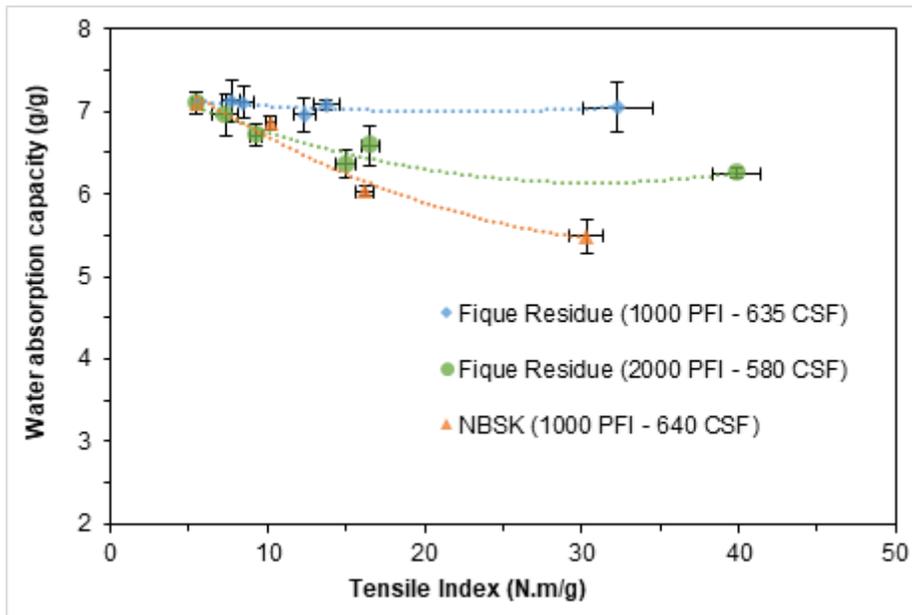


Figure 8

Water absorption capacity of tissue handsheets prepared by blending reinforcement pulps with bleached eucalyptus kraft and plotted against the tensile index of handsheets. The amount of reinforcing fibers increases from left to right and tensile index increased proportionally. Error bars indicate one standard deviation

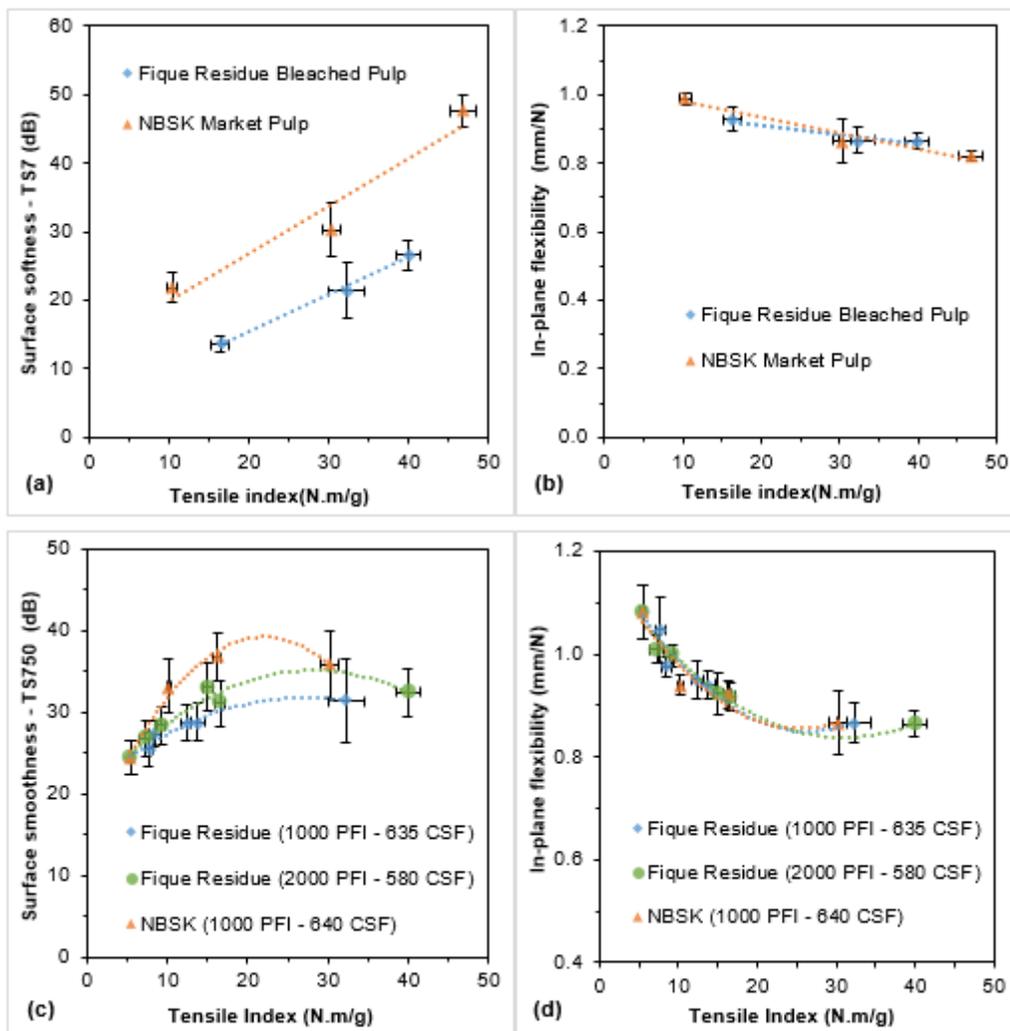


Figure 9

Comparison of softness between fique residue and NBSK pulp by evaluating tradeoffs between softness and tensile strength (a) Tradeoff between surface softness and tensile index for individual pulps (b) Tradeoff between bulk softness and tensile index for individual pulps (c) Tradeoff between surface softness and tensile index for pulp blends with BEK (d) Tradeoff between bulk softness and tensile index for pulp blends with BEK. The amount of reinforcing fibers increases from left to right in Figures c and d. Error bars indicate one standard deviation.

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