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Studies on *Ipomea carnea* and *Cannabis sativa* as an alternative pulp blend for softwood: Optimization of soda pulping process

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This study presents a blend of *Cannabis sativa* and *Ipomea carnea* as a substitute to softwood for soda pulping. Optimized soda pulping conditions are: active alkali, 16% (as Na_2O); max. temp., 165°C; time (at 165°C) 180 min; and liquor to wood ratio, *C. sativa*, 3.5:1 & *I. carnea*, 4.0:1. Screened pulp yields were: *I. carnea*, 47.86% (kappa number 34); and *C. sativa*, 49.50% (kappa number 33). Anthraquinone (AQ, 0.1%) also improves pulp yield and reduces kappa number, respectively, for: *I. carnea*, 0.24%, 8 units; and *C. sativa*, 0.24%, 4 units. At same chlorine demand (5.80%), pulp brightness improved of *I. carnea* (87.1%) and *C. sativa* (84%) by CEHH bleaching sequence. Therefore, *I. carnea* fibers may be used as brightness improver of pulp.

Keywords: Black liquor, Bleaching,*Cannabis sativa*, *Ipomea carnea*, Mechanical strength properties, Soda pulping

Introduction

World demand for paper and paperboard is likely to grow by 2.1% annually in the long term, reaching an estimated 490 million tonnes (MT) by 2020[1]. Presently, India produces about 5.6 MT of paper and board per year. In 2007, total domestic demand for paper stands at 7.2 MT against the production of 6.7 MT and by 2015 demand may reach to 12.5 MT. Annual paper consumption per capita in India is only 8 kg as against world per capita paper consumption of 50 kg[2]. Total fiber consumption for production of paper and paperboard in India will nearly be doubled between 2006 and 2016, growing from 7.4 MT/y to 13.7 MT/y. India's total wood fiber deficit as per forecast is to increase at an 11.3% annual rate through 2016[3]. Therefore, there is need to search new alternative and unexploited sources of fibers[4-6]. Except fibre length, other morphological characteristics (cell wall thickness, lumen diameter, flexibility coefficient and wall fraction) of *Ipomea carnea* Jacq[7], a common weed known as amari, resemble with softwoods[8] (*Pinus kesiya*, *Picca abies*). Such types of fibers collapse readily to double walled ribbons on pressing and exhibit plastic deformation, thus giving more surface contact area for bonding[6,9,10.] On the contrary, *Cannabis sativa* L., a common weed known as soft hemp, contains thin walled and long libriform and lignified fibers[11,12.]. *I. carnea* has grown rapidly in barren wastelands[11]. Proximate chemical analysis[7] and alkali-O_2 delignification studies[5] suggested its suitability for papermaking. *C. sativa* consists of bast fibres[13] (length, 5-50 mm with an average of 20 mm; and width, 7-50 µm with an average of 22 µm) and woody fibers. Long bast fibres were used for sailing and fishing gear[13]. Hemp core fiber[12,14,15] (av fiber length, 1.7 mm) is comparable with hardwood fiber. Dicotyledons like hemp does not have high silica in ash in contrast with monocotyledons like straw or other grasses[14,15].

This study presents a blend of C. *sativa* and *I. carnea* as substitute to softwood for soda pulping.

Materials and Methods

Chips of *C. sativa* (8% bast fibres and 92% core fibres) and *I. carnea* (small slivers) were obtained and macerated with 67% HNO_3 (10 ml) and boiled in a water bath (100±2°C) for 10 min for fiber length determination[16]. Slivers were then washed, placed in small flasks with 50 ml distilled water and fiber bundles were separated into individual fibers using a small mixer with a plastic end to avoid fiber breaking. Macerated fiber suspension was finally placed on a slide (standard, 7.5 cm×2.5 cm) by means of a medicine dropper[17]. For fiber diameter, lumen

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Table 1—Fiber dimensions of *I. carnea* and *C. sativa*.

Particulars	*I. carnea*[22]	*C. sativa*	*Picca abies*	*Pinus kesiya*
Fiber length, mm	0.6	2.42	2.3	2.3
Percent change	(–73.91)a,b	(+5.21)a,b	–	–
Fiber width, µm	33.18	29.5	41.7	40.7
% change	(–20.43)a (–18.48)b	(–29.26)a (–27.52)b	–	–
Cell wall thickness, µm	1.47	6.5	6.0	5.9
% change	(–75.50)a (–75.08)b	(+8.33)a (+10.17)b	–	–
Lumen diameter	30.3	14.5	35.7	34.8
% change	(–15.21)a (–12.93)b	(–59.38)a (–58.33)b	–	–
Wall fraction, 2 x cell wall thickness x100 / Fiber diameter	8.9	44.07	29.0	29.0
% change	(–69.31)a,b	(–54.13)a,b	–	–
Runkel ratio, 2 x cell wall thickness / Lumen diameter	0.10	0.90	0.30	0.50
% change	(–66.67)a (–80.00)b	(+200.00)a (+80.00)b	–	–

a=*Picca abies*, b=*Pinus kesiya*

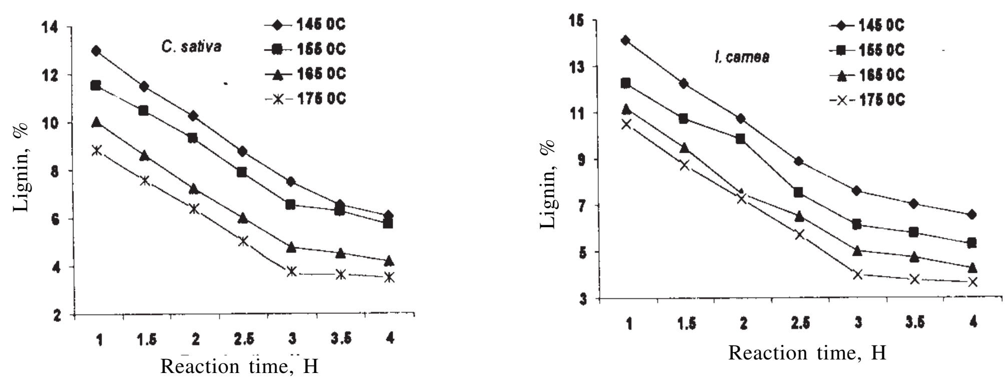

Fig. 1 Effect of temperature on reduction of lignin during soda pulping of *C. sativa* and *I. carnea* at 16% active alkali (as Na_2O)

diameter and cell wall thickness determination, cross-sections were stained with 1:1 aniline sulphate–glycerine mixture to enhance cell wall visibility (cell walls retains a characteristic yellowish colour). All fiber samples were viewed under a calibrated microscope; a total of 25 randomly chosen fibers were measured from each sample for a total of 75 fiber measurements from each stalk. Two derived values were also calculated using fiber dimensions: wall fraction as twice cell wall thickness/ fiber diameter and Runkel ratio as (2×fiber cell wall thickness)/ lumen diameter[16,18] (Table1).

Pulping Studies

I. carnea and *C. sativa*, collected from nearby vicinity of Institute at Saharanpur (India), were air dried and leaves and flowers were removed by stroking on a hard surface. Both plants were chopped manually into 15-25 mm long pieces. Chips of *C. sativa* and *I. carnea* were digested in WEVERK electrically heated rotary digester (capacity, 0.02 m^3) having four bombs (capacity, 1 l each). *C. sativa* and *I. carnea* were cooked at different cooking conditions [max. temp., 145-175°C; cooking time, 1-4 h; active alkali, 12-20% (as Na_2O); and liquor to wood ratio, *C. sativa*, 3.5:1 & *I. carnea*, 4:1]. At optimum cooking conditions, anthraquinone (AQ; 0.0-0.2%, oven dry weight basis) was added to observe its effect on pulp yield and kappa number (Table 2). After cooking, pulps were washed on a laboratory flat stationary screen having 300 mesh wire bottom for removal of residual cooking chemicals. Pulp was disintegrated and screened

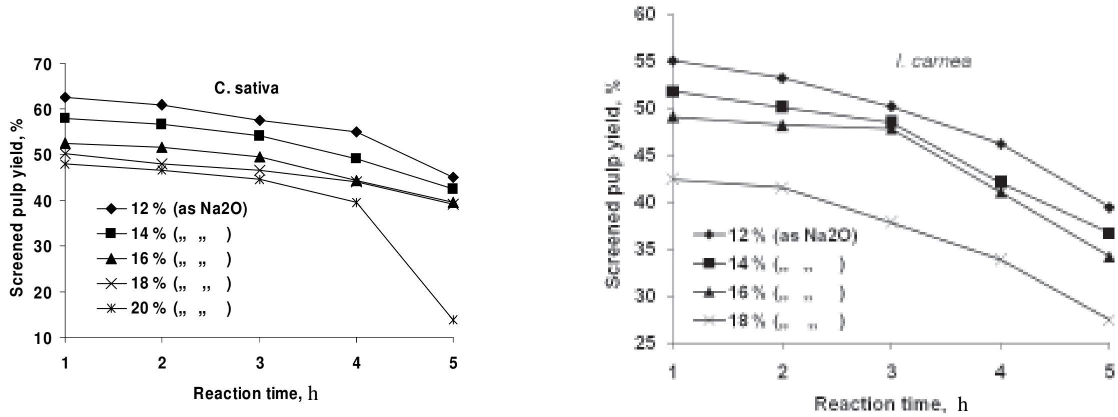

Fig. 2—Effect of active alkali on pulp yield during soda pulping of *C. sativa* and *I. carnea* at 16% active alkali (as Na_2O)

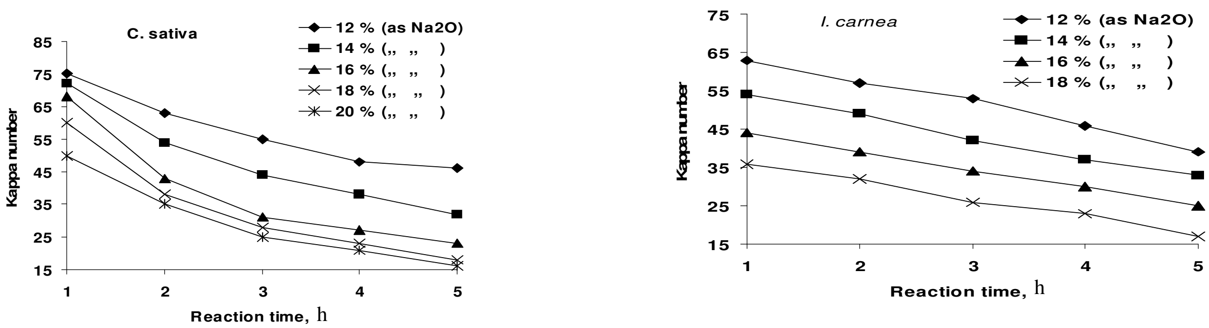

Fig. 3 — Effect of active alkali on kappa number during soda pulping of *C. sativa* and *I. carnea* at 16% active alkali (as Na_2O)

through a WEVERK vibratory flat screen (slot size, 0.15 mm) and screened pulp was washed, pressed and crumbled. Pulps were analyzed for kappa number (T 236 cm-85), pulp yield and lignin (T 222 om-88), screening rejects and residual alkali as per Tappi Standard Test Methods: 2000 (Figs 1-3).

Spent Liquor Analysis and Pulp Evaluation

Spent liquor characteristics of *C. sativa* and *I. carnea* and *C. sativa* generated at optimum cooking conditions were analyzed for black liquor solids (T650 sp-96), soda black liquor (T625 wd-99) and pH (T252 om-02) as per Tappi Standard Test Methods:2000; BOD_5 (D6238-98-2003), COD (D1252-06-1997), and calorific value (D407-44-1969) as per American Standard Test Methods; viscosity by Brookfield viscometer and solid in black liquor by Twaddle meter (Table 3). Unbleached pulp was disintegrated in PFI mill at different beating levels (Tappi T 200 sp-96). Laboratory hand sheets (60 g/m^2) were prepared (T 205 sp-95) and tested for various physical strength properties (T 220 sp-96 (Tables 4, 5).

Pulp Bleaching

Soda and soda-AQ pulps of *C. sativa* and *I. carnea* were bleached by CEHH bleaching (Table 6).

Results and Discussion

Morphological Characteristics

I. carnea fibers possess less values for cell wall thickness (75.08-75.50%), wall fraction (69.31%) and Runkel ratio (66.67-80.00%) compared to softwoods (*Pinus kesiya*, and *Picca abies*), indicating *I. carnea* fibers are readily converted into double walled ribbon structure and exhibit plastic deformation on pressing and thus, offering more surface contact area for fibre bonding (Table 1). This gives good physical strength and also rattleness. *C. sativa* fibers possess more values for cell wall thickness (8.33-10%), wall fraction (65.86%) and Runkel ratio (80.00-200.00%) compared to softwoods

Table 2 — Effect of AQ on pulp yield, kappa no. and screening rejects of *C. sativa* and *I. carnea*.

	Cannabis sativa				*Ipomea carnea*			
AQ dose %	Unscreened pulp yield %	Rejects %	Screened pulp yield %	Kappa No	Unscreened pulp yield %	Rejects %	Screened pulp yield %	Kappa No
0.0	50.50	1.00	49.50	33	48.53	1.05	47.86	34
0.05	50.39	0.47	49.92	28	48.48	0.95	43.53	28
0.10	50.72	0.22	50.50	28	48.41	0.31	48.10	26
0.20	49.43	0.16	49.37	26	46.00	0.23	45.77	25

Cooking conditions: Active alkali = 16% (as Na_2O), Maximum cooking time = 3 h at 165°C, Liquor to wood ratio = 3.5:1 for *C. sativa* and 4.0:1 for *I. carnea*, Digester pressure = 6.5 kg/cm^2

Table 3 — Soda and soda-AQ spent liquor characteristics of *C. sativa* and *I. carnea*

Particulars	*C. sativa*		*I. carnea*		Bamboo
	Soda	Soda-AQ	Soda	Soda-AQ	Kraft
Black liquor solid, %	24.45	24.10	25.10	24.80	18.80
Residual alkali, %	4.05	3.30	3.90	3.10	4.20
Inorganic as NaOH, %	28.35	27.55	28.26	27.65	35.00
Organic, %	71.65	72.45	71.7	72.35	65.00
BOD_5 day at 20°C, mg/l	29989.9	3112.5	24778	2481.77	-
COD, mg/l	72560	72650	76850	76925	-
Silica, %	3.45	3.22	2.21	1.71	-
Calorific value, Cal/g	2998.9	3112.5	2777.8	2882.1	2882.1
Brook field viscosity at 30°C cp Lv spindle no.	12.80	12.65	13.70	13.25	14.80
pH of liquor at 30 °C	10.05	10.30	10.12	10.05	10.4
°TW at 30°C	17.50	17.00	19.20	18.50	-

(*Pinus kesiya*, and *Picca abies*), indicating that force applied to fibres from sheet will be more and *C. sativa* fibre will give more tear. Short fibre length (0.6 mm) of *I. carnea* fibre is compensated by *C. sativa* fibre length (2.42 mm). Mechanical strength properties are increased by longer fibre and very sensitive to fibre length as well to degree of fibre bonding. Therefore, a proper blend of *I. carnea* and *C. sativa* may give strong paper.

Influence of Temperature and Time

Curves were plotted between residual lignin and reaction time at different reaction temperatures. Each curve can be approximated by two straight lines at each temperature (Fig. 1). Curves with steeper slopes are related to rapid solubilisation of bulk of lignin, whereas much gentle slopes to slow solubilisation of residual lignin and have different velocity constants. Bulk delignification corresponds to removal of lignin present in middle lamella[19] and residual delignification corresponds to removal of lignin in primary wall, secondary wall layers and interconnection cavities[20,21]. As temperature decreases from 175 to 145°C, both cooking time to reach transition from bulk to residual delignification and pulp and lignin content corresponding to transition point

Table 4 — Strength properties of unbleached soda pulps of *C. sativa* and *I. carnea*

AQ doses %	Beating time min	Beating level ⁰SR	Drainage time, s	Burst index kPam²/g	Tensile index Nm/g	Tear index mNm²/g	Double fold Nos
Cannabis sativa							
12	0	17	4	0.73	20.97	5.48	16
	8	25	12	2.84	41.52	5.99	32
	30	40	22	3.86	58.92	5.67	83
	33	45	25	3.95	62.03	5.23	92
14	0	15	3	0.93	20.87	4.93	12
	8	24	12	3.80	45.60	5.80	33
	31	42	23	4.62	57.74	5.49	92
	34	45	25	4.86	58.00	5.05	107
16	0	16	4	0.72	17.59	4.59	16
	10	27	15	3.41	48.58	6.51	75
	30	41	22	4.65	70.71	5.48	192
	33	45	25	5.05	73.74	4.81	212
18	0	18	4	0.96	20.70	5.07	18
	10	27	15	3.10	45.85	5.67	93
	29	41	21	4.07	61.98	5.32	200
	33	45	25	4.52	67.23	4.32	213
20	0	18	4	0.97	27.16	4.50	18
	22	31	16	3.85	58.08	5.32	95
	29	39	21	4.50	61.79	4.70	201
	33	45	25	4.07	66.19	4.30	210
Ipomea carnea							
12	0	12	3	0.64	9.36	3.06	2
	10	27	12	1.30	30.76	4.25	30
	30	41	17	1.75	43.12	3.54	40
	34	45	22	1.70	61.19	3.25	41
14	0	9	3	0.60	12.84	3.80	5
	10	26	12	1.93	47.13	4.35	28
	30	39	15	2.51	54.11	4.10	46
	34	45	22	2.60	55.00	3.87	48
16	0	18	3	0.64	8.30	2.86	6
	9	22	10	2.60	50.31	5.36	39
	30	38	14	3.36	66.00	4.10	88
	34	45	22	3.61	68.00	4.05	93
18	0	19	3	0.45	8.46	3.44	4
	10	24	11	2.45	51.36	5.16	43
	31	41	17	3.10	62.03	4.56	75
	34	45	22	3.25	63.00	4.25	85
Pinus kesiya							
0	–	45	–	5.6	62.26	8.8	–

increases. After transition point, curve is almost horizontal line indicating termination of bulk delignification phase. Curves plotted between residual lignin and reaction time at 175°C coincides curve plotted at 165°C. A temperature of 165°C at maximum cooking time (3 h) may be considered as an optimum cooking temperature and time for soda cooking of *C. sativa* and *I. carnea.*

Influence of Alkali Charge

Screened pulp yield (Fig. 2) increases with increasing active alkali from 12 to 16% (as Na_2O) and then declines sharply whereas both kappa number (Fig. 3) and screening rejects decline sharply and beyond alkali level of 16% remains almost constant. Total screened pulp yields are: *I. carnea*, 47.86% (kappa number, 34); and

Table 5 — Strength properties of unbleached soda- AQ pulps of *C. sativa* and *I. carnea*

AQ doses %	Beating time min	Beating level ^{0}SR	Drainage time, s	Burst index kPam^2/g	Tensile index Nm/g	Tear index mNm/g	Double fold Nos
Cannabis sativa							
0.0	0	16	4	0.73	17.59	4.59	2
	10	27	15	3.41	48.58	6.51	75
	30	41	22	4.65	70.71	5.48	192
	33	45	25	6.05	73.74	4.81	212
0.05	0	19	5	2.25	34.21	7.29	18
	10	27	15	3.51	57.48	5.98	113
	30	41	22	4.78	71.65	4.26	195
	33	45	25	5.22	74.18	4.09	215
0.1	0	20	6	2.75	35.67	5.25	18
	10	32	18	4.50	67.21	6.16	125
	30	40	20	5.65	72.45	5.32	220
	33	45	25	5.70	75.55	4.78	223
0.2	0	29	6	2.73	40.41	5.98	3
	10	32	18	4.15	56.13	6.22	130
	30	41	20	5.19	60.51	5.32	200
	33	45	25	5.48	71.43	5.05	213
Ipomea carnea							
0.0	0	8	3	0.64	8.30	2.26	6
	9	22	10	2.60	50.31	5.36	39
	30	38	14	3.36	66.00	4.10	88
	32	45	22	3.61	68.00	4.05	93
0.05	0	13	4	0.75	23.75	2.40	15
	8	22	10	2.62	47.07	4.38	43
	30	38	14	3.52	67.46	3.55	110
	34	45	22	3.61	69.98	3.26	115
0.1	0	13	4	0.75	24.38	2.62	15
	15	34	10	3.50	64.18	4.26	93
	31	42	14	3.71	71.54	3.78	125
	33	45	22	3.81	70.21	3.50	128
0.2	0	16	5	0.95	23.35	2.72	15
	13	30	16	3.25	61.61	4.18	82
	32	40	19	3.74	71.92	3.58	125
	34	45	22	3.76	70.98	3.50	124

C. sativa, 49.50% (kappa number, 33). An active alkali charge of 16% (as Na_2O) may be considered as optimum cooking dose for soda cooking of *C. sativa* and *I. carnea*.

Influence of Anthraquinone (AQ)

On increasing AQ dose, there is a gradual increment in pulp yield and continuous reduction in both kappa number and screening rejects (Table 2) but increment in pulp yield and mitigation in kappa number and screening rejects are not so much pronounced beyond an AQ dose of 0.1%. Addition of 0.1% AQ dose improves pulp yield by 0.24% and reduces kappa number by 8 units for *I. carnea* and improves pulp yield by 1.0% and decreases kappa number by 4 units for *C. sativa*.

Black Liquor Analysis

Characteristics of soda and soda-AQ spent liquor of *C. sativa* and *I. carnea* under optimum pulping conditions are compared with kraft spent liquor of bamboo (Table 3). Calorific values of spent liquors are slightly on higher side than that of bamboo due to high solid contents. Silica contents in spent liquors are very low compared to bamboo. High calorific value with very low silica content

Table 6 — Bleaching conditions and results of soda and soda AQ pulps at *C. sativa* and *I. carnea*

Particulars	*C. sativa*		*I. carnea*	
	Soda	Soda-AQ	Soda	Soda-AQ
Unbleached pulp kappa no.	33.00	28.00	34.00	26.00
Chlorination stage, (C)				
Amount of chlorine added on pulp, %	5.80	4.90	5.80	4.23
Amount of chlorine consumed on pulp, %	5.72	4.85	5.76	4.18
Amount of chlorine consumed on Cl_2 basis, %	98.62	98.97	99.31	98.80
Final pH	1.94	1.96	1.96	2.04
Alkali extraction stage (E)				
NaOH added on pulp, %	2.90	2.50	2.90	2.10
Initial pH	11.64	11.58	11.54	11.58
Final pH	10.30	10.28	10.20	10.37
Hypochlorite 1st stage (H_1)				
Hypo added as avail able Cl_2 on pulp, %	1.70	1.40	1.70	1.30
Hypo consumed as available Cl_2 basis, %	1.65	1.38	1.67	1.25
Hypo consumed on Cl_2 basis, %	97.00	98.57	98.23	96.15
Final pH	8.11	8.00	7.79	7.88
Hypochlorite 2nd stage (H_2)				
Hypo added as available Cl_2 on pulp, %	0.85	0.70	0.85	0.70
Hypo consumed as available Cl_2 on pulp %	0.80	0.68	0.83	0.68
Hypo consumed on Cl_2 basis, %	94.00	97.00	94.00	97.64
Final pH	9.17	9.50	9.60	9.45
Total Cl_2 added on pulp, %	8.25	7.00	8.50	6.50
Total Cl_2 consumed on pulp, %	8.17	6.91	8.42	6.16
Bleaching looses, %	9.00	9.00	10.00	10.40
Bleached pulp yield, %	45.0	46.0	43.06	43.30
Pulp brightness	84.0	83.5	87.1	86.4
Bleaching conditions	C	E	H_1	H_2
Consistency, %	3	10	9	9
Temp, ^{0}C	27±2	55±2	45±2	45±2
Time, min.	25	120	60	120

is an advantageous factor towards energy conservation in chemical recovery system.

Mechanical Strength Properties

Mechanical strength properties (tear, tensile and burst indexes and double fold) of *C. sativa* and *I. carnea* increase with increase in active alkali up to 16% (as Na_2O) at each respective freeness level and then all mechanical properties decline due to carbohydrate degradation. Optimum mechanical strength properties are achieved at 45°SR for *I. carnea* and 44°SR for *C. sativa* (Table 4). Beating time for *C. sativa* is slightly more (3 min) than *I. carnea* to get optimum freeness level. Soda-AQ pulp shows an improvement over soda pulp due to preservation of carbohydrates (Table 5).

Pulp Bleaching

CEHH bleaching (Table 6) of *C. sativa* produces pulp [yield, 44.90%; brightness, 84% (Elrepho); total chlorine demand (TCD), 5.80%]. Use of AQ reduces total chlorine demand by 0.90% with slight gain in pulp yield and loss in brightness. *I. carnea* soda pulp produces a pulp [brightness, 87.1% (Elrepho); TCD 5.80%]. However, soda-AQ pulp of *I. carnea* shows slight drop in brightness. It means that addition of *I. carnea* pulp in a blend improves overall pulp brightness.

Conclusions

A proper blend of *I. carnea* and *C. sativa* can produce stronger paper comparable to softwoods (*Pinus kesiya*, and *Picca abies*) except liquor to wood ratio (*C. sativa*, 3.5:1; and *I. carnea*, 4:1). High calorific value of spent liquor of *I. carnea* and *C. sativa* with very low silica content is advantageous factor towards energy conservation in chemical recovery system. CEHH bleaching of *C. sativa* produces pulp [brightness, 84% (Elrepho); TCD, 5.80%], whereas *I. carnea* soda pulp produces pulp [brightness, 87.1% (Elrepho); TCD, 5.80%]. Therefore, *I. carnea* pulp may be used as brightness improver. Optimum mechanical strength properties are achieved at 45°SR for *I. carnea* and 44°SR for *C. sativa*.

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