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Application of Enzyme for Bleaching of Wheat Straw AQ-Soda Pulp

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ABSTRACT

Pulp and paper industry is one of the major contributors to environmental pollution due to its toxic and carcinogenic discharges during its various processes e.g., chlorinated bleaching of the pulp. Use of alternative bleaching chemicals e.g., enzyme and/or elemental chlorine free (ECF) or total chlorine free (TCF) agents are the best option to reduce the load of carcinogenic chlorinated compounds on our environment. Present study was carried out to highlight the application of enzyme during bleaching of wheat straw anthraquinon (AQ)-soda pulp to considerably reduce the toxic load on the environment. Wheat straw AQ-soda pulp was treated with acid (A) to reduce its metal contents followed by oxygen (O) delignification. Delignified pulp was then subjected to enzymatic (X) treatment with xylanase prior to bleaching to increase the effectiveness of successive bleaching with hydrogen peroxide (P). Handsheets were prepared from the treated pulp after each experimental trial to study its various optical, physical and chemical properties. Brightness of bleached pulp was improved up to ISO 73% with AOXP$_1$P$_2$ bleaching sequence. The improved brightness up to ISO 76% was achieved when alkali extraction stage was incorporated before bleaching with hydrogen peroxide (AOXEP$_1$P$_2$). TDS, COD and BOD$_5$ of effluent samples during different experimental stages was monitored and compared with NEQS. It was evident from the results that enzymatic treatment not only helped to reduce the effluent load but also reduced the consumption of bleaching chemicals to achieve good brightness values.

Key words: *delignification, brightness, xylanase, hydrogen peroxide*

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Introduction

Pulp and paper industry contributes greatly to environmental pollution because of its high demand for energy and consumption of chemicals. Resultantly, effluent loaded with high concentration of suspended solids and organics is released into the environment [1]. The large amounts of pollutants are released mainly during pulping and bleaching stages [2]. Chlorine, being a strong oxidizing agent and economically cheaper, has traditionally been preferred as a bleaching chemical, however, it has adverse effects on the environment [3]. Environmental concerns demanded the use of total chlorine free (TCF) or elemental chlorine free (ECF) bleaching processes [4]. TCF bleaching usually includes oxygen, oxone, hydrogen peroxide and ozone based stages [5-10], hence, avoids the formation of highly absorbable organic halogens (AOX) during TCF bleaching.

Bio-bleaching of pulp using xylanase enzyme is the most appropriate biological substitute of environmentally corrosive bleaching agents. Xylanase is used for the removal of lignin-carbohydrate complex which is formed during the pulping stage and hinders the entry of bleaching chemicals into the pulp structure [11]. A number of benefits associated with xylanase pre-treatment have been reported in the literature that includes increased brightness of pulp, reduced consumption of bleaching chemicals that ultimately lead to decreased amount of chlorinated compounds in the effluent from bleaching section [12-16].

Materials and Methods

Pulping

AQ-soda wheat straw pulp was prepared in a closed loop digester and pulping was carried out on the conditions mentioned in Table 1.

After cooking, digester was switched off and the steam was released from the digester. The pulp was transferred in a pulp washer (fitted with a # 40 mesh size seive) to wash the pulp thoroughly and then passed through the laboratory defibrator (Weverk, Sweden) for 30 seconds at 3000rpm where water was added to disintegrate the pulp and then passed through 0.15mm slot screen to separate the shives from cooked wheat straw pulp. The screened pulp was then dewatered and stored in plastic bags at 4^{o}C until further experimentation.

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Development of Optimal Bleaching Sequence

Acidic Treatment of Pulp (A- Stage)

Acidification of AQ-soda pulp was carried out by using 0.5 N H_2SO_4 to remove its metal contents and hence to increase the effectiveness of bleaching. The effect of various reaction conditions i.e., acid dose, acidification time, temperature and pulp consistency were investigated to find the optimal reaction conditions for acidification (Fig. 1).

Oxygen Delignification (O-Stage)

Oxygen delignification of acidified pulp was carried out in a closed loop digester by taking 500g o.d. pulp at 10% pulp consistency. Pulp on o.d. basis was treated with 2% alkali solution and 0.5% of magnesium sulfate and the temperature was raised to 100^{o}C. Then oxygen was fed at 5 bar pressure and the reaction was continued for 60 minutes. After 60 minutes of treatment, digester was switched off, steam was released from the digester and distilled water was added to stop further reaction (Fig 3).

Enzymatic Treatment (X-Stage)

Xylanase enzyme i.e. Pulpzyme HC (Novozymes, Denmark) at 1% concentration was selected for enzymatic treatment of pulp prior to bleaching. Various xylanase doses, temperature and time for enzymatic stage were investigated to develop optimal reaction conditions at 4% consistency (Fig. 4).

Hydrogen Peroxide Bleaching (P-Stage)

After enzymatic treatment the pulp was exposed to hydrogen peroxide bleaching stage and peroxide, alkali and EDTA doses, temperature, time and pulp consistency were investigated to develop an optimal bleaching reaction (Fig. 5).

Each set of above experimental trials was followed by washing of pulp to neutral pH with distilled water on 200 mesh screen to remove the soluble fractions of pulp after every stage. The optimized pulp sample was dewatered and then stored at 4^{o}C in plastic bag until further experimentation.

Testing of Pulp and Paper Characteristics:

Hand sheets were made from the treated pulp samples after each experimental trial using the TAPPI test method, T 205 sp-95, 1995 [16] to test various optical and physical properties while the treated pulp samples were tested for various chemical properties.

Brightness, consistency, yield, tensile strength, burst strength and tear strength was was measured according to the TAPPI test methods [18-23]

The kappa number, acid insoluble lignin content, alpha-cellulose content and acid insoluble iron content of treated pulp samples were determined according to TAPPI test methods, T 236 om-99, 1999, T 222 om-98, 1998, T 203 cm-99, 1999 and T 434 cm-83, 1983 TAPPI test methods, respectively [24-27].

Analysis of Effluents

Effluent samples were collected during various pulping and bleaching processes. Grab sampling procedure was adopted to study the effect of individual process load on environment in terms of TDS, COD and BOD_5.

Results and Discussion

Acidification (A-Stage)

The removal of iron from pulp is important to effectively increase the brightness of pulp. The brightness of unbleached wheat straw AQ-soda pulp was measured to be 38.8% ISO. However, when pulp at 10% consistency was treated with acid at room temperature for 30 minutes, a slight increase in brightness was observed at variable acid doses (Fig. 1). As the minor difference exists in brightness values at different doses, the lowest dose of acid (0.5%) with 38.9% ISO brightness was chosen for further treatment. Acidic treatment of pulp under same conditions but with 0.5% acid dose for varying time intervals resulted in the lowest brightness value at 10 minutes i.e. 39.6% ISO while the highest value was found at 45 minutes i.e. 40.6% ISO. When pulp was treated with acid under the same conditions for 10 minutes at variable temperatures, the highest value 44.3% ISO was measured at 45°C and the lowest value 43% ISO was found at room temperature. At 35°C and 65°C, same brightness value was recorded i.e. 43.7% ISO and 55°C treatment temperature resulted in 43.3% ISO brightness that reflected that treatment at different temperatures have no major effect on brightness of pulp.

When pulp at variable consistency was treated under the other optimized conditions, it was evident that brightness gain was 44.1% ISO at 5% pulp consistency which did not deviate while increasing the pulp consistency up to 10%. Similarly, 43.7% ISO brightness value was observed at both 12% and 15% pulp consistency (Fig. 1).

The effect of acidification at optimized conditions on iron content (ppm) was evaluated by measuring acid insoluble iron (ppm) which was found to decrease from 266ppm for unbleached pulp to 204ppm for acidified pulp (Fig. 2). Acid-insoluble iron is considered that portion of iron present which is potentially chemically reactive, and its presence in the pulp may interferes with successive bleaching chemicals [28]. These results indicate that acidic treatment of pulp was helpful to decrease the iron content from unbleached pulp and to increase the brightness.

Oxygen Delignification (O-Stage)

Brightness is a commonly referred industry term for the numerical value of the reflectance factor of a handsheet sample with respect to the blue light of specific spectral and geometric characteristics [17]. And as brightness increase is related to removal of residual lignin level so to decrease the maximum lignin content from pulp, oxygen delignification stage was incorporated in bleaching sequence. Oxygen delignification resulted in increased brightness of hand sheet (52.3% ISO) and reduced kappa number (3.4) of pulp. The comparison between Ub (Unbleached pulp), A and AO stages shows the difference in brightness values (Fig. 3). The results of AQ-soda pulp samples treated at three different consecutive stages Ub, A and AO were statistically evaluated and correlation co-efficient (R^2) was found to be 0.9992. This high value of R^2 confirms that a very strong correlation exists between kappa number and brightness of the pulp. The results were further evaluated by measuring acid insoluble lignin which was found to decrease from A-stage to O-stage from 2.7% to 1.66% respectively. It shows that with the increase in brightness, kappa number decreases which indicates that oxygen treatment is effective to reduce lignin content. These results are also supported by the work of Hedjazi *et al* [29] who found that oxygen treatment is the most suitable and effective approach to initiate the bleaching process for reducing the cost of bleaching chemicals in the subsequent stages by reducing lignin content.

Enzymatic Treatment (X-Stage)

When oxygen delignified pulp at 4% consistency was treated with variable enzyme doses for 45 minutes at 55^{o}C, maximum brightness was achieved at 200g/t i.e. 53.8% ISO and the lowest value (52.7% ISO) was noted at 50g/t of enzymatic dose. Similar results were found at 100g/t and 200g/t of enzyme dose i.e. 53.3% ISO brightness (Fig. 4). During optimization of treatment temperature, pulp was treated with 100g/t of enzyme dose at variable temperature while the other conditions were same. The highest value of brightness 52.9% ISO was obtained at 45°C and the lowest 52.7% ISO brightness at 35°C (Fig. 4). Effect of time optimization trials shows that treatment with enzyme under the optimized conditions for 75 minutes resulted in 53.1% ISO brightness while 105 minutes decreased the brightness to 52% ISO. From the results at optimized conditions, it was found that application of enzyme to the oxygen delignified pulp prior to peroxide bleaching resulted in further increase in brightness of up to 52.9% ISO and drop in kappa number to 3.2 which indicated the maximum removal of lignin from the pulp. This fact has been theorized by Reddy *et al* [30] that pulpzyme is an enzyme that breaks the link between carbohydrates and lignin and depolymerizes the hemicelluloses. The separated lignin and depolymerized hemicelluloses are eliminated during the process of washing and ultimately results in higher brightness of the pulp.

Peroxide Bleaching (P-Stage)

P-stage was carried out using 3% hydrogen peroxide dose on o.d. pulp basis in the presence of 3% EDTA dose at 90°C for 180 minutes with varying alkali charges. Increasing trend in brightness was observed with the increase in alkali doses (Fig. 5). The lowest brightness value (60.7% ISO) was obtained at 0.5% alkali dose that was found to be increased (67.1% ISO) at 1.5% alkali dose. At 2.5% alkali dose, the brightness was recorded at 67.4% ISO. The highest brightness (67.5% ISO) was obtained at 3.5% alkali dose. It is noteworthy that at 2.5% and 3.5% alkali doses, slight difference in brightness value exists. Therefore, lowest dose (2.5%) was selected for further experimentation. In optimizing the pulp consistency, the pulp was bleached by using 2.5% alkali dose while all the other experimental conditions remained the same with varying pulp consistency. The highest brightness value 67.8% ISO was observed at 10% pulp consistency that was selected for further treatment (Fig. 5).

Results of peroxide dose optimization trails (Fig. 5) showed that with increasing the doses of peroxide, brightness increases. Maximum brightness was achieved at 10% dose i.e. 74.3% ISO and the lowest was achieved at 3% dose i.e. 67% ISO. 7% peroxide dose resulted in 71.5% ISO brightness while 5% dose resulted in 69.3% ISO brightness. As there is a not a significant difference by increasing the dose from 5% to 7%, therefore, 5 % hydrogen peroxide charge was selected for further experiments. Results of experimental trials for the optimization of treatment time at 5% peroxide dose revealed (Fig. 5) that with increase in reaction time, brightness value increased. It is obvious from these results that very little difference exists in brightness value at variable treatment time. So keeping in view the fact that longer treatment time at mill requires consumption of energy and increase in surrounding temperature, treatment for 120 minutes was selected to be used further for bleaching that resulted in 71.9% ISO brightness being more effective in terms of cost and time.

In optimizing the treatment temperature for effective bleaching, the pulp was bleached at variable temperature for 120 minutes. It is interesting to note that (Fig. 5) brightness was found to be increased with increasing temperature. But keeping in view the bleaching section of pulp and paper industry where the temperature is already very high, the best option for further experiments was observed to be 60^{o}C that showed 69.2% ISO brightness. Reaction conditions for the purpose of bleaching also involved optimization of EDTA dose. The highest brightness i.e. 70.6% ISO was achieved at 5% dose and the lowest value was found at 1% dose i.e. 68.8% ISO. At 2%, 3% and 4% doses, brightness values were found to be 70.2%, 70.5% and 70.4% ISO respectively. As not a significant increase was observed by increasing the EDTA dose from 2% to 5%. Therefore, 2% EDTA was considered as an optimum dose for further experimentation (Fig. 5).

To further increase the brightness, a second peroxide stage was incorporated and peroxide doses were also optimized by varying its doses i.e. 3%, 5% and 7% on o.d. pulp basis. Almost, the similar trend was observed in context of the increase in brightness. However the highest value 74.6% ISO was found at 7% dose and the lowest value 73% ISO was observed at 3% dose while at 5% peroxide dose, brightness was found to be 74% ISO. As the slight different exists among the brightness value so least peroxide dose 3% was found to be suitable for further experimentation. The purpose of various bleaching trials was to optimize the reaction conditions to develop an accurate and reproducible bleaching sequence of industrial significance. The resultant optimal conditions for $AOXP_1P_2$ bleaching trail are presented in table 2.

The hand sheets made from above mentioned treated pulp samples were also studied for physical properties and variable results were found. Fig 6 shows that the highest tensile index 43.38 Nm/g was found at AOP_1P_2 sequence whereas the lowest tensile index 37.14 Nm/g at $AOXEP_1P_2$ bleaching sequence. Whereas the maximum value of tear index was achieved at $AOXEP_1P_2$ sequence i.e. 5.32 mNm^2/g and the lowest value 4.57 mNm^2/g at AOP_1P_2 bleaching sequence. Similarly, variable results were found for burst index. The highest value 2.08 $Kpam^2/g$ was observed at AOP_1P_2 sequence whereas the lowest value at $AOXEP_1P_2$ sequence i.e. 1.58 $Kpam^2/g$. The highest yield of pulp 92.4 % was achieved at $AOXEP_1P_2$ bleaching sequence whereas the lowest 85.0 % at AOP_1P_2. This is indicative of the fact that incorporating an alkaline extraction stage is beneficial and helped to improve brightness, tear index and yield of the final bleached pulp.

The effectiveness of hydrogen peroxide is limited by the fact that the radicals generated during the peroxide decomposition are responsible for the degradation of cellulose. Alpha cellulose was found to be preserved in optimized peroxide treated pulp [$AOXEP_1P_2$] which demonstrates that the application of enzyme and use of chelating agent (EDTA) during peroxide bleaching found to be helpful to prevent the degradation of cellulose fibers (Fig 7).

Pulp at optimized bleaching stages was also tested for acid insoluble lignin. It was found that acid insoluble lignin reduced from 2.7 % of unbleached pulp to 0.98 % of optimized peroxide bleached pulp that confirms that application of enzyme was also effective to remove the lignin contents and ultimately help to boost brightness values.

Study of effluent load

The present study advocates the application of enzyme along with eco-friendly chemicals like oxygen and hydrogen peroxide to bleach the pulp as an effective alternative approach to hypochlorite bleaching. Incorporation of enzymatic stage increases the brightness as well as reduces the consumption of other chemical during the successive bleaching stages with increased pulp yield. The use of enzyme and total chlorine free chemicals for bleaching helped to reduce the overall TDS and BOD load on to the effluent treatment plant and completely avoided toxic chlorinated discharges. Figure 8 shows the effluent load during each individual stage of $AOXP_1P_2$ bleaching sequence and its comparison with NEQS for industrial effluents [31].

Conclusion

The present study presents the application of enzyme and the use of chlorine free chemicals i.e., oxygen and hydrogen peroxide to delignify and bleach the pulp. An eco-friendly bleaching sequence [$AOXEP_1P_2$] was developed for AQ-soda wheat straw pulp.

The developed sequence helped to increase the brightness, reduce the lignin content of the bleached pulp, limit the consumption of bleaching chemicals to achieve the desired brightness and hence, reduces the overall effluent load on the treatment plant and ultimately on the environment.

Acknowledgment

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Table 1 AQ-soda wheat straw pulping conditions

Wheat Straw	1000.0 g
Alkali Charge	15 % on o.d pulp weight
AQ Charge	0.5% on o.d pulp weight
Unidol AT	0.1% on o.d pulp weight
Raw material to liquor Ratio	1:5
Water to steam pressure	7: 7.5 bar
Reaction time	180 min
Reaction temperature	165°C

Table 2 Optimal reaction conditions of various bleaching stages in $AOXP_1P_2$ bleaching trail

Reaction Conditions	**Bleaching Stages**				
	A	**O**	**X**	**P_1**	**P_2**
Temp (°C)	35	100	45	60	60
Time (min)	10	60	45	120	120
Pulp Consistency (% on o.d.p)	10	10	4	10	10
H_2SO_4 (% on o.d.p)	0.5	--	--	--	--
O_2 Pressure (bar)	--	5	--	--	--
Enzyme (g/t)	--		100		
H_2O_2 (% on o.d.p)	--		--	5	3
NaOH (% on o.d.p)	--	2	--	2.5	2.5
$MgSO_4$ (% on o.d.p)	--	0.5	--	--	--
EDTA (% on o.d.p)	--	--	--	2	2

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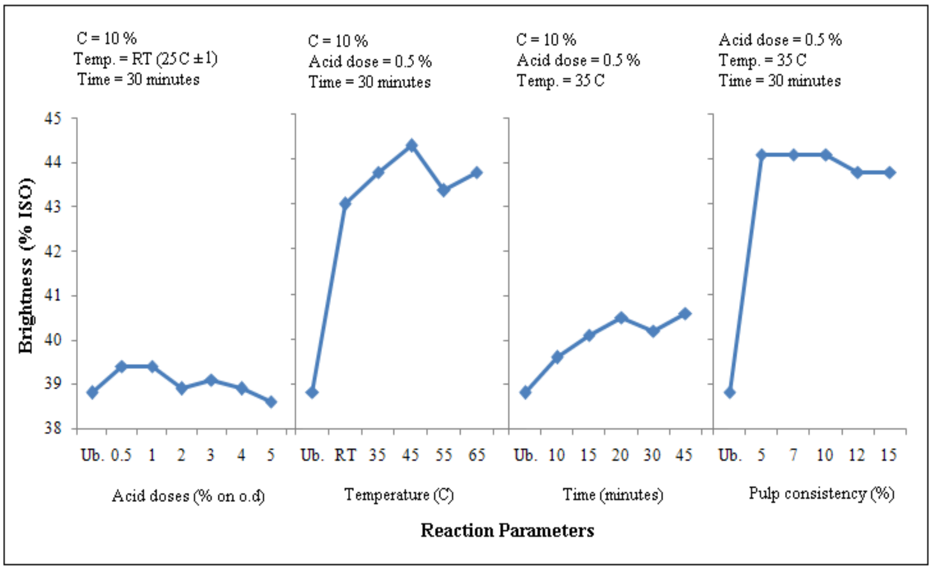

Fig. 1 Effect of different reaction conditions during A-Stage on brightness of hand sheets
(Ub stands for unbleached, C is consistency, RT for room temperature i.e. 25 ± 26 °C and temp for temperature)

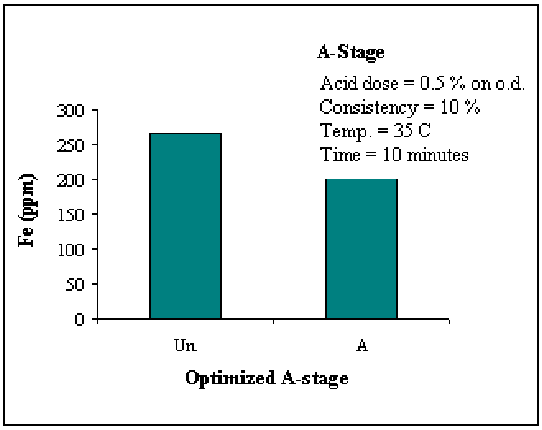

Fig. 2 Effect on iron of pulp at optimized acidification stage

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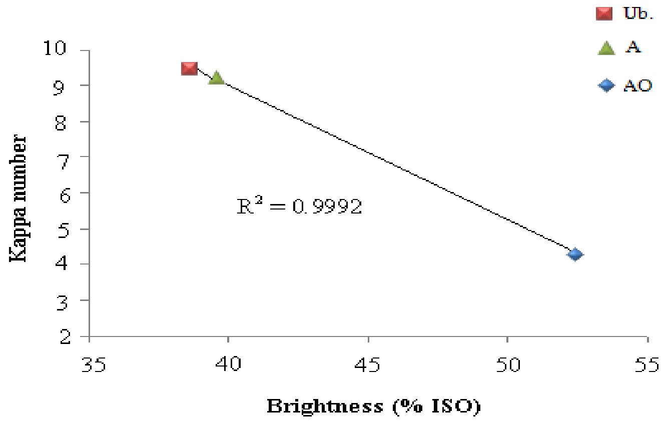

Fig. 3. Correlation of kappa number and brightness for AQ-soda pulp samples treated at various stages

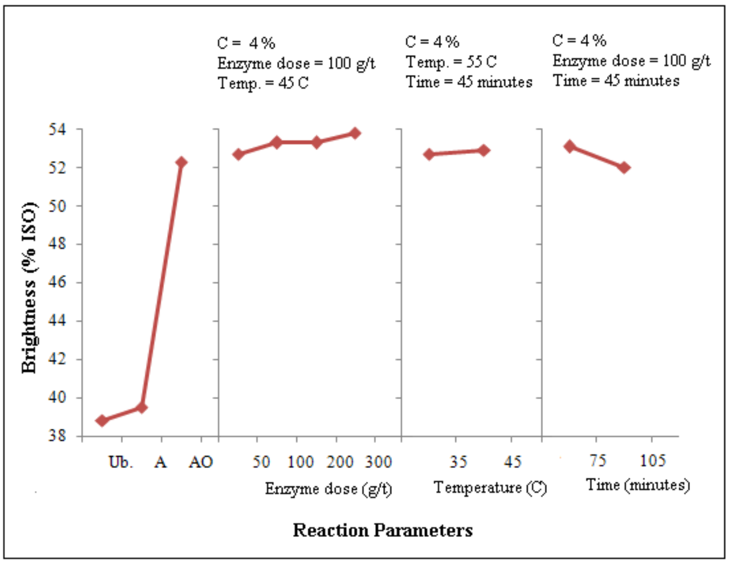

Fig. 4 Effect of different reaction conditions during X-Stage on brightness of hand sheets

(Ub stands for unbleached, A for acidified pulp at 0.5 % acid dose on o.d. pulp basis, AO for oxygen delignified, C is consistency and temp is temperature)

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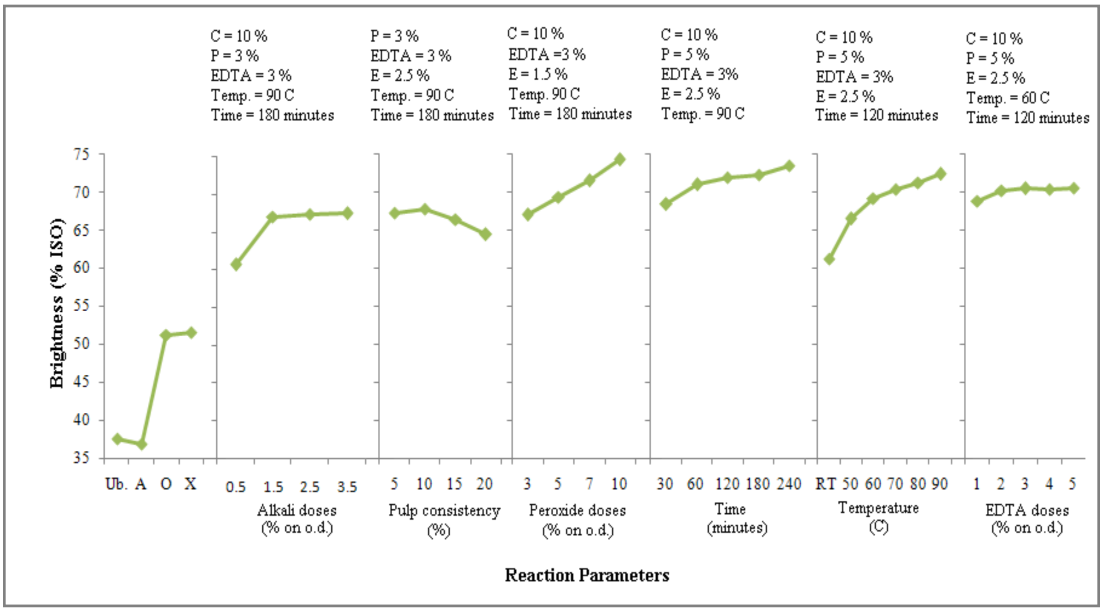

Fig. 5 Effect of different reaction conditions during P-Stage on brightness of hand sheets

(Ub stands for unbleached, A for acidified pulp at 0.5 % acid dose on o.d. pulp basis, AO for oxygen delignified pulp, AOX for enzymatic treated pulp, C is consistency, P is peroxide, E is alkali and temp is temperature)

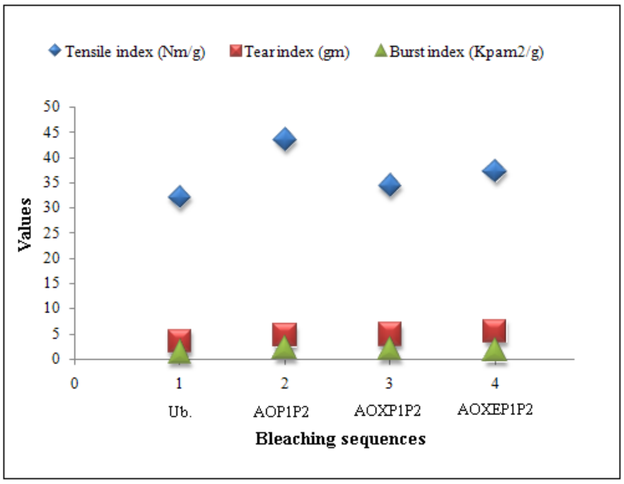

Fig. 6 Effect of different optimized bleaching sequences on strength properties of hand sheets

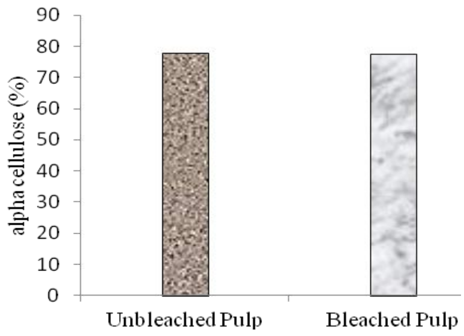

Fig. 7 Comparison of alpha-cellulose contents of bleached and unbleached pulp

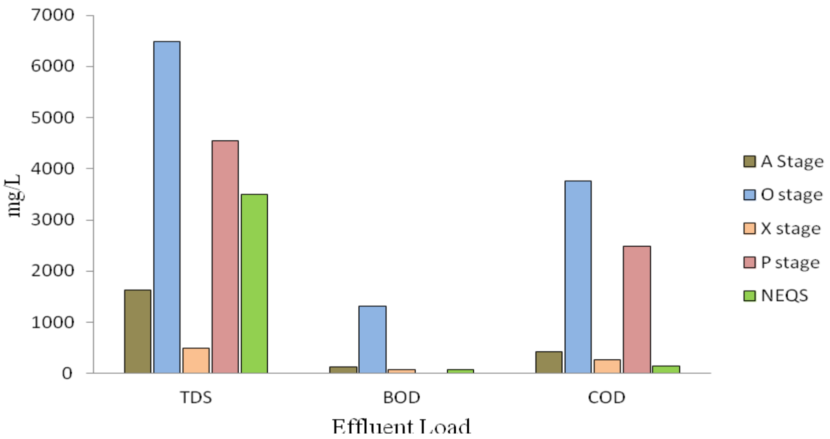

Fig. 8. Effluent load collected from various optimized bleaching stages