

# The Potential of Wheat Straw High Yield MEA Pulp for Enhancing Strength Properties of Recycled Paper

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This study investigated the blending of unbleached wheat straw high yield MEA (monoethanolamine) pulp with recycled pulp to improve the strength properties of recycled pulp. First, the cooking temperature in MEA high yield pulping was stepwise reduced from 160 °C to 120 °C to enhance pulp yield as much as possible. The optimum temperature for the intended application was 130 °C. In the second series of cooking performed at this temperature, the MEA charge was gradually reduced. However, with the reduction of MEA charge, the Kappa number rapidly increased. The solvent recycling was considered in a third series by reusing black liquor two times. The Kappa number increase, due to partial reuse of black liquor, was low, and the pulp strength remained at a high level. A MEA straw pulp, prepared without black liquor reuse, was refined with low energy input to various beating degrees to evaluate the refining behavior and strength development. The MEA straw pulp samples with different beating degrees were blended with recycled pulp. The ratio of blending varied between 5% and 20%. The results revealed that MEA straw pulp was well-suited as a reinforcement pulp. All strength properties of the recycled pulp, except tear strength, were improved.

*Keywords:* paper recycling; Non-wood; Wheat straw; Monoethanolamin; High-yield; Inter-fiber bonding; Refining

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## INTRODUCTION

Across the literature, it has been emphasized repeatedly that recycled pulps have inferior strength properties compared to the original virgin pulps (McKee 1971; Horn 1975; Yamagishi and Oye 1981; Ferguson 1992; Jahan 2003; Hubbe and Zhang 2005; Nazhad 2005; Salam 2015). The main reasons for the reduction of strength properties are changes in structural integrity, length, swelling behavior, and bonding potential of the fibers along the recycling chain (Ellis and Sendlachek 1993; Hubbe *et al.* 2007). The loss in bonding potential can be mainly traced back to hornification during paper drying (Nazhad and Paszner 1994; Diniz *et al.* 2004; Welf *et al.* 2005; Billosta *et al.* 2006; Salam 2015). Further fiber damage occurs during paper printing, converting, storage, usage, re-pulping, deinking, and re-refining (Hubbe *et al.* 2007). It has been demonstrated that pulp grades behave differently during recycling (Chatterjee *et al.* 1991; Howard and Bichard 1992; Alanko *et al.* 1995; Nazhad 2004; Salam 2015); however, the impact seems to be similar for chemical pulps produced of softwood, hardwood, or non-wood. The most significant loss of the strength properties takes place in the course of the first recycling, mainly caused by hornification during drying on the paper machine. Repeated

fiber recycling leads to some additional losses until the fourth or fifth recycling loop, where the fiber strength becomes constant. The strength loss during repeated recycling may accumulate up to 30% (Nazhad 2004).

Europe has a remarkably high paper-recycling rate for packaging paper. Efforts to further increase this rate are faced with the problem of declining recycled paper quality, mainly due to the reduced fiber bonding ability at intensified recycling and lacking input of higher-graded graphic paper or fresh fibers. Four ways have been suggested for restoring the bonding ability of recycled fibers: Refining, chemical treatment or additives, fractionation, and using virgin fibers as blending material (Howard 1990). Refining introduces physical changes into the pulp fibers with only minor changes in the chemical composition (Casey 1960). It modifies the pulp through delamination of the layer structure and formation of external and internal fibrils *via* generation of fines, and thus improves the specific bond strength (Laine *et al.* 2002). But it also diminishes the inherent strength of the individual fibers and may cause problems in dewatering; thus, it may reduce the production rate. The bonding ability of the fiber surface can also be improved by additives, especially starch, or by enzyme or oxidation treatment, preferably with ozone or hydrogen peroxide. Sodium hydroxide can be applied for improving the swelling capacity (Katz *et al.* 1981; Freeland 1995). Fractionation can successfully be applied to regain the recycled pulp strength. In this case fine fibers and fiber fragments are removed, which primarily increases the inherent pulp strength and improves dewatering. Another practical approach is to blend recycling paper furnishes with virgin fibers or with recycling paper grades containing a higher percentage of virgin fibers (Strümer and Götsching 1979; Nazhad and Paszner 1994; Hubbe *et al.* 2007).

It has been reported that paper recycling goes along with the reduction of hemicellulose content on the fiber's surface (Oksanen *et al.* 1997; Cao *et al.* 1998; Nazhad 2005). Consequently the amount of functional carboxylic groups available for hydrogen bonds between fibers is lowered, resulting in poor fiber bonding capacity and low bonding-related strength properties. It has further been shown in many studies that virgin fines material plays an important role for promoting bonding in the fiber network of recycled paper (Htun and de Ruvo 1978; Corson 1980; Laivins and Scallan 1996; Nazhad 2005; Hubbe *et al.* 2007).

These facts give serious reason to assume that monoethanolamine (MEA) straw pulp, as a pulp with high content of hemicelluloses and fine, flexible fibers, but quite low lignin content (Hedjazi *et al.* 2009; Salehi *et al.* 2014), may be an excellent blending material for improving the strength of recycled pulp. Another principal advantage of straw pulp is the high initial beating degree and the extremely low energy demand for beating if necessary. Therefore, the focus of this study was aimed at the preparation of a reinforcement pulp for a recycled fiber-based packaging paper.

## EXPERIMENTAL

### Materials

Wheat straw was collected from cultivation areas in northern Germany. The moisture content of the collected straw was approximately 15%. Debris and impurities were separated and then the cleaned straw was chopped into small pieces.

The chopped straw was stored under standard climate conditions in a storage room until used. A raw material analysis was performed previously for wheat straw of the same province (Salehi *et al.* 2014).

The carbohydrate composition was determined according to a method developed by Puls (1983). The acid-insoluble lignin and ash content were analysed according to TAPPI T222 om-02 (2007) and T211 om-02 (2007) standards. For the blending tests, a recycling paper, grade (B12), dispatched from LEIPA Georg Leinfelder GmbH (Schwedt, Germany) was used.

### *Pulping*

Pulping was performed with a constant straw charge of 2500 g, and oven-dried in a specially designed 80-L digester (University of Hamburg, Hamburg, Germany). It was equipped with a central rotary paddle and worked in horizontal position, thus simulating a horizontal tube continuous digester. It had a programmable temperature control unit, and heating was conducted indirectly by steam within 20 min. After cooking, the unbleached pulp either was passed through a pulper and was then screened in a vibrating screener (Weverk, Stockholm, Sweden) with 0.15-mm slots to determine accepted and rejected yield, or it was passed through a Sprout-Bauer 12" laboratory refiner (Gratz, Austria) at 4% consistency and 0.35-mm disc clearance.

### *Refining*

Laboratory pulp beating was performed in a Jokro mill (FRANK-PTI, Birkenau, Germany) according to the German standard Zellcheming V/5/60 (1960). Refining in pilot scale was done with a Voith laboratory refiner, model LR 40 (Heidenheim, Germany) at a pulp consistency of 4%. The refiner was equipped with standard refiner plates for short fiber pulp and operated at a rotation speed of 2000 rpm. The refining intensity was controlled at 0.3 J/m. The straw pulp was refined under specific refining energies of 0 kWh/t, 20 kWh/t, 45 kWh/t, 70 kWh/t, 95 kWh/t, and 120 kWh/t.

### *Papermaking*

Handsheets were made with a Rapid Koethen sheet former (FRANK-PTI, Birkenau, Germany). For blending tests, MEA straw pulp was mixed with the recycled fibers in a disintegrator directly before preparation of handsheets.

## **Methods**

### *Pulp characterization*

The following test methods were used for pulp evaluation: Fiber classification using a Kajani fiberlab analyzer (Metso, Espoo, Finland); Kappa number, Zellcheming IV/37/80 (1980); beating degree, Zellcheming V/7/61(1961); breaking length, tensile, tear, and burst strength, Zellcheming V/12/57 (1957); strength index calculation according to TAPPI T220 sp-01 (2007), and SCT index (Short Span Compressive Strength) according to TAPPI T826 om-04 (2007).

## RESULTS AND DISCUSSION

The principal characteristics of MEA pulping of non-woods, its advantages, and its disadvantages have been presented in previous papers (Salehi *et al.* 2014). A special feature of MEA pulping is its high selectivity. The pulping yield is much higher in comparison to soda pulping, even at a lower kappa number.

The high carbohydrate content and the high flexibility of straw fibers promote good fiber bonding and high tensile strength. However, when such a pulp is dried a serious loss of bonding potential occurs due to the hornification effect. To maintain this beneficial feature, such MEA straw pulps should not be dried. This means, in consequence, that MEA pulping should be operated in an integrated mill. Thus, this pulp quality has an impact on the production capacity, which has to be kept small, tailored to the mill's needs. The mill should run with simple technology and without any bleaching. With these considerations in mind, a favorable application of MEA pulping is the production of reinforcement pulp directly in a mill that produces packaging paper based on recycled fibers and may have problems meeting market demands due to the declining recycled fiber quality. In this case, a higher Kappa number can be accepted as long as the higher lignin content has no negative impact on the bonding potential.

As pulp yield and energy input into the pulping process are important factors with regards to the production cost, a series of MEA cooks was carried out with stepwise reduction of the cooking temperature from 160 °C to 120 °C, while the other cooking parameters were kept constant, as can be seen in Table 1.

**Table 1.** MEA and Soda Cooks Performed at Different Temperatures

Cooking Process	Temperature (°C)	AQ (%)	NaOH (%)	Screened Yield (%)	Rejects (%)	Total Yield (%)	Kappa Number
MEA	160	0.1	-	56.3	1.2	57.5	16.9
	150	0.1	-	58.4	1.7	60.1	19.3
	150	0	-	56.7	3.3	60.0	22.5
	140	0	-	59.1	2.5	61.6	21.5
	130	0	-	59.8	4.2	64.0	25.4
	120	0	-	52.3	14.0	66.3	36.0
Soda	160	0	16	-	-	50.5	22.1
	130	0	16	-	-	50.5	31.6
	130	0	12	-	-	55.8	36.8
	120	0	16	-	-	53.0	38.2

Constant: Liquor/straw ratio 3:1; cooking time 30 min; MEA pulping: MEA/water ratio 50/50; MEA charge 150%; Defiberation in a pulper and screening with 0.15 mm slots  
soda pulping: Defiberation in a refiner

As with soda pulping, the addition of anthraquinone (AQ) had a beneficial effect on the selectivity of MEA cooking. Therefore, it has been used in standard MEA cooking experiments. This is why AQ was charged in the first two cooks of this series at high temperature. Further experiments were performed without AQ. Due to its expensive cost, its use is restricted. Moreover, the positive effect of AQ is less pronounced at a high yield level. As shown in a direct comparison of the results achieved in cooking at 150 °C with and without AQ, the biggest effect of AQ was the reduction in the reject content. The

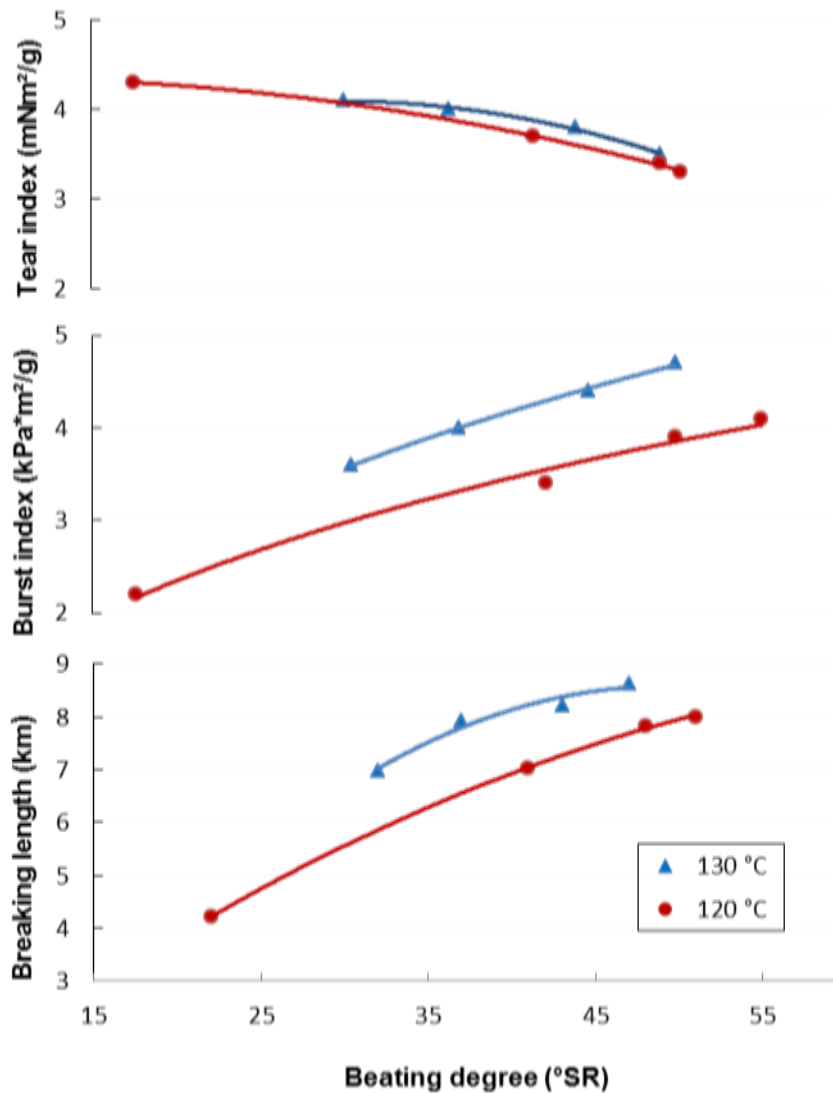
impact of reduced cooking temperature in MEA straw cooking on the attained Kappa number was astonishingly small. With reduction of the cooking temperature from 160 °C to 130 °C the Kappa number increased only from 17 to 25, although no AQ was added in the cook conducted at low temperatures. In contrast, a clear increase of the yield could be attained, but at a higher reject content. However, a further decrease in temperature to 120 °C resulted in a considerable increase of the Kappa number and the rejected content.

In a series of soda cooks, an attempt was made to improve the yield by lowering the cooking temperature, as shown in Table 1. In consideration of the strong increase of the reject content with decreased cooking temperature, the working off procedure for the soda pulps was changed to exploit the full benefit of temperature reduction on pulp yield. Therefore, instead of defiberation in a pulper and subsequent screening in the accepted pulp and rejects, the whole pulp was defiberized in a refiner; thus no rejects were obtained. Despite this modification, the total yields in soda pulping were considerably lower than the screened yield obtained in MEA pulping. Moreover, the increase in Kappa number with reduced cooking temperature to 130 °C was more distinct. Ultimately, the soda cooks performed at a lower temperature resulted in a much less favorable yield to Kappa number relationship compared to the corresponding MEA cooks.

The MEA cooking series with gradual reduction of the cooking temperature was performed to find the minimum cooking temperature applicable to produce reinforced pulp for recycled fiber-based packaging paper. At 120 °C the Kappa number increased considerably. A longer cooking time to further reduce the Kappa number was not practical if the cook were to be performed in a modern horizontal tube digester. Therefore, it was important to know whether a MEA straw pulp with a higher Kappa number, produced at such a low temperature, was still suitable for the aimed end use. For this reason, the basic strength properties of those MEA pulps produced at 130 °C and 120 °C were tested and were compared in Fig. 1.

The MEA pulp produced at 130 °C showed good tensile and burst strength, which indicated a high bonding ability of the fibers. The pulp strength was at the same level for MEA pulp produced at 160 °C with the same raw material used in a previous study (Salehi *et al.* 2014). While the tear strength of the pulp produced at 120 °C was similar, both, tensile and burst strength were clearly lower. Presumably, the fibers were less flexible and may have had fewer bonding-active carbohydrates on the fiber surface. Because of its lower bonding potential, the pulp produced at 120 °C was less suited as reinforcement pulp. Based on these results it can be concluded that 130°C was the lower temperature limit for industrial production of high yield MEA straw pulp to be used as reinforcement material for packaging paper or board, based on recycled fibers.

One of the most important parameters in all organosolv pulping processes is the charge and consumption of the organic solvent. This is particularly important, if the solvent is relatively expensive and has to be recovered as completely as possible to keep the costs of chemicals within acceptable limits. The recovery efforts can be reduced to a great extent, if less organic solvent is used in cooking. Therefore, the MEA charge was stepwise reduced in the next cooking series. Based on the results of the first cooking series (Table 1), a cooking temperature of 130 °C was chosen. The finishing-off procedure of passing the whole pulp after cooking through the refiner was carried out in the same manner as the soda cooks.



**Fig. 1.** Strength properties of high yield MEA straw pulp produced at 130 °C and 120 °C (Jokro mill beating)

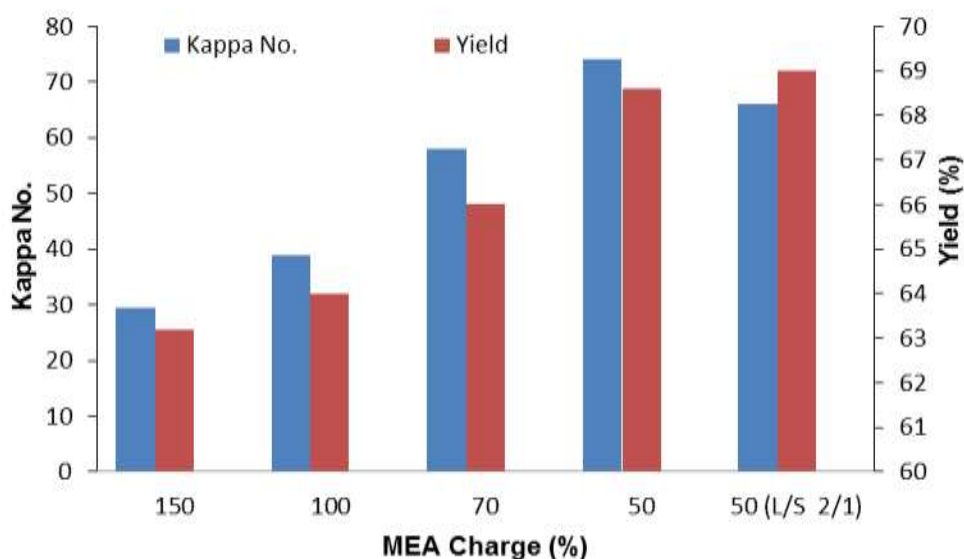
It can be seen by comparing the results of the first cook in this second series with the corresponding cook of the first series that the change of the working off procedure resulted in a higher amount of accepted yield at a somewhat higher Kappa number. In the first four cooks of this series, the saved amount of MEA was substituted with water to keep the liquor/straw ratio constant at 3/1. In the last cook, the water charge was also reduced to adjust the liquor/straw ratio to 2/1 (Table 2).

**Table 2.** Impact of MEA Charge on High Yield MEA Cooking

MEA/Water Ratio	MEA (%)	Liquor/Straw Ratio	Yield (%)	Kappa Number
50/50	150	3/1	63.2	29.5
33/67	100	3/1	64.0	39.0
23/77	70	3/1	66.0	58.0
17/83	50	3/1	68.6	74.0
17/83	50	2/1	69.0	66.0

Constant: Cooking temperature: 130 °C; Cooking time: 30 min

With reduction of the MEA charge, the Kappa number increased rapidly (Fig. 2). At an MEA/water ratio of 50/50 the Kappa number attained with a 150% MEA input was around 30, but when the MEA input was lowered to only 50%, the Kappa number increased to 74.

**Fig. 2.** Impact of MEA charge on kappa number and yield

With regards to the strength properties of the MEA pulp produced at 120 °C (Fig. 1), it can be concluded that these pulps with high Kappa numbers also showed less bonding ability, because the chemical fiber modification was not strong enough. Moreover, the results showed that the yield gain obtained by lowering the MEA charge was not very distinct. Even with the lowest MEA charge, a yield of 70% could not be reached. Based on the chemical composition of wheat straw, especially its high content of water and alkali soluble extractives, a high yield above 70% was not expected in a chemical cooking process, not even with the highly selective MEA process. The paddle digester used for the cooking experiments allowed operation at a low liquor/straw ratio of 2/1. The lower liquor/straw ratio resulted in a lower Kappa number than the

corresponding cook performed with the higher ratio (Fig. 2). This result indicated a certain potential to improve MEA pulping also under other pulping conditions, particularly with a higher MEA charge.

As noted before, the solvent in all organosolv pulping processes must be recovered as completely as possible. There are different methods of solvent recovery in organosolv pulping dependent upon the nature and behaviour of the applied solvent. In the case of MEA pulping, the recovery of the charged monoethanolamine is complicated, because of its high boiling point. A comprehensive study on the recovery of MEA from black liquor and characterization of MEA lignin was conducted, and the result will be published soon.

An uncomplicated method of solvent recovery is the direct reuse of the black liquor in the subsequent cook as part of the cooking liquor. In another series of MEA cooks, 30% of MEA charge was added in the form of recycled black liquor. This procedure was performed twice to learn how cooking was affected by this measure. The results are summarized in Table 3. The Kappa number rose with black liquor recycling, from 31 to 33 after first recycling and to 36 after the second recycling. As expected, the Kappa number increase was lower. Both the yield and pulp strength remained unaffected. Presumably, partial black liquor recycling can be regarded as an option to facilitate MEA recycling, but further studies, especially more recycling loops, are necessary to make that conclusion.

**Table 3.** Impact of Applying Recycled Black Liquor on MEA High Yield Pulping of Wheat Straw

Liquor/Straw Ratio	MEA Charge	Total Yield (%)	Kappa Number	Brightness (%ISO)
3/1	100% fresh MEA	64	31	23.4
3.3/1	70% fresh MEA 30% MEA in recycled black liquor I	65	33	21.8
3.3/1	70% fresh MEA 30% MEA in recycled black liquor II	64	36	21.9
Constant: Cooking temperature: 130 °C; cooking time: 30 min; MEA charge 150%				

For further experiments on refiner beating, the pulp obtained in MEA cooking at 130 °C with 150% MEA charge, but without black liquor recycling, was selected. After cooking, the pulp was first pressed in a water press (0.2 MPa) to separate the black liquor for reuse in cooking, then it was washed with hot tap water. The pulp was refined with a Voith LR40 refiner to study the refinability of the MEA high yield pulp under industrial-like conditions. The refining parameters are summarized in Table 4.

The LR40 refiner made it possible to control the specific edge load (intensity) and net specific energy. With these two parameters, the response of pulp on refining could be well studied. Table 5 shows the impact of the refining application of a net specific energy range from 0 kWh/t to 120 kWh/t with a constant specific edge load of 0.3 J/m on the mechanical properties of the selected MEA pulp. The basic strength properties developed were similar to the beating performed in a Jokro mill (Fig. 1), but the strength losses that occurred in refiner beating were higher (Fig. 3).



**Table 4.** Refining Conditions for MEA High Yield Wheat Straw Pulp

No.	1	2	3	4	5	6
Refining time (s)	0	108	225	339	448	554
Inlet pressure (bar)	1.1	1.0	1.1	1.2	1.3	1.3
Total load power ( kW)	1.63	2.47	2.56	2.55	2.55	2.53
Net refining power (kW)	0	0.84	0.93	0.92	0.92	0.90
Total spec. energy (kWh/t)	0	59	128	198	267	337
Net spec. energy (kWh/t)	0	20	45	70	95	120
Temperature (°C)	47.9	49.4	50.9	52.3	53.7	55.0
Beating degree (°SR)	32.0	38.5	40.5	48.0	51.0	55.0
Constant: Refiner speed (rpm): 2000; defibration time (min): 2; dwelling time (min): 5; consistency (%): 4.0; spec. edge load (J/m): SEL 0.30						

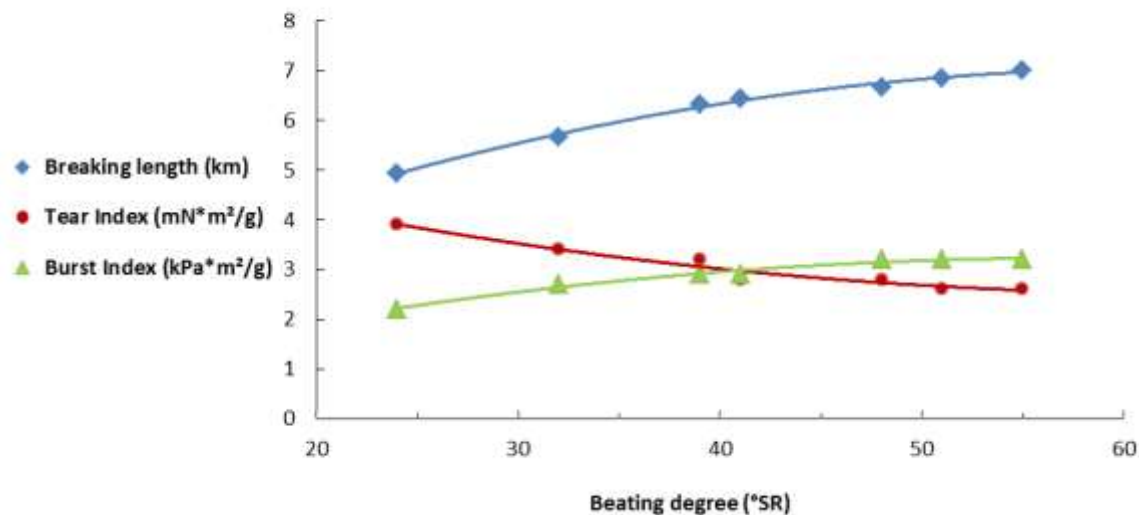
A certain loss in strength was explained by the changed finishing-off procedure after cooking, which refined the rejects together with the accepted pulp fraction without separation. The main reason for the lower strength after refiner beating was the more intense fiber damage and shortening at less internal and external fiber fibrillation. In the case of wood pulp, especially softwood kraft pulp, the decline of pulp strength during refiner beating was considerably greater due to the different fiber structure and the required, much longer refining treatment. In the case of wheat straw pulp, a very short refining treatment was sufficient to reach the targeted beating degree or breaking length. Only 4 min of beating the MEA high yield straw pulp was enough to obtain a breaking length of over 6 km. This also meant a large amount of refining energy was saved compared to the refining of softwood kraft pulp. This energy could alternatively be used as reinforcement pulp for recycled fibers.

**Table 5.** Mechanical Properties of Wheat Straw MEA High Yield Pulps

	Unrefined	1	2*	3	4*	5	6*
Beating Degree (° SR)	23.5	32.0	38.5	40.5	48.0	51.0	55.0
Breaking Length (km)	4.9	5.7	6.3	6.4	6.7	6.8	7.0
Tensile Index (Nm/g)	48,3	55.7	62.1	63.1	65.2	67.1	68.8
Tear Index (mN*m <sup>2</sup> /g)	3.9	3.4	3.2	2.8	2.9	2.6	2.6
Burst Index (kPa*m <sup>2</sup> /g)	2.2	2.7	2.9	2.9	3.2	3.2	3.2
Porosity (mL/min)	362.4	185.6	75.1	51,9	20.9	10.8	0.7
Air Permeability (Gurley) (s)	33.4	69.9	160.8	231.2	878.9	4276	5129
* Selected samples for next step							

Straw pulp is very susceptible to mechanical impact. Therefore, refining straw pulp must be conducted carefully to avoid serious deterioration of the dewatering efficiency on the paper machine. In this case, the beating degree increased from 23.5 °SR to 32 °SR just by the defibration treatment in the refiner. In the course of refining, the

fast increase of the beating degree was associated with a strong decline of the pulp porosity and air permeability. Therefore, refining should be kept very short to avoid dewatering problems. Under industrial conditions straw pulp is often not refined at all.



**Fig. 3.** Development of strength properties of wheat straw MEA high yield pulp during refiner treatment

For this specific application, the high specific bond strength and good bonding-related strength properties of reinforcement pulp were important, and thus some refining was necessary. The extent of refining should be a compromise between strength development and deterioration of drainage. The strength increment at prolonged beating was moderate, but the dramatic changes in porosity and air permeability indicated a strong decline in the dewatering ability. Therefore, a beating degree of 40 °SR should not be exceeded. To obtain more information to support this statement, the morphological properties of the refined MEA straw pulp samples were analyzed (Table 6).

**Table 6.** Morphological Fiber Properties of Refined Wheat Straw MEA High Yield Pulps

No.	1	2*	3	4*	5	6*
Net Spec. Energy ( kWh/t)	0	20	45	70	95	120
Fiber Length (l) (mm)	0.70	0.67	0.65	0.64	0.60	0.57
Fiber Width (μm)	19.7	19.9	20.0	20.5	20.3	20.6
Fiber CWT (μm)	6.2	6.3	5.8	7.2	6.8	6.9
Fiber Curl (%)	16.3	14.9	14.7	16.1	17.0	16.3
Fiber CSA (μm <sup>2</sup> )	565	599	515	704	655	649
Fibrillation (%)	16.5	17.6	18.8	19.0	19.2	19.7
Kink Index (l) (1/m)	1244	1078	10718	1226	1343	1260
Fines (l) (%)	15.4	15.7	15.7	15.8	17.0	17.6
* Selected sample for next step						

With increased energy input, a steady decline of the fiber length took place, but fiber shortening remained in a narrow limit due to the low refining intensity applied. Nonetheless, the initially low tear strength of the straw pulp, which was of marginal importance for the aimed application, was further reduced due to weakening of the individual fibers during refining (Fig. 3). Long fibers have more binding sites and can create a stronger network, which is especially important when recycled fibers with poor bonding ability should be reinforced. Moreover, the flexible straw fibers could improve the network strength additionally by mechanical entanglement. Thus, the moderate reduction of fiber length was beneficial for good tensile and burst strength. Straw pulp showed high heterogeneity and had a high initial content of fines due to the parenchyma cells, vessel fragments, and other small particles originating mostly from the leaves fraction of the straw plant. With refining, secondary fines are formed, which bring the fibers into even closer contact and thus increase the bonding area. In the course of refining, the external fibrillation increased steadily. The carbohydrate rich fibrils released from the secondary fiber wall make the fiber surface rougher and also increase the bonding area.

**Table 7.** Experimental Design of the Blending Tests

Factors	Low	Medium	High	Unit
Mixing level	5	12.5	20	% MEA straw pulp
Refiner energy	20	70	120	kWh/t
Responses: Beating degree ( $^{\circ}$ SR), porosity (mL/min), tensile index (Nm/g), tear index (mN*m <sup>2</sup> /g), burst index (kPa*m <sup>2</sup> /g), and SCT index (Nm/g)				

**Table 8.** Experimental Design of the Mixing Series and Selected Results from Paper Testing

Run	Mixing Level (%)	Refining Energy (kWh/t)	Beating Degree ( $^{\circ}$ SR )	Tensile Index (Nm/g)	Burst Index (kPa*m <sup>2</sup> /g)	Tear Index (mN*m <sup>2</sup> /g)	SCT Index (Nm/g)	Porosity (mL/min)
1	20	70	34.0	36.8	2.0	5.5	23.2	512
2	5	20	31.0	30.4	1.5	5.4	20.6	1108
3	12.5	70	35.0	34.2	1.8	5.7	22.4	732
4	5	120	31.5	31.1	1.6	5.6	20.2	933
5	12.5	20	32.5	31.2	1.7	5.9	20.9	847
6	12.5	70	33.5	33.8	1.7	5.8	20.5	707
7	20	20	34.5	35.3	1.9	5.6	22.4	618
8	20	120	38.5	37.9	2.0	5.5	24.1	378
9	5	70	33.5	30.9	1.5	5.7	19.4	1049
10	12.5	70	35.0	32.1	1.6	5.6	20.6	733
11	12.5	120	36.0	34.8	1.7	5.8	21.2	652
100 % Rec.	-	-	28.0	31.1	1.4	5.7	19.2	1446

These fiber modifications improved tensile and burst strength. The morphological fiber modifications during refining confirmed that gentle refining of MEA high yield pulp was advisable. It improved flexibility and conformability of the fibers, which allowed them to easily flatten under compression during papermaking. These ribbons form fibers that improve inter-fiber bonding. At excessive beating, however, too many fines and fibrils formed and the dewatering properties were adversely affected. A certain increase of the dewatering time was accepted, when the straw pulp was added in a small percentage as blending material to the recycled fibers.

To examine whether MEA high yield straw pulp could actually be used as reinforcement pulp in the production of packaging paper, it was added to the recycled fibers (B12 grade). In this series, unrefined recycled fibers were used as a matrix to work out clear effects. It can be assumed that the effects were less distinct if the recycled fibers were beaten before blending with the straw pulp or together with it, which was feasible because both furnish components only needed gentle beating. Effects of the mixing level and the energy input during refiner beating varied within the scope of a small test program (Table 7). Results are summarized in Table 8, and the correlation coefficients of responses are given in Table 9.

**Table 9.** Fitted Correlation Coefficient and p-values for Beating Degree, Tensile Index, Tear Index, Burst Index, SCT Index, and Porosity

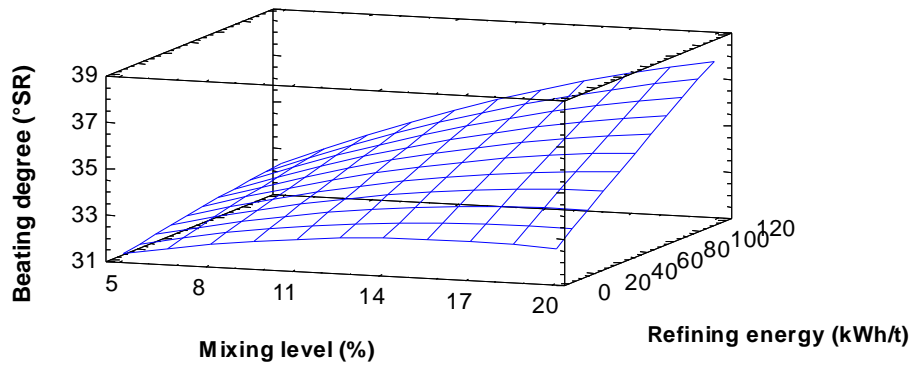
Selected Factors	Beating Degree		Tensile Index		Burst Index	
	Estimate	p-value	Estimate	p-value	Estimate	p-value
A	10.47	0.0231	68.32	0.0004	52.81	0.0008
B	5.54	0.0653	10.50	0.0229	1.25	0.3144
AA	0.40	0.5541	1.14	0.3337	1.19	0.3255
AB	1.59	0.2630	1.19	0.3243	0.00	1.0000
BB	0.00	0.9542	0.24	0.6471	0.00	1.0000
$R^2$	0.783		0.942		0.917	
$R^2$ Adj.	0.566		0.884		0.834	
	Tear Index		SCT Index		Porosity	
	Estimate	p-value	Estimate	p-value	Estimate	p-value
A	0.10	0.7604	29.20	0.0029	1077.42	0.0000
B	0.00	1.0000	0.83	0.4031	159.74	0.0001
AA	7.52	0.0406	1.30	0.3065	6.69	0.0491
AB	1.40	0.2898	1.97	0.2191	2.76	0.1575
BB	0.16	0.7080	0.35	0.5786	0.01	0.9371
$R^2$	0.647		0.872		0.996	
$R^2$ Adj.	0.293		0.745		0.992	

A: Mixing level; B: Refining energy

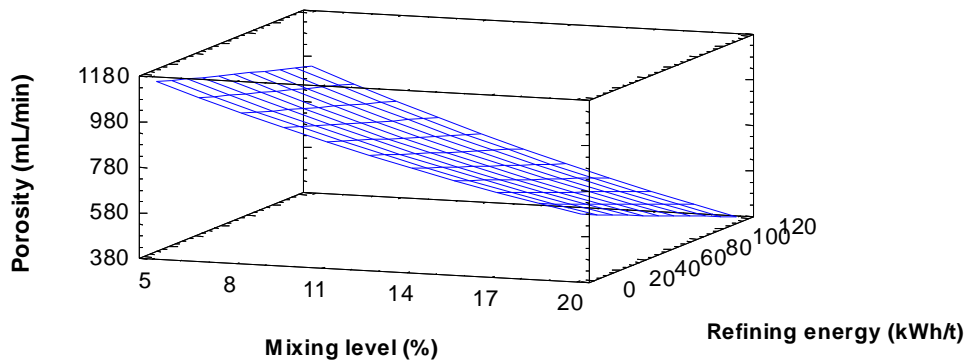
The statistical evaluation was illustrated in Fig. 4 in the form of different three-dimensional plots that revealed the expected effects. The addition of low refined MEA straw pulp increased the beating degree moderately, but the effect was considerably enhanced by more intense refining. A corresponding

development was given for pulp porosity and thus for the dewatering ability. The reduction of drainage was moderate at a 5% addition of wheat straw pulp.

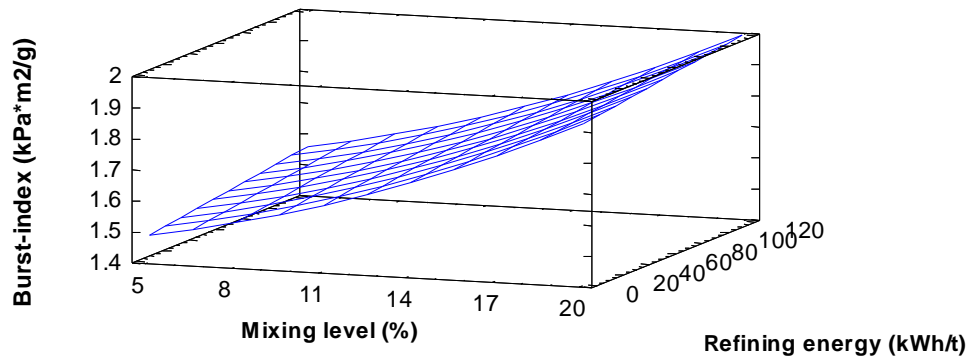
A

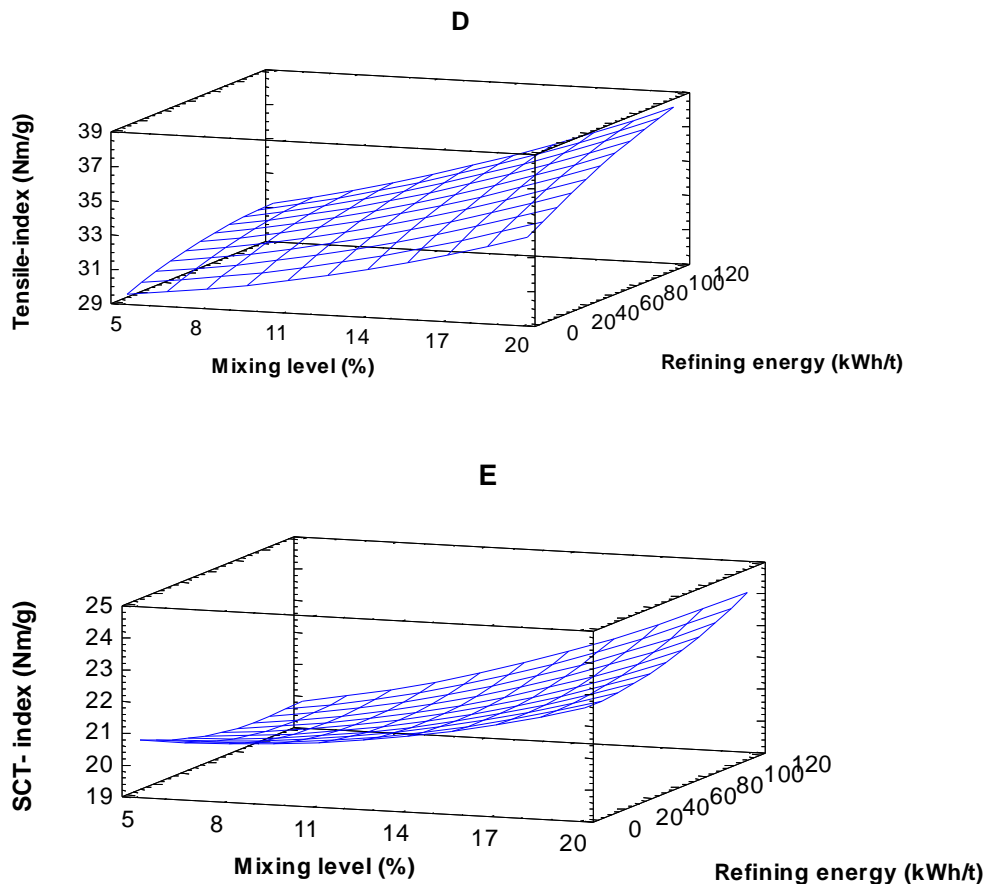


B



C





**Fig. 4.** Response surfaces of (A) beating degree, (B) porosity, (C) burst index, (D) tensile index, and (E) SCT index

At a higher addition and higher energy input during refining, the porosity was further reduced and could become a critical factor with respect to the dewatering properties, especially if the recycled fibers were also refined to a higher beating degree.

With regards to the tear strength, the effect of adding MEA straw pulp seemed to be indifferent. With regards to the bonding-related strength properties, the addition of the straw pulp showed a clear positive effect that was enhanced by the mixing level and the refining energy. The improvement of the SCT index indicated that the compression strength, which is of particular importance for packaging paper, could be improved up to 25% at the addition of straw pulp with highest beating degree. As mentioned before, a balance must be found between improvement of the strength properties and the reduction of dewatering ability. With this in mind, the realistic improvement of the compression strength by the addition of MEA straw pulp was clearly lower and depended on the acceptable extent of drainage reduction.

## CONCLUSIONS

1. To fulfil the requirements of high yield but low lignin content, the very selective MEA process was chosen. It was shown that the yield in MEA straw pulping was considerably enhanced by reducing the cooking temperature to 130 °C at a short cooking time of only 30 min.
2. In comparison to soda cooks performed as a reference, a much better yield to Kappa number ratio was achieved. The high yield MEA pulp showed high bonding-related strength properties. Blending tests with recycled fibers revealed a good suitability as a reinforcement pulp.
3. The promising results must be verified by further, more detailed investigations on a laboratory, pilot, and mill scale. Furthermore, the research on black liquor recovery and lignin utilization must be continued with enhanced intensity to arrive at a coherent concept.

## ACKNOWLEDGEMENTS

The authors wish to thank the University of Hamburg and Thünen Institute of Wood Research for their contributions.

## REFERENCES CITED

- Alanko, K., Paulapuro, H., and Stenius, P. (1995). "Recyclability of thermomechanical pulp fibers," *Paperi ja Puu [Paper and Timber]* 77(5), 315-328.
- Billosta, V., Brandström, J., Cochaux, A., Joseau, J. P. and Ruel, K. (2006). "Ultrastructural organization of the wood cell wall can explain modification caused in fiber during the pulping process," *Cellulose Chem. and Tech.* 40(3-4), 223-229
- Cao, B., Tschirner, U., and Ramaswamy, S. H. (1998). "Impact of pulp chemical composition on recycling," *TAPPI Journal* 81(12), 119-127.
- Casey, J. P. (1960). *Pulp and Paper, Chemistry and Chemical Technology* (2<sup>nd</sup> Ed. Vol. II), Interscience Publisher Inc., New York.
- Chatterjee, A., Lee, J., Roy, D. N., and Whiting, P. (1991). "Effect of recycling on the surface characteristics of paper," in: *Proceedings of the TAPPI International Paper Physics Conference*, Kona, HI, pp. 129-142.
- Corson, S. R. (1980). "Fibre and fine fractions influence strength of TMP," *Pulp and Paper Canada* 81(5), 69-76.
- Diniz, J. M. B. F., Gil, M. H., and Castro, J. A. A. M. (2004). "Hornification – Its origin and interpretation in wood pulps," *Wood Sci. and Tech.* 37(6), 489-494. DOI: 10.1007/s00226-003-0216-2
- Ellis, R. L., and Sendlachek, K. M. (1993). "Recycled versus virgin-fiber characteristic: A comparison," in: *Secondary Fiber Recycling*, R. J. Spangenberg (ed.), TAPPI Press, Atlanta, GA, pp. 7-19.
- Ferguson, L. D. (1992). "Effect of recycling on mechanical RFP strength properties," *Paper Technology* 33(10), 14-20.

- Freeland, S. (1995). "Caustic Soaking: A pilot plant trial," in: *Recycling Symposium Proceedings*, pp. 117-123.
- Hedjazi, S., Kordsachia, O., Patt, R., and Kreipl, A. (2009). "MEA/water/AQ-pulping of wheat straw," *Holzforschung* 63, 505-512. DOI: 10.1515/hf.2009.110
- Horn, R. A. (1975). "What are the effects of recycling on fiber and paper properties?," *Paper Trade Journal* 159 (7/8), 78-82.
- Howard, R. C. (1990). "The effect of recycling on paper quality," *Journal of Pulp and Paper Science* 16(5), 143-149.
- Howard, R. C., and Bichard, W. J. (1992). "The basic effect of recycling on pulp properties," *Journal of Pulp and Paper Science* 18(4), 151-159. DOI: 10.1557/proc-266-195
- Htun, M., and de Rovo, A. (1978). "The implication of fines fraction for the properties of bleached kraft sheet," *Svensk Papperstidning* 81(16), 507-510.
- Hubbe, M. A., and Zhang, M. (2005). "Recovered kraft fiber and wet-end dry-strength polymers," in: *Proceedings of the TAPPI 2005 Practical Papermakers Conference*, Atlanta, GA, USA.
- Hubbe, M. A., Venditti, R. A., and Rojas, O. J. (2007). "What happens to the cellulosic fibers during papermaking and recycling? A review," *BioResources* 2(4), 739-788. DOI: 10.15376/biores.2.4.739-788
- Jahan, M. S. (2003). "Changes of paper properties of nonwood pulp on recycling," *TAPPI Journal* 2(7), 9-12.
- Katz, S., Liebergott, N., and Scallan, A. M. (1981). "A mechanism for the alkali strengthening of mechanical pulps," *TAPPI Journal* 64(7), 97-100.
- Laine, J., Lindström, G., Nordmark, G., and Risinger, G. (2002). "Studies on topochemical modification of cellulose fibers. Part 5. Comparison of the effect of surface and bulk chemical modification and beating on pulp and paper properties," *Nordic Pulp and Paper Research Journal* 18(3), 325-332. DOI: 10.3183/NPPRJ-2003-18-03-p325-332
- Laiwins, G. V., and Scallan, A. M. (1996). "The influence of drying and beating on the swelling of fines," *Journal of Pulp Paper Science* 22(5), 178-184.
- McKee, R. C. (1971). "Effect of repulping on sheet properties and fiber characterization," *Paper Trade Journal* 155(21), 34-40.
- Nazhad, M. M., and Paszner, L. (1994). "Fundamentals of strength loss in recycled paper," *TAPPI Journal* 77(9), 171-179.
- Nazhad, M. M. (2004). "The influence of refining energy and intensity on enhancing the bonding potential of an OCC pulp," *Appita Journal* 57(3), 191-198.
- Nazhad, M. M. (2005). "Recycled fiber quality – A review," *Journal of Industrial and Engineering Chemistry* 11(3), 314-329.
- Oksanen, T., Buchert, J., and Viikari, L. (1997). "The role of hemicelluloses in the hornification of bleached kraft pulps," *Holzforschung* 51(4), 355-360. DOI: 10.1515/hfsg.1997.51.4.355
- Puls, J. (1983). "Chemical analysis of lignocellulosic residue," in: *Energy from Biomass: 2nd E.C. Conference: Proceedings of the International Conference on Biomass*, A. Strub, P. Chartier, G. Schleser (eds.), Applied Science Publishers Ltd., London, pp. 863-867.
- Salam, A. (2015). *The Synthesis, Characterization, and Application of Polysaccharides-based Additives to Increase the Dry Strength of Paper*, PhD Thesis, Forest Biomaterials, North Carolina State University, Raleigh, NC, USA.



- Salehi, K., Kordsachia, O., and Patt, R. (2014). "Comparison of MEA/AQ, soda and soda/AQ pulping of wheat and rye straw," *Industrial Crops and Products* 52, 603-610. DOI: 10.1016/j.indcrop.2013.11.014
- Strümer, L., and Götttsching, L. (1979). "Physical properties of secondary fiber pulps under the influence of their previous history. Part 3: Influence of the paper manufacturing process," *Wochenblatt für Papierfabrikation*. 107(3), 69-76.
- TAPPI T222 om-02. (2007). "Acid-insoluble lignin in wood and pulp," TAPPI Press, Atlanta, GA.
- TAPPI T211 om-02. (2007). "Ash in wood, pulp, paper, and paperboard: Combustion at 525°C," TAPPI Press, Atlanta, GA.
- TAPPI T220 sp-01. (2007). "Physical testing of pulp handsheets," TAPPI Press, Atlanta, GA.
- TAPPI T826 om-04. (2007). "Short span compressive strength of containerboard," TAPPI Press, Atlanta, GA.
- Yamagishi, Y., and Oye, R. (1981). "Influence of recycling on wood pulp fiber – Changes in properties of wood pulp fiber with recycling," *Japan TAPPI Journal* 35(9), 787-797. DOI: 10.2524/jtappij.35.787
- Welf, E. S., Venditti, R. A., Hubbe, M. A., and Pawlak, J. J. (2005). "The effects of heating without water removal and drying on swelling as measured by water retention value and degradation as measured by intrinsic viscosity of cellulose papermaking fibers," *Prog. Paper Recycling* 14(3), 1-9.
- Zellcheming IV/37/80 (1980). "Determination of the Kappa number," ZELLCHEMING Association, Weiterstadt, Germany.
- Zellcheming V/12/57 (1957). "Determination of the breaking length, tear and burst of paper," ZELLCHEMING Association, Weiterstadt, Germany.
- Zellcheming V/5/60 (1960). "Beating of pulp," ZELLCHEMING Association, Weiterstadt, Germany.
- Zellcheming V/7/61 (1961). "Determination of the beating degree of pulp," ZELLCHEMING Association, Weiterstadt, Germany.

Article submitted: March 6, 2017; Peer review completed: June 24, 2017; Revisions accepted: July 16, 2017; Published: September 19, 2017.

DOI: 10.15376/biores.12.4.8255-8271