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Eco-Friendly Oxygen Bleaching of *Anthocephalus indicus* Pulp for the Reduction of Chemical Consumption and Pollutant Generation

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Kraft pulps of *Anthocephalus indicus* of kappa number 39.49 and 24.76 were bleached adopting O/C/E/H/H, O/C/E/H/P and conventional C/E/H/H bleaching sequences. It was observed that bleached pulps possessed comparable strength properties; burst index,5.33 +0.27; tensile index,87.30 $\pm$ 1.55; tear index,4.95 $\pm$ 0.21 however, bleached pulp yield was about 7-8 % higher for kappa number 24.76. Intrinsic viscosity/degree of polymerization of pulps also holds the same trend. COD, BOD_5 and AOX values were 104.95,37.56 and 7.54 kg/tone of pulp (kg/tp) for O/C/E/H/H and minimum 45.25,14.79 2.56 kg/tp for O/C/E/H/P bleaching effluents of pulp of kappa number 36.49 COD, BOD_5 and AOX values were 74.56, 27.46 and 5.20 kg/tone for the C/E/H/H bleached pulp; 38.11,14.36 and 2.80 Kg/tp for the O/C/E/H/H 32.23,12.57 and 2.40 kg/tp bleaching effluents of pulp of kappa number 24.76. Thus, COD, BOD and AOX values were highest for C/E/H/H followed by O/C/E/H/H and minimum for O/C/E/H/P and appreciably decreased on oxygen treatment.

Keywords: Kappa number, lignin, viscosity, bleaching, degree of polymerization

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Bleaching is a chemical process applied to unbleached pulps to improve their brightness either by dissolving out the chromophoric/chromogenic groups containing residual lignin from pulps (lignin removing bleaching) or by modifying the (lignin retaining bleaching). In India, chlorination/alkali extraction/hypochlorite/ hypochlorite (C/E/H/H) bleaching sequence is generally adopted for the bleaching of chemical pulps. However, the chlorination stage is the primary source of pollutants in the resulting effluent, particularly chlorinated organic compounds that pose serious environmental concerns [1,2]. Oxygen is one of the most abundant elements in earth's environment which acts as relatively inexpensive bleaching agent for cellulose pulps [3,4]. Use of alkaline oxygen as bleaching agent starts in the 1950s but ineffective due to its low selectivity towards lignin and other chromophoric groups and carbohydrate degradability and poor strength properties. These short coming were circumvented in the 1960s by use of magnesium salts and silicates as carbohydrate protective agent. At present the oxygen bleaching is normally developed as a step preceding a conventional bleaching [5-7] but high capital is still a issue because of low solubility of oxygen in process liquors requires the use of high pressure [8,9] Therefore, an attempt has been made to substitute the elemental chlorine, partially, to bleach the *Anthocephalus indicus* kraft pulps prepared under optimized conditions by oxygen treatment. Effluents generated were characterized and compared with effluent generated by C/E/H/H bleaching sequence. The main advantage of use of Oxygen as bleaching agent is the effluent contain low chlorides or organo-chlorides which facilitates their recycling and recovery [10,11]. The results of this investigation are presented and discussed in this paper.

EXPERIMENTAL

Bleaching of Pulps

Anthocephalus indicus kraft pulps of kappa number 36.49 and 24.76 were bleached adopting O/C/E/H/H, O/C/E/H/P and conventional C/E/H/H bleaching sequences. For all pulps, 2.00 and 1.00 percent alkali was charged, and oxygen treatment was performed at optimal circumstances with an oxygen pressure of 8.0 kg/cm and a treatment temperature of 100 °C. The pulps had kappa numbers of 36.49 and 24.76, respectively, and 0.25 percent MgSO4 at 12% consistency. For thirty minutes, chlorination (C) was conducted at room temperature with a 3% regularity. For 60 minutes, alkali extraction (E) was performed at 68.0 $\pm$ 2.0^{0}C with 8% regularity. The hypochlorite stage of the hypochorite (H) therapy, which used calcium hypochlorite, lasted 90 minutes at 42.0 $\pm$ 2.0^{0}C and 8% regularity. A pH of 9.5 $\pm$ 0.5 was maintained during this time. Hydrogen peroxide (P)

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treatment was carried out at 65.0 ± 2.0 at 8 % regularity for 60 minutes. In the P stage, chelating agents such as magnesium sulfate and sodium salt of ethylenediamine tetra acetic acid (EDTA) were used, along with sodium silicate as a buffer. Other conditions are recorded in table 1. Bleached pulp yield and brightness of each pulp was determined.

Beating, Sheet Making and Physical Strength Characteristics of Bleached Pulps

In the PFU mill, bleached pulps were pounded at various revolutions. Using a conventional British sheet forming machine, hand sheets weighing 65.0 ± 2.0 gsm for every pulp were generated. The sheets were then dried in the air, conditioned at 27.0 + 1.0 ^{0}C and 65.0 ± 2.0 % relative humidity, and evaluated for a variety of strength qualities. Interpolation of the results was done at freeness 250 (ml) csf.

Intrinsic Viscosity of Pulps

The viscosity of pulps was determined by scan method, SCAN 15:62 as relative viscosity in a capillary tube viscometer and results were converted to intrinsic viscosity by applying Martin's formula [12]. The average degree of polymerization (DP) was calculated from the valve of intrinsic viscosity by using the formula proposed by Immsrgut, et, al. 1956 [13].

Effluent Collection, Storage and COD, BOD and AOX

Effluents from C/E/H/H, O/C/E/H/H and O/C/E/H/P bleaching were collected, combined, let to dry out any remaining chlorine about two hours in the sun, and then stored at 4^{0}C. the effluent from oxygen stage was discarded, COD, BOD and AOX were determined using standard procedures.

RESULTS AND DISCUSSION

Impact of Bleaching Order on Pulp Brightness, Strength, and Yield

Anthocephalus indicus kraft pulps of kappa number 36.49 and 24.76 were bleached adopting C/E/H/H, O/C/E/H/H and O/C/E/H/P bleaching sequences. Bleached pulp yield 52.64,52.23 and 52.97 % (52.60 ± 0.37 %) for pulp of kappa number 36.49 and 45.66, 45.34, 45.44 % (45.50 ±0.16 %) for the pulp of kappa number 24.76 was comparable in the respective case for C/E/H/H, O/C/E/H/H and O/C/E/H/P bleaching (Table 2). However, pulp with higher kappa number (36.49) exhibited approximately 7–8% higher yield than that with lower kappa number (24.76), which may be attributed to reduced carbohydrate degradation during bleaching [2-3]. Kraft pulp of kappa number 36.49, exhibited burst index,5.22, 5.56 and 5.60; tensile index, 85.95,87.63 and 88.85 and tear index, 5.16,4.84 and 5.06 while pulp of kappa number 24.76 exhibited burst index,5.06, 5.08 and 5.40; tensile index, 85.75,87.04 and 88.24 and tear index, 4.92, 4.74 and 4.95 adopting C/E/H/H, O/C/E/H/H and O/C/E/H/P bleaching sequences, respectively (Table 2). These result indicated that the pulp of kappa number 36.49 and 24.76 yielded bleached pulps possessing almost comparable strength properties, burst index,5.33 ±0.27; tensile index, 87.30 ±1.55 and tear index, 4.95 ±0.21.

Table 1. Bleaching conditions and pulp properties for different bleaching sequences.

S. No	Particulars	Bleaching Sequence					
		C/E/H/H		O/C/E/H/H		O/C/E/H/P	
1	Kappa Number unbleached pulp	36.49	24.76	36.49	24.76	36.49	24.76
2	Unbleached pulp yield* %	58.98	49.49	58.98	49.49	58.98	49.49
3	Kappa Number After O Stage	-	-	17.56	13.12	17.56	13.12
4	Chlorine, * % (C)	6.00	4.00	2.90	2.15	2.90	2.15
5	NaOH, * % (E)	1.50	1.00	075	0.50	0.75	0.50
6	Hypochlorite, * % (H)	2.00	1.40	1.00	075	1.00	0.75
7	Hypochlorite, * % (H)	1.25	0.80	0.50	4.00	-	-
8	H_2O_2, % Sodium Silicate % $MgSO_4$, % EDTA, %	-	-	-	-	0.75 1.50 0.25 0.10	0.50 1.25 0.25 0.10
9	Pulp Yield ,* %	52.64	45.66	52.23	45.34	52.97	45.44

Notes: Values are expressed on oven-dry pulp basis (*). "–" indicates not applicable. C = Chlorination, E = Alkali extraction, H = Hypochlorite, O = Oxygen, P = Peroxide.

Table 2. Bleached pulp yield, strength properties at freeness 250 (ml) csf and brightness of *Anthocephalus indicus* kraft pulps.

S. No	Bleaching Sequence	Kappa Number	Pulp Yield %*	Brightness % ISO	Burst Index, kpam^2/g	Tensile Index, Nm/g	Tear Index mNm/g
1	C/E/H/H	36.49	52.64	74.36	5.22	85.94	5.16
	O/C/E/H/H	17.56**	52.23	78.55	5.56	87.63	4.84
	O/C/E/H/P	17.56**	52.97	81.36	5.80	88.85	5.06
2	C/E/H/H	24.76	45.66	76.63	5.06	85.75	4.92
	O/C/E/H/H	13.12**	45.34	78.92	5.26	87.04	4.74
	O/C/E/H/P	13.12**	45.44	82.75	5.40	88.24	4.95

*Percentage based on o.d raw material. ** Kappa number after oxygen treatment.

Table 3. Intrinsic viscosity/degree of polymerization of *Anthocephalus indicus* kraft pulps.

S. No	Particulars	Bleaching sequence					
		CEHH	OCEHH	OCEHP	CEHH	OCEHH	OCEHP
1	Kappa number	36.49	17.56*	17.56*	24.76	13.12*	13.12*
2	Intrinsic viscosity cm^3/g	380	425	440	350	402	412
3	degree of polymerization	570	637	660	525	603	623

*kappa number of pulps after oxygen treatment

Brightness of C/E/H/H bleached pulps was 74.36, and 76.63 %, ISO for pulp of kappa number 36.49 and 24.76. It was improved to 78.55 and 78.92 %, ISO for O/C/E/H/H bleaching and 81.36 and 82.75 %, ISO for O/C/E/H/P bleaching (Table-2). The highest brightness was achieved in O/C/E/H/P followed by O/C/E/H/H while the conventional C/E/H/H sequence showed the lowest brightness. This improvement can be attributed to enhanced lignin removal and reduced formation of chromophoric groups in oxygen- and peroxide-based systems [11,13,14]

Effect of Bleaching Sequence on Intrinsic Viscosity/ Degree of Polymerization of Pulps

Values for intrinsic viscosity/degree of polymerization were 380/570, 425/637 and 440/660 respectively for the pulp of kappa number 36.49 and 350/525, 402/603 and 415/623 respectively for the pulp of kappa number 24.76 adopting C/E/H/H, O/C/E/H/H and O/C/E/H/P bleaching sequences. (Table -3) The pattern of strength properties is also in agreement with the values for intrinsic viscosity/degree of polymerization of different pulps.

The chlorination followed by alkali extraction of chlorinated compounds [14, 15]. These contaminants increase the need for biological and chemical oxygenides (BOD and COD, respectively), absorbable organic halides (AOX), toxic chlorinated compounds including genotoxic compounds and colour of the effluent. Therefore, one strategy would be to partially replace elemental chlorine with oxygen, then bleach with chlorine dioxide (D) and hydrogen peroxide (P), referred as Elemental chlorine free (ECF) bleaching. It is also possible to bleach the pulp by replacing elemental chlorine and chlorinated compounds completely with other environmentally favourable bleaching agents viz.; oxygen, ozone, hydrogen peroxide and even pretreatment with biological bleaching agents such as enzymes, referred as Total chlorine free (TCF) bleaching. Both the modifications in bleaching improves the effluent quality and reduces the effluent load considerably, as the recovery furnace could recycle the effluent after the oxygen and ozone stages. But the techniques are costly and require strict control on operational parameters, particularly in the ozone treatment stage to maintain the quality and quantity of pulp. However, these ECF and TCF bleaching techniques are in practice in developed countries.

Table 4. COD, BOD_5 and AOX of effluents on bleaching *Anthocephalus indicus* kraft pulps.

S. No	**Particular**	**Bleaching Sequence**					
		CEHH	OCEHH	OCEHP	CEHH	OCEHH	OCEHP
1	Kappa number	36.49	17.56*	17.56*	24.76	13.12*	13.12*
2	COD Kg/tp	104.95	52.04	45.25	74.56	38.11	32.23
3	BOD_5 Kg/tp	37.56	18.10	14.79	27.46	14.36	12.57
4	AOX Kg/tp	7.54	3.28	2.56	5.20	2.80	2.40

*kappa number of pulps after oxygen treatment

In a developing country like India, there are many difficulties to adopted ECF/TCF bleaching due to high capital cost, necessity of strict control on the operational parameters and energy crises. Thus, C/E/H/H bleaching is still being practiced by most of the mills. Probably complete substitution of elemental chlorine would not be acceptable or possible at the moment. However, partial substitution of elemental chlorine by oxygen could be an acceptable modification in the bleaching process hither to being practiced. partially delignifide (~ 50% of unbleached pulp) pulp by oxygen treatment prior to chlorination would reduce the amount of lignin entering in the bleaching section leading to lower chlorine demand for the bleaching of the pulps [16]. This would improve the quality and reduce the effluent load entering into recipient natural water resources. The process may further be improved, by replacing remaining use of chlorine by chlorine dioxide/ hydrogen peroxide or even by ozone/hydrogen peroxide in combination with enzymatic treatment of pulps in due course of time 4-7.

Effect of Bleaching Sequence on COD, BOD_5 and AOX of Effluents

Values for bleach effluents were COD 104.95, 52.04 and 45.25 kg/tp; BOD_5 37.56, 18.10 and 14.79 kg/tp and AOX 7.54, 3.28 and 2.56 kg/tp for the pulp of kappa number 39.49. These value for the pulp of kappa number 24.76 were COD 74.56, 38.11 and 32.23 kg/tp; BOD_5 27.46, 14.46 and 12.57 kg/tp and AOX 5.20, 2.80 and 2.40 kg/tp for C/E/H/H, O/C/E/H/H and O/C/E/H/P bleaching sequences, respectively (Table- 3). Reduced amounts of residual lignin in the pulp may be the cause of reduced COD, BOD_5, and AOX levels for the pulp with a low kappa number. Further, the considerably lower COD, BOD_5 and AOX values for oxygen treated pulps may due to the fact that the effluent generated in the oxygen treatment stage were discarded for the determination of these value as same may be recycled in chemical recovery section. Recycling of effluent in recovery section would give additional amount of alkali coupled with generation of addition energy on burning of the organic matte present in the effluent. This would be an additional advantage of oxygen treatment beside decrease in effluent load generated in subsequent bleaching stages.

The data indicated that the difference in COD, BOD_5 and AOX values for oxygen untreated and oxygen treated pulp was very wide for both the pulps of kappa number 39.49 and 24.76. Oxygen treatment resulted in a substantial reduction in the differences between these values. This may be attributed to the fact that the difference kappa number in untreated pulps was of the order of 14.77 and it was narrowed down to only 4.44 after the oxygen treatment consequently, difference in COD, BOD_5 and AOX values was also narrowed down, considerably.

CONCLUSIONS

Pulps with kappa numbers of 39.49 and 24.76 exhibited comparable strength properties when subjected to different bleaching sequences, including C/E/H/H, O/C/E/H/H, and O/C/E/H/P [17]. However, the bleached pulp yield was approximately 7% higher for the pulp with a kappa number of 39.49 compared to that with a kappa number of 24.76 [16, 18].

Oxygen pre-treatment significantly reduced critical effluent parameters such as chemical oxygen demand (COD), biochemical oxygen demand (BOD_5), and adsorbable organic halides (AOX), indicating improved environmental performance [16,18]. Among the bleaching sequences studied, the O/C/E/H/P sequence showed the greatest enhancement in pulp brightness and effluent quality, followed by O/C/E/H/H, in comparison to the conventional C/E/H/H sequence [18.19].

A substantial difference in COD, BOD_5, and AOX levels was observed between effluents from oxygen-treated and untreated pulps of both kappa numbers. However, these differences were considerably reduced when comparing oxygen-treated pulps of kappa numbers 39.49 and 24.76 [20, 21]. This suggests that the treatability of effluents generated from these two oxygen-treated pulps is nearly similar, particularly when the O/C/E/H/P bleaching sequence is employed.

Therefore, it can be concluded that pulps with higher kappa numbers may be preferred over lower kappa number pulps for oxygen bleaching due to their higher pulp yield advantage, provided that the effluent treatment plant capacity is not a limiting factor [17, 22].

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