Helianthus annus Waste Stalks, as a Substitute Raw Material for Mixed Hardwood Semi-Chemical Pulp

Hamidreza Rudi,[a,]* Hossein Resalati,[b] and Mohsen Shekarbeigi [c]

Agricultural wastes, such as *Helianthus annus* (sunflower), have begun to receive considerable attention in the forest industry in recent years to alleviate the shortage in woody raw materials. This paper mainly focuses on the influence of partial replacement of mixed hardwood NSSC pulp with sunflower stalk NSSC pulp. Experimental cooks were implemented on chips prepared from de-pithed stalks. Optimum cooking obtained reflecting a pulp yield of 43.8±2.08% and kappa number of 82.6±1.67. The pulps were refined and then substituted with mixed hardwood NSSC pulp at three levels of 10, 20, and 30%. The signs of improvement in most paper properties showed the benefits of using sunflower NSSC pulp with freeness of 328 mL CSF and 372 mL CSF up to 30% as a supplementary pulp with MWPI NSSC pulp in making fluting paper. The exclusive 30% of the higher freeness pulp (372 mL CSF) can be applied, provided that at least 5% of unbleached long fiber pulp be added to the mixed pulp.

Keywords: Fluting paper; MWPI; Nonwood plants; Pulp; Sunflower stalk; Tensile index

*Contact information: a: Department of Cellulose and Paper Technology, Faculty of New Technologies and Energy Engineering, Shahid Beheshti University, Zirab, Mazandaran, Iran; b: Department of wood and Paper science and technology, Faculty of Natural Resources, Sari University of Agricultural Sciences and Natural Resources, Iran; c: M.Sc. of Wood & Paper Science and Technology,*Corresponding author:h_rudi@sbu.ac.ir*

INTRODUCTION

The rapid increase in the population of developing countries and the demand for different grades of paper and paper products has led to greater and diverse utilization of non-wood fiber resources, especially agricultural-based fibers (Jahan Latibari *et al.* 2011). Non-wood fibers account for around 10% of worldwide pulp production, and in some regions like Asia, these are the only or main pulp fibers (FAO 2005). The use of non-wood fibers for paper production gains importance in order to reduce wood consumption (Kasmani *et al.* 2011). Hence, several studies examined the feasibility of substituting wood-based materials with crop residues from annual plants to produce pulp and paper (Wong 1995; Atik 2002; Rodriguez *et al.* 2008; Samariha and Khakifiroz 2011; Matin *et al.* 2015; Sharma *et al.* 2015).

Helianthus annus (sunflower) is an annually harvested non-wood fiber that can be grown in many regions in Iran. This species is a promising industrial crop on account of the oil production from its seeds (Anonymous 2005), and the stalks as an agricultural residue is mostly unused and burnt. According to the some reports this species contains fibers with desired dimensions and the major chemical components in reasonable amounts for pulp production when compared with other nonwood plants and wood species (Eroglu *et al.* 1992; Jemenez *et al.* 1993; Khristova *et al.* 1998; Law and Jiang 2001; Ververis *et al.* 2004; Rodriguez *et al.* 2008). Moreover, some literature has demonstrated that the fibers of the sunflower stalk are promising raw material for the production of pulp (Eroglu *et al.* 1992; Jemenez *et al.* 1993; Khristova *et al.* 1998; Lopez *et al.* 2005), which can be used to produce, for example, liner and fluting paper (Marchael and Rigal 1999).

Fluting paper is one of main products in Mazandaran wood and paper industries (MWPI) factory, the major Iranian paper producer manufacturing 85000 tons/year, 113-175 g/m^2 fluting paper. This product comes from a pulp production line that generates approximately 300 tons/day NSSC pulp from mixed Northern hardwoods. The present available fiber supply is quite lacking, and can meet roughly one third of the fiber needs for the factory. The use of non-wood fibers resource such as sunflower woody stalk seems a hopeful approach may initiate a partial solution for the intensive fiber scarcity.

Therefore, the main purpose of this study was to prepare NSSC pulp from sunflower stalk and investigate the possibility of partially substitute of the MWPI NSSC pulp with the sunflower pulp to produce fluting paper. Additionally, the strength-enhancing effect of adding long fiber was examined when a drop of strengths in the mixed sunflower and MWPI pulp was detected.

EXPERIMENTAL

Materials

Fibers

Sunflower stalks were collected from the nearby vicinity of Behshar and Neka, Northern cities of Iran. The leaves and branches were separated from the stalks. The stalks were then delivered to the R&D lab in MWPI factory, de-pithed, chopped into about 3 to 5 cm long chips manually and air-dried until used.

Fiber characterization

Fiber dimensions of sunflower stalk were measured according to the Franklin method (1954), using a calibrated microscope, IMT-2Olympus. Each measurement was implemented on 30 randomly chosen fibers. For chemical characterization, samples were milled to pass through a 40-mesh screen and retained on 80-mesh screen, using a vibrating screen, Haver EML 200 digital T. Relevant TAPPI test methods were used as follows: cellulose, using acid nitric method (Rowell and Young, 1997), 1 % NaOH soluble (T212), lignin (T222), ash (T211), and extractives (T204). All measurements were repeated three times.

Pulping

Experimental cooks were performed with NSSC white liquor including sodium sulfite (Na_2SO_3) and sodium carbonate (Na_2CO_3) at a 4 to 1 weight ratio sulfite to carbonate on 250 grams of stalk chips in each trial using a 10 liter experimental rotating digester with electrical heating (HATTO). The cooked chips were separated from the spent liquor by washing on a 200-mesh screen and then disintegrated in a kitchen mixer for 90 seconds.

After pulping, optimum cooking conditions were selected with respect to the pulp yield and kappa number. Corresponding TAPPI standard methods used in this section including: Consistency (T240), freeness (T227), pulp yield (T257), and kappa number (T236).

NSSC pulp from sunflower stalk was refined using a Labtech PFI Mill according to T248-cm-85 standard until NSSC pulp with primary freeness of 613 mL CSF reached to two final freeness of 328 and 372 mL CSF with respect to the acceptable shive content at MWPI industries company (≈2.5 %). Factory NSSC pulp obtained a freeness of about 414 mL CSF at 6400 revolutions and was considered as control pulp, and imported unbleached long fiber pulp was refined to freeness of 500 mL CSF. Recently the described type of pulp was only added to the mixture of mill and sunflower NSSC pulp with freeness of 372 mL CSF, where a drop in strengths has been detected in the resultant papers.

Table 1 represents the different scenarios of mixing the pulps in this study exploring the potential of partial substitute of MWPI pulp with sunflower pulps up to 30 percent. The shive content of the pulp was determined using 5 grams of pulp processed in an automatically Shive Analyzer to isolate the uncooked materials. Fines content of the pulps was measured using a Messmer Buechel fiber classifier according to the standard method, TAPPI 233-cm-82.

Table 1. Different Scenarios of Mixing the MWPI, Sunflower and Long Fiber Pulps

MWPI NSSC 414 mL CSF	Sunflower NSSC 328 mL CSF	Sunflower NSSC 372 mL CSF	Unbleached long fiber 500 mL CSF
100	0	0	0
90	10	0	0
80	20	0	0
70	30	0	0
90	0	10	0
80	0	20	0
70	0	30	0
65	0	30	5
60	0	30	10

Preparation and characterization of handsheets

Handsheets with grammage of 127±5 g/m^2 were made of both control and experimental pulps using a laboratory sheet maker, Labtech Handsheet Maker and pressed at two steps as 30 psi for 5 min and 50 psi for 2 min. Handsheets after air-drying were conditioned according to SCAN-P2:75 at a constant temperature of 23±1˚C and relative humidity of 50±2 % before testing. Characterization experiments involved the following parameters: sheet making (TAPPI 205), basis weight (TAPPI 410), density (TAPPI 426), air permeability (TAPPI 547), tensile index, breaking length and stiffness (TAPPI 404), tear index (TAPPI 414), burst index (TAPPI 403), CMT (TAPPI 809), and

RCT (TAPPI 818). Finally, the average ± standard deviation values of 10 handsheets have also been represented.

RESULTS AND DISCUSSION

Fiber Characterization

The fiber dimension measurements of sunflower stalk are summarized in Table 2. The results of fiber biometry show that sunflower stalk contained rather short (0.96±0.30 mm long) and thin-walled (5.9±0.3 µm thick) fibers. However, the fiber length and cell wall thickness of sunflower stalk were greater than those of some nonwood and hardwood species such as cotton, and also higher than that of hardwoods fibers such as olive tree (Ververis *et al.* 2004).

Table 2. Dimensions of Sunflower Fibers

Fiber dimensions	Value, %
length	0.96±0.30 (mm)
Cell diameter	23.7±0.5 (µm)
Lumen diameter	11.9±0.8 (µm)
Cell wall thickness	5.9±0.3 (µm)

The chemical composition of sunflower stalk is given in Table 3. The cellulose content of sunflower stalk was found to be 47.37±0.8 %, which lies within the acceptable range (≥40%) for pulp, but the ash content (7.5±0.4) and extractives (3.61±0.3) were high, which is common in nonwood plants. In addition, the value measured for 1% NaOH soluble substances corresponds to the amount of low-molecular-weight carbohydrates consisting mainly of hemicellulose and degraded cellulose in wood and pulp (TAPPI 212). It was measured to be about 34±1.6 %, which was relatively high, as expected for nonwood fibers (Eroglu *et al.* 1990). Based on this composition, a low pulp yield and high kappa number may be anticipated (Jahan Latibari *et al.* 2011).

Table 3. Chemical Composition of Sunflower Fibers (% OD basis)

Fiber dimensions	Value, %
cellulose	47.37±0.8
lignin	21.2±2.1
ash	7.5±0.4
Extractives 1% NaOH solubles	3.61±0.3 34±1.6

Pulping Aptitude and Pulp Properties

Table 4 represents the results of NSSC pulping conditions on sunflower stalk. Three levels of chemical charge (10%, 15%, and 20%, as Na_2O and based on O.D. of the stalk) were considered. Cooking times were 120 and 180 minutes a constant pulping temperature of 170 ºC. For each treatment three replicate samples were made. The results show that A low yield and high kappa number pulp can be produced using NSSC process, implying a relatively slow model of delignification from sunflower woody stalks. This effect can be attributed to two factors. First, higher solubility in 1% NaOH (34±1.6) and higher lignin content (21.2±2.1) provides further polysaccharide deterioration without meaningful dissolving the lignin structures. Second, it can be ascribed to the chip quality prepared from the stalk. The presence of biological knots in the node section of the stalk is another suspecting parameter which impair pulp yield (Macleod 2007). Thereby, a sharply decrease in pulp yield was seen from 56.02±2.92% to 43.80±2.08%, varying mild to vigorous cooking conditions, but the kappa number with insignificant change reached from 94.71±2.17 to 82.55±1.67. However, the yield found was slightly lower and the kappa number was higher than those obtained for most other nonwood plants (Abrantes *et al.* 2007; Kaur *et al.* 2010; Khakifirooz *et al.* 2013). Although obtained pulp seems to be used for papers with lower performance such as brown papers, higher kappa number gives the papers more stiffness which is necessary for some grades of papers such as fluting medium (Marchael and Rigal 1999), as intended in present investigation.

Table 4. NSSC Pulping Variables of Sunflower Stalk and the Properties of Resultant Pulps

Pulping variables		Pulp properties	
Chemical charge (Na_2O %)	Cooking time (minute)	Yield (%)	Kappa number
10	120	53.47	92.52
	120	55.38	96.87
	120	59.20	94.73
	Ave.	56.02±2.92	94.71±2.17
15	120	50.28	90.83
	120	47.17	85.55
	120	46.15	89.88
	Ave.	47.87±2.15	88.75±2.81
20	120	48.66	88.57
	120	49.80	88.05
	120	46.60	85.45
	Ave.	48.35±1.62	87.36±1.67
20	180	41.92	80.91
	180	43.46	82.50
	180	46.03	84.25
	Ave.	43.80±2.08	82.55±1.67

The freeness of obtained pulp was high (613 mL CSF) due more to the non-uniform distribution of fibers. Therefore, the fibers were subjected to refining with a laboratory PFI Mill. Consequently, the initial freeness of 613 mL CSF of sunflower stalk NSSC pulp was modified to freeness values of 372 and 328 mL CSF by low refining revolutions of about 150 and 500 PFI-rev. respectively. Consistent reports pointed out that the freeness of pulps prepared from nonwood raw material decreases faster than wood pulps (Farsheh *et al.* 2011). In other words, nonwood pulps require much lower refining energy than wood pulps to reach to the same level of freeness (Fadavi *et al.* 2012). Table 5 shows the fiber length distribution, fines content (less than 200 mesh fraction), and shive content of refined sunflower and factory pulp.

The fractionation process using the Messmer Buechel fiber classifier with screens of 14, 30, 50, 100, and 200 mesh sizes not only separates fibers according to the fiber length, but also, it separates the fraction of fine fibers which passed through the 200 mesh size screen (-200 fraction). MWPI pulp refined to 6400 PFI-rev posing a freeness of 414 mLCSF contained 23.99 % fine fibers while the fine values for sunflower pulps, 372 and 328 mL CSF were 15.17% and 15.46% following refining with 150 and 500 PFI-revolutions, repectively. An inherent problem of nonwood raw materials is the high content of small parenchyma cells, leading to a high level of fines in the pulp (Jahan *et al.* 2007); this poses resistance to water drainage, which may retard their utilization (Oinonen and Koskivirta 1999). However, the higher fine content is expected to contribute with the papers higher strength properties such as breaking length (Hartman 1984; Page 1989), tensile index, and folding endurance (Kamaludin *et al.* 2012).

Table 5. Messmer Buechel Fiber Classification and Shive Content of Used Pulps after Refining

Cup No.	Mesh size	Fiber retained, %		
		MWPI NSSC 414 mL CSF	Sunflower NSSC 372 mL CSF	Sunflower NSSC 328 mL CSF
1	+14	0.44	4.25	3.33
2	-14 to +30	31.93	30.90	28.03
3	-30 to +50	23.64	22.53	24.32
4	-50 to +100	14.63	11.33	11.96
5	-100 to +200	5.37	15.82	16.80
6	-200	23.99	15.17	15.46
Shive content, %		0.26	3.42	2.32

Paper Properties

In order to compare the pulp produced in MWPI (control) and mixed sunflower and mill pulp (experimental), handsheets with the same basis weight, 127±5 g/m^2, were made from both control and experimental samples. Experimental samples were prepared consisting of factory NSSC pulp mixed with up to 30% of sunflower NSSC pulp. Also, up to 10% of imported unbleached long fiber was added to the mixed factory and sunflower pulp (372 mL CSF), to which a drop in physical and strength properties was detected.

Analysis of mill NSSC pulp and mixed sunflower and mill pulp with freeness of (328 mL CSF) showed that addition of sunflower pulp to the factory pulp up to 30% had an increasing effect on physical and mechanical properties of resultant papers. Table 6 represents the variation of paper density and air permeability due to addition of sunflower pulp (328 mL CSF) to the MWPI pulp. In all scenarios, paper density was significantly higher than the control sample. The paper's resistance against air permeation was substantially increased relative to paper made with the control pulp. Air permeability of papers made of mixed pulp having 30% sunflower pulp (328 mL CSF) was measured 8.33±0.55 s, which represents at least a doubling of the values obtained from control paper, 4.57±0.72 Seconds.

Since paper density has also considerable impact on the performance of many paper grades (Gimaker *et al.* 2011); in all treatment addition of sunflower pulp (328 mL CSF) had a rising effect on tensile index, breaking length, stiffness, CMT, and RCT, but tear index and burst index presented a decreasing tendency. Short and thin-walled fibers of sunflower pulp are most likely suspected to deteriorate tear index and burst index of the paper sheets. Table 7 represents the effect of adding the sunflower pulp (328 mL CSF) to the MWPI pulp on strengths characteristics.

Table 6. Physical Properties of Handsheet Papers made from Mixture of MWPI Pulp, Control Sample, (A) and Sunflower Pulp, 328 mL CSF (B)

Type of pulp	Density (g/cm^3)	Gurley (Sec.)
A	0.526±0.012	4.56±0.7
B	0.618±0.140	32.17±1.8
90%A+10%B	0.568±0.014	6.03±1.1
80%A+20%B	0.531±0.010	6.46±0.8
70%A+30%B	0.525±0.026	8.33±0.5

Table 7. Strength Properties of Handsheet Papers made from Mixture of MWPI Pulp, Control Sample, (A) and Sunflower Pulp, 328 mL CSF, (B)

Type of pulp	Tensile index (N.m/g)	Breaking length (Km)	Stiffness (kN/m)	CMT (N)	RCT (kN/m)	Tear index ($mN.m^2/g$)	Burst index ($kPa.m^2/g$)
A	35.11±0.61	3.57±0.04	483±17	186±5.5	1.38±0.09	5.01±0.07	2.13±0.02
B	41.29±2.95	4.20±0.30	685±61	263±10.4	1.94±0.01	6.06±0.05	2.01±0.02
90%A+10%B	35.14±0.49	3.77±0.04	511±12	194±10.1	1.46±0.05	5.80±0.11	2.10±0.06
80%A+20%B	37.02±1.20	3.77±0.02	563±14	196±6.1	1.86±0.07	5.52±0.10	1.98±0.08
70%A+30%B	37.41±4.19	3.81±0.04	567±30	211±8.3	1.82±0.07	5.08±0.08	1.98±0.08

Sunflower pulp with freeness of 372 mL CSF was also added to the MWPI pulp. The results showed that paper density, tensile index, breaking length, and burst index of papers from mixed pulps had been decreased. The measured values for these properties were significantly lower than that of obtained for papers produced from factory pulp. Tables 8 and 9 exhibit the effect of addition the sunflower pulp (372 mL CSF) to the MWPI pulp on physical and strengths properties of the paper sheets.

Table 8. Physical Properties of Handsheet Papers made from Mixture of MWPI Pulp, Control Sample, (A); Sunflower Pulp, 372 mL CSF (E)

Type of pulp	Density (g/cm^3)	Air permeability (Sec.)
A	0.526±0.012	4.56±0.7
E	0.553±0.014	21.77±2.48
90%A+10%E	0.521±0.013	5.30±0.1
80%A+20%E	0.539±0.004	5.60±0.1
70%A+30%E	0.514±0.008	6.13±0.3

Table 9. Strength Properties of Handsheet Papers made from Mixture of MWPI Pulp, Control Sample, (A); Sunflower Pulp, 372 mL CSF, (E)

Type of pulp	Tensile index (N.m/g)	Breaking length (Km)	Stiffness (kN/m)	CMT (N)	RCT (kN/m)	Tear index ($mN.m^2/g$)	Burst index ($kpa.m^2/g$)
A	35.11±0.61	3.57±0.04	483±17	186±5.5	1.38±0.09	5.01±0.07	2.13±0.02
E	34.41±1.01	3.61±0.11	702±30	194±6.0	1.85±0.04	5.71±0.05	1.69±0.04
90%A+10%E	33.12±1.03	3.38±0.03	491±17	194±9.1	1.32±0.03	5.66±0.09	1.32±0.03
80%A+20%E	33.58±0.48	3.50±0.05	535±15	208±4.5	1.39±0.07	5.59±0.06	1.39±0.10
70%A+30%E	32.09±2.03	3.27±0.02	540±33	218±4.0	1.49±0.07	5.48±0.09	1.71±0.07

Since long fibers are commonly used in manufacturing papers with high strength (Sood *et al.* 2005), therefore, the imported unbleached long fiber was added up to 10% to the mixed pulps to rectify the drop of strengths. As illustrated in Figs. 1, 2, 3, and 4, addition of at least 5% unbleached long fiber pulp as a strength-increasing alternative to the mixed pulps of MWPI NSSC and sunflower stalk NSSC (372 mL CSF), with the proportion of 70/30, not only compensate the loss of paper properties, but even higher value of strengths than the papers of MWPI NSSC pulp has been achieved. Moreover, as it would be expected, a significant increase in strengths was obtained by adding of 10 percent of unbleached long fiber pulp to the mixed MWPI and sunflower stalk NSSC pulps.

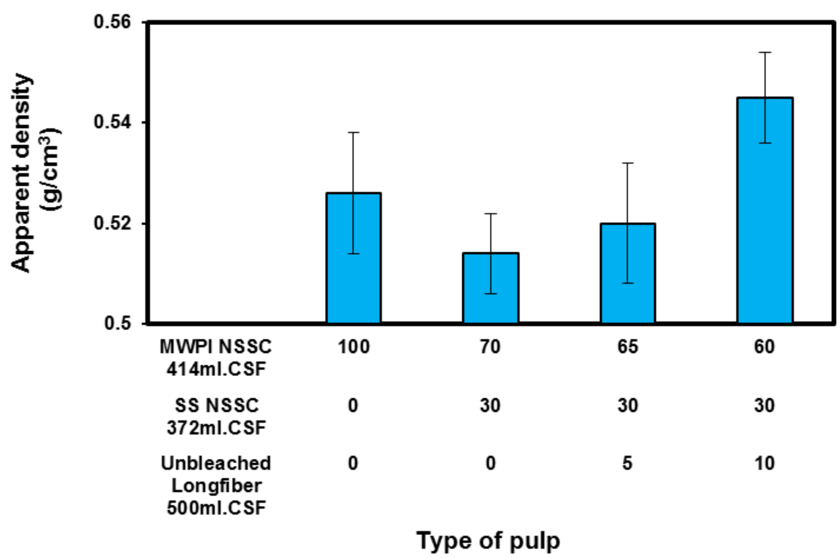

Fig. 1. Effect of adding long fiber on density of papers made of mixed MWPI and sunflower NSSC pulp

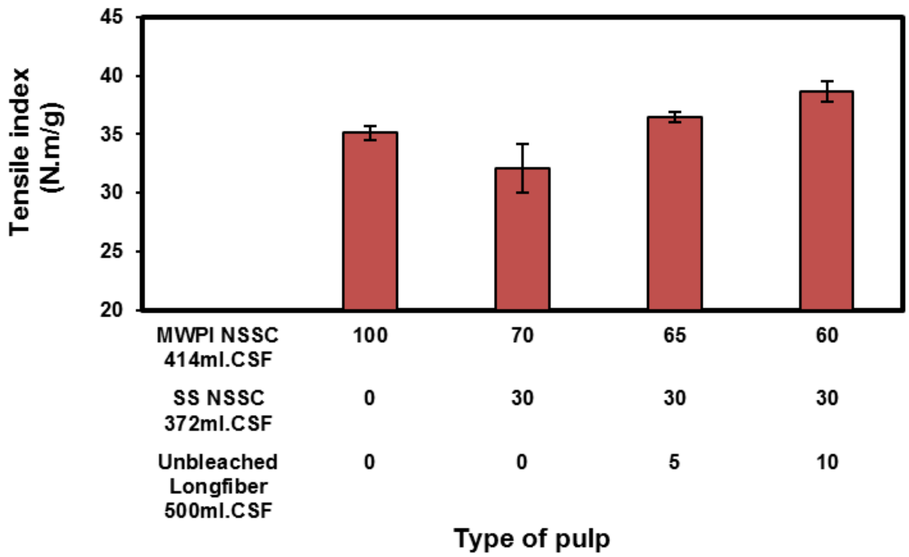

Fig. 2. Effect of adding long fiber on tensile index of papers made of mixed MWPI and sunflower NSSC pulp

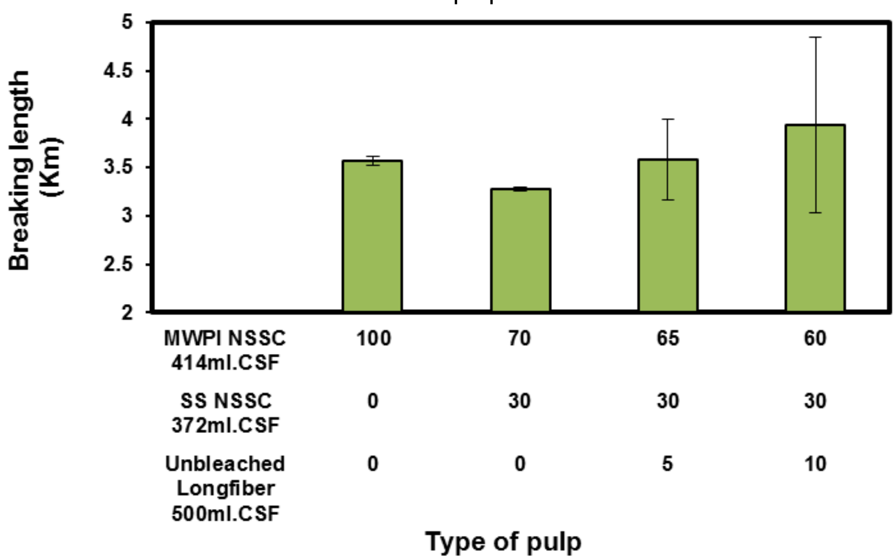

Fig. 3. Effect of adding long fiber on breaking length of papers made of mixed MWPI and sunflower NSSC pulp

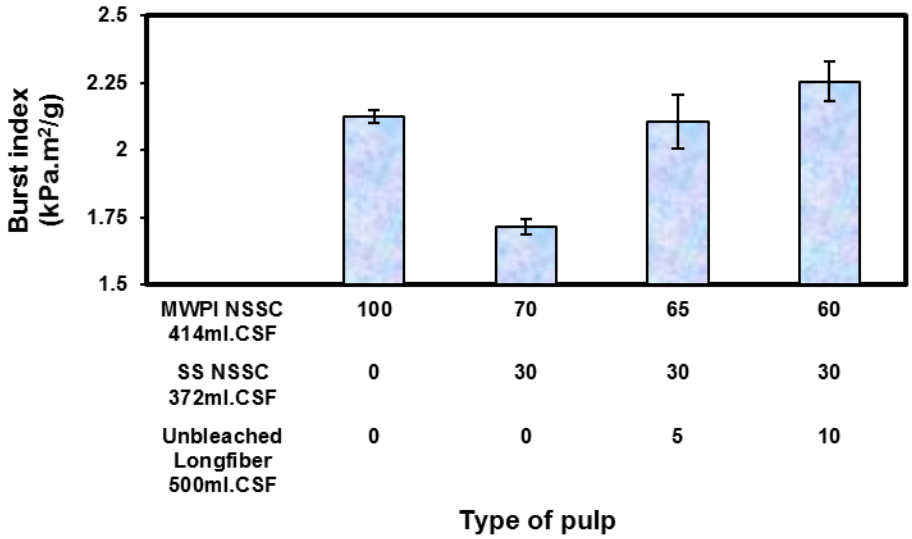

Fig. 4. Effect of adding long fiber on burst index of papers made of mixed MWPI and sunflower NSSC pulp

CONCLUSIONS

1. The overall results showed that the substitution of MWPI NSSC pulp with sunflower stalk NSSC pulp as supplementary pulp can produce fluting papers with physical and mechanical properties improved or very similar to those made by MWPI pulp.
2. Applied scenarios confirm up to 30% replacement provides pulps with acceptable properties.
3. The exclusive addition of 30% sunflower pulp with lower freeness of 328 mLCSF to the MWPI pulp produced results better than those required in production of fluting paper.
4. Addition of 30% sunflower pulp (372 mL CSF) produced results slightly lower (such as burst index) than those obtained from MWPI pulp. Thereby, in such case addition of at least 5% long fiber would be a compensating strategy for loss of the paper properties.

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