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Parameters of Effluent from Bleaching Pulp with Potassium Hydroxide and Calcium Hypochlorite

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Abstract

Conventional process of bleaching pulp produces black liquor that has adverse effects on the environment due to the type of chemical that is used in the process. Since the dark pulp needs to be bleached for a better pulp, two chemicals were used in the bleaching process to determine their pollution load in the effluent. In this research, kenaf stem pulp was bleached with 20%, 60% and 90% of potassium hydroxide and calcium hypochlorite separately at intervals of 1, 2 and 3hours.Their effluents were analysed considering chemical oxygen demand, biochemical oxygen demand, total suspended solid, total solid and pH to determine the environmental quality. The results showed that potassium hydroxide bleaching effluent has lower chemical oxygen demand and biochemical oxygen demand values during the 3 hour periods at the three different concentrations than that from calcium hypochlorite. At 20% potassium hydroxide effluent has higher values of total solid than calcium hypochlorite during the 3 hour periods but lower total solid values at 60% and 90% during the 3 hour periods. The values of total suspended solid were higher in potassium hydroxide effluent during the 3 hour periods at the three different concentrations. The pH remained alkaline in all the effluents. The bleached pulps have different physical appearances with 60% concentration for 2 hours being the best on physical examination because 20% was not well pulped while 90% was over pulped. The statistical analysis result showed that chemical, time and concentration interactions were mostly significant ($p > 0.05$).

Keywords: Kenaf pulp; Bleaching; Chemical oxygen demand; Biochemical oxygen demand; Potassium hydroxide; Calcium hypochlorite; Total solid

Abbreviation: TSS: Total Suspended Solids; TS: Total Solids; TDS: Total Dissolved Solids; COD: Chemical Oxygen Demand; BOD: Biological Oxygen Demand; ECF: Elemental Chlorine Free; TCF: Totally Chlorine Free

Introduction

Man has been using the natural resources since beginning of civilization. The primitive man was totally depend on natural resources for his existence. With the increase in population, the over exploitation of the resources led to its destruction [1]. Many of the industries are totally depend on natural resources for their raw material. This over dependence is the cause of various environmental problems such as pollution of land, water, and air. Industrial wastes resulting from all manufacturing industries are a major cause of air, water, and land pollution. Paper mill is a major industrial sector utilizing a huge amount of ligno- cellulosic materials and water during the manufacturing process, and release chlorinated lingo sulphonic acids, chlorinated resin acids, chlorinated phenols and chlorinated hydrocarbons in the effluents [2]. Paper industry uses wide variety raw materials employing different type of pulping and bleaching processes depending on the type of raw materials and requirement of pulp furnish for final paper making. The paper mill effluents are highly coloured, highly toxic and are a major source of aquatic pollution. Many chemicals have been identified in effluents which are produced at different stages of papermaking.

Their toxic nature is derived from the presence of several naturally occurring and xenobiotic compounds which are formed and released during various stages of papermaking [3]. The pulp and paper mills rank high in terms of water use during paper production on the other hand they contribute to pollution loads in rivers through effluent discharge [4]. Effluent quality is commonly judged on the basis of such aggregate characteristics as biochemical oxygen demand, chemical oxygen demand, total suspended solids (TSS), total solids (TS), total dissolved solids (TDS) turbidity, pH, color etc. [5,6]. The major problems of the wastewaters from paper mills are high organic content, dark brown coloration, adsorbable organic halide and toxic pollutants. These pollutants are mixed together and a complex-colored effluent is formed with high chemical oxygen demand (COD), biological oxygen demand (BOD) and total dissolved solid pollution load and color contributing substances from the pulp paper mill effluents causes serious aquatic and soil pollution [7]. Both unbleached and bleached pulp is produced to meet the requirement of paper.

Figure 1:

Requirement of bleaching chemicals are also varying depending on the quality of paper and brightness and environmental considerations for cleaner and greener paper [8]. Bleaching is engaged on the brown pulp obtained after pulping in order to meet the desired colour dictated by product standards. Bleaching is often undertaken, primarily for two purposes: first, to increase brightness; second, to remove residual lignin. Lignin can be thought of as the 'glue' holding the cellulose fibres of wood together [8]. Wastewater from the pulp bleaching end is responsible for most of the colour, organic matter and toxicity of water discharge of the industry [9]. The pulp produce by chemical process requires bleaching to produce bright pulps. The operation generates high environmental load and poses serious threat to the environment [10].

The need to reduce the environmental impact of bleaching has led to development of other bleaching methods. Modified bleaching techniques have made possible the development of elemental chlorine free (ECF) and totally chlorine free (TCF) processes that have alleviated the problem of the environmental impacts of bleaching effluent [11]. The effects of organochlorine discharges on people and on natural ecological systems are not fully understood or quantified. It is especially difficult to quantify the levels of discharge of particular types of organochlorines from pulp mills. The uncertainty surrounding the effects of chlorine bleaching is reflected in divergent views about its control [12,13]. The environmental impacts of the effluent from bleaching vary and can depend on the fibre feedstock, the pulping process, the bleaching sequence, and whether the effluent is treated or untreated. Regardless of the method of bleaching, the effluent may have certain characteristics which can damage the environment, such as suspended solids and the capacity to deplete oxygen from the receiving waters [8]. However, a feature of chlorine bleached effluent is the presence of organochlorines, some organochlorines break down quickly; and some, including dioxins and furans, have long lives [8]. New pulping and bleaching technology, more stringent effluent regulations, environ-mental pressure groups, and new market demands have had a considerable influence on modern bleaching practices [12]. In our part of the world, conventional bleaching is still used quite extensively in some mills.

In this study, effluents from bleaching kenaf pulp with potassium hydroxide and calcium hypochlorite in a single stage process under laboratory conditions were analysed to check the effects of chemicals, concentrations and time on their physicochemical parameters.

Materials and Methods

The pulped kenaf stem was bleached with 20%, 60% and 90% concentrations of potassium hydroxide and calcium hypochlorite and the bleaching time were varied from 1hr, 2hrs and 3hrs at room temperature. At the end of each bleaching, the sample was filtered with a fine mesh sieve of size 0.027 to get the effluent used in the analyses. The effluents were stored in plastic containers which were refrigerated at 4^{oc} until needed. The tests were carried out in triplicate and each value is an average of three samples. The effluent was analysed using the Standard Method for Examination of Water and Wastewater [14]. The parameters determined were COD, BOD, pH, TSS, and TS. The COD was determined with potassium dichromate, closed reflux method, BOD was determined by 5 days incubation at 20^{0c}, TSS and TS by gravimetric, residue drying at 100^{0c} and pH by handy pH meter. THREE-WAY ANOVA was used for analysis of the data.

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Table 1: Mean and SE of parameters over the concentration, time and chemical factors for bleaching.

Concentration	Time	Chemical	COD (mg/l)	BOD (mg/l)	pH	TS (mg/l)	TSS (mg/l)
			Mean + SE	Mean + SE	Mean + SE	Mean + SE	Mean + SE
20%^{a}	1 Hour	Ca(CIO)2	762 + 736.77^{a}	20 + 6.25	13.7 + 0.04	3832 + 3430.88	360 + 2269.93
		KOH	460 + 736.77^{b}	16.88 + 6.25	13.7 + 0.04	12875+2269.93	7837+3430.88
	2 Hours	Ca(CIO)2	4600 + 736.77^{a}	35 + 6.25	13.79 + 0.04	1105 + 3430.88	367.5 + 2269.93
		KOH	388 + 736.77^{b}	13.75 + 6.25	13.5 + 0.04	16252.5+2269.93	9094+3430.88
	3 Hours	Ca(CIO)2	1650 + 736.77^{a}	18.13 + 6.25	13.56 + 0.04	473 + 3430.88	330 + 2269.93
		KOH	408 + 736.77^{b}	17.5 + 6.25	12.43 + 0.04	6037.5+2269.93	936+3430.88
60%^{b}	1 Hour	Ca(CIO)2	822 + 736.77^{a}	18.13 + 6.25	14.19 + 0.04	14980 + 3430.88	490 + 2269.93
		KOH	996 + 736.77^{b}	12.5 + 6.25	13.56 + 0.04	5090+3430.88	3588 + 2269.93
	2 Hours	Ca(CIO)2	6800 + 736.77^{a}	25.63 + 6.25	14.08 + 0.04	17407 + 3430.88	820 + 2269.93
		KOH	784 + 736.77^{b}	9.38 + 6.25	13.52 + 0.04	4492.5 + 3430.88	2849+ 2269.93
	3 Hours	Ca(CIO)2	6600 + 736.77^{a}	25 + 6.25	14.28 + 0.04	10365 + 3430.88	507.5 + 2269.93
		KOH	312 + 736.77^{b}	16.88 + 6.25	11.47 + 0.04	2043 + 3430.88	2037.5 + 2269.93
90%^{b}	1 Hour	Ca(CIO)2	3800 + 736.77^{a}	7.5 + 6.25	13.21 + 0.04	18685 + 3430.88	537.5 + 2269.93
		KOH	6200 + 736.77^{b}	14.38 + 6.25	12.4 + 0.04	1221 + 3430.88	572.5 + 2269.93
	2 Hours	Ca(CIO)2	6400 + 736.77^{a}	33.13 + 6.25	13.27 + 0.04	8200 + 3430.88	837.5 + 2269.93
		KOH	846 + 736.77^{b}	18.75 + 6.25	12.5 + 0.04	562.5 + 3430.88	536+ 2269.93
	3 Hours	Ca(CIO)2	3240 + 736.77^{a}	18.75 + 6.25	13.29 + 0.04	3329 + 3430.88	1970 + 2269.93
		KOH	678 + 736.77^{b}	27.5 + 6.25	11.56 + 0.04	4572 + 3430.88	550 + 2269.93

Table 2: Mean and SE of **COD (mg/l)** over the concentrations, time and chemicals for Bleaching.

Concentration	Time	Chemical	Mean + SE
20%^{a}	1 Hour	Ca(CIO)2	762 + 736.77^{a}
		KOH	460 + 736.77^{b}
	2 Hours	Ca(CIO)2	4600 + 736.77^{a}
		KOH	388 + 736.77^{b}
	3 Hours	Ca(CIO)2	1650 + 736.77^{a}
		KOH	408 + 736.77^{b}
60%^{b}	1 Hour	Ca(CIO)2	822 + 736.77^{a}
		KOH	996 + 736.77^{b}
	2 Hours	Ca(CIO)2	6800 + 736.77^{a}
		KOH	784 + 736.77^{b}
	3 Hours	Ca(CIO)2	6600 + 736.77^{a}
		KOH	312 + 736.77^{b}
90%^{b}	1 Hour	Ca(CIO)2	3800 + 736.77^{a}
		KOH	6200 + 736.77^{b}
	2 Hours	Ca(CIO)2	6400 + 736.77^{a}
		KOH	846 + 736.77^{b}
	3 Hours	Ca(CIO)2	3240 + 736.77^{a}
		KOH	678 + 736.77^{b}

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ANOVA Table

Source	Sum of Squares	df	Mean Square	F	Sig.
Chemical	61894934.778	1	61894933.778	57.012	.000
Time	10443254.222	2	5221627.111	4.81	0.021
Conc	28285270.222	2	14142635.111	13.027	.000
Chemical * Time	56799056.889	2	28399528.444	26.159	.000
Chemical * Conc	9085430.222	2	4542715.111	4.184	0.032
Time * Conc	35811596.444	4	8952899.111	8.247	0.001
Chemical * Time * Conc	10527527.111	4	2631881.778	2.424	0.086
Error	19541704.000	18	1085650.222		
Corrected Total	232388772.889	35			

R Squared = .916 (Adjusted R Squared = .836).

The Anova table above reveals that all the factors and all the interactions except the three (3) level interactions were significant ($p < 0.05$).

Table 3: Mean and SE of **BOD (mg/l)** over the concentrations, time and chemicals for Bleaching.

Concentration	Time	Chemical	Mean + SE
20%[a]	1 Hour	Ca(ClO)2	20 + 6.25
		KOH	16.88 + 6.25
	2 Hours	Ca(ClO)2	35 + 6.25
		KOH	13.75 + 6.25
	3 Hours	Ca(ClO)2	18.13 + 6.25
		KOH	17.5 + 6.25
60%[a]	1 Hour	Ca(ClO)2	18.13 + 6.25
		KOH	12.5 + 6.25
	2 Hours	Ca(ClO)2	25.63 + 6.25
		KOH	9.38 + 6.25
	3 Hours	Ca(ClO)2	25 + 6.25
		KOH	16.88 + 6.25
90%[a]	1 Hour	Ca(ClO)2	7.5 + 6.25
		KOH	14.38 + 6.25
	2 Hours	Ca(ClO)2	33.13 + 6.25
		KOH	18.75 + 6.25
	3 Hours	Ca(ClO)2	18.75 + 6.25
		KOH	27.5 + 6.25

ANOVA Table

Source	Sum of Squares	df	Mean Square	F	Sig.
Chemical	321.007	1	321.007	4.109	0.058
Time	384.635	2	192.318	2.462	0.113
Conc	38.542	2	19.271	0.247	0.784
Chemical * Time	577.170	2	288.585	3.694	0.045
Chemical * Conc	187.847	2	93.924	1.202	0.324
Time * Conc	293.229	4	73.307	0.938	0.464
Chemical * Time * Conc	67.882	4	16.97	0.217	0.925
Error	1406.250	18	78.125		
Corrected Total	3276.562	35			

R Squared = .571 (Adjusted R Squared = .165).

Table above shows that only the time – chemical interaction is significant ($p < 0.05$) while the rest were not.

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Table 4: Mean and SE of **pH** over the concentrations, time and chemicals for Bleaching.

Concentration	Time	Chemical	Mean + SE
20%[a]	1 Hour	Ca(ClO)2	12.76 + 0.14[a]
		KOH	9.8 + 0.14[b]
	2 Hours	Ca(ClO)2	12.14 + 0.14[a]
		KOH	12.5 + 0.14[a]
	3 Hours	Ca(ClO)2	11.96 + 0.14[a]
		KOH	11.7 + 0.14[a]
60%[b]	1 Hour	Ca(ClO)2	13.85 + 0.14[a]
		KOH	13.31 + 0.14[a]
	2 Hours	Ca(ClO)2	13.12 + 0.14[a]
		KOH	13.41 + 0.14[a]
	3 Hours	Ca(ClO)2	8.95 + 0.14[a]
		KOH	2.24 + 0.14[b]
90%[c]	1 Hour	Ca(ClO)2	11.86 + 0.14[a]
		KOH	1.86 + 0.14[b]
	2 Hours	Ca(ClO)2	10.96 + 0.14[a]
		KOH	2.4 + 0.14[b]
	3 Hours	Ca(ClO)2	8.89 + 0.14[a]
		KOH	3.27 + 0.14[b]

ANOVA Table

Source	Sum of Squares	df	Mean Square	F	Sig.
Chemical	128.293	1	128.293	3486.233	0
Time	64.173	2	32.087	871.917	0
Conc	188.110	2	94.055	2555.849	0
Chemical * Time	5.997	2	2.998	81.474	0
Chemical * Conc	85.291	2	42.645	1158.845	0
Time * Conc	102.787	4	25.697	698.283	0
Chemical * Time * Conc	39.531	4	9.883	268.551	0
Error	0.662	18	0.037		
Corrected Total	614.845	35			

R Squared = .999 (Adjusted R Squared = .998)

Table above shows that all the factors and interactions are significant ($p < 0.05$).

Table 5: Mean and SE of **TS (mg/l)** over the concentrations, time and chemicals for Bleaching.

Concentration	Time	Chemical	Mean + SE
20%[a]	1 Hour	Ca(ClO)2	3832 + 3430.88[a]
		KOH	7837 + 3430.88[a]
	2 Hours	Ca(ClO)2	1105 + 3430.88[a]
		KOH	9094 + 3430.88[b]
	3 Hours	Ca(ClO)2	473 + 3430.88[a]
		KOH	936 + 3430.88[a]

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60%^{b}	1 Hour	Ca(CIO)2	14980 + 3430.88^{a}
		KOH	3588 + 3430.88^{b}
	2 Hours	Ca(CIO)2	17407 + 3430.88^{a}
		KOH	2849 + 3430.88^{b}
	3 Hours	Ca(CIO)2	10365 + 3430.88^{a}
		KOH	2043 + 3430.88^{b}
90%^{ab}	1 Hour	Ca(CIO)2	18685 + 3430.88^{a}
		KOH	1221 + 3430.88^{b}
	2 Hours	Ca(CIO)2	8200 + 3430.88^{a}
		KOH	536 + 3430.88^{b}
	3 Hours	Ca(CIO)2	3329 + 3430.88^{a}
		KOH	4572 + 3430.88^{b}

ANOVA Table

Source	Sum of Squares	df	Mean Square	F	Sig.
Chemical	232054444.444	1	232054444.444	9.857	0.006
Time	137025850.889	2	68512925.444	2.91	0.080
Conc	130359500.222	2	65179750.111	2.769	0.089
Chemical * Time	55919537.556	2	27959768.778	1.188	0.328
Chemical * Conc	401358908.222	2	200679454.111	8.524	0.002
Time * Conc	48529081.778	4	12132270.444	0.515	0.725
Chemical * Time * Conc	166987577.778	4	41746894.444	1.773	0.178
Error	423753788.000	18	23541877.111		
Corrected Total	1595988689.889	35			

b. R Squared = .734 (Adjusted R Squared = .484)

The Anova table above shows that while all the factors are not significant, only chemical – concentration interaction is significant ($p < 0.05$).

Table 6: Mean and SE of **TSS (mg/l)** over the concentrations, time and chemicals for bleaching.

Concentration	Time	Chemical	Mean + SE
20%^{a}	1 Hour	Ca(CIO)2	360 + 2269.93^{a}
		KOH	12875 + 2269.93^{b}
	2 Hours	Ca(CIO)2	367.5 + 2269.93^{a}
		KOH	16252.5 + 2269.93^{b}
	3 Hours	Ca(CIO)2	330 + 2269.93^{a}
		KOH	6037.5 + 2269.93^{b}
60%^{b}	1 Hour	Ca(CIO)2	490 + 2269.93^{a}
		KOH	5090 + 2269.93^{b}
	2 Hours	Ca(CIO)2	820 + 2269.93^{a}
		KOH	4492.5 + 2269.93^{a}
	3 Hours	Ca(CIO)2	507.5 + 2269.93^{a}
		KOH	2037.5 + 2269.93^{a}
90%^{b}	1 Hour	Ca(CIO)2	537.5 + 2269.93^{a}
		KOH	572.5 + 2269.93^{a}
	2 Hours	Ca(CIO)2	837.5 + 2269.93^{a}
		KOH	562.5 + 2269.93^{a}
	3 Hours	Ca(CIO)2	1970 + 2269.93^{a}
		KOH	550 + 2269.93^{a}

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ANOVA Table

Source	Sum of Squares	df	Mean Square	F	Sig.
Chemical	198340277.778	1	198340277.778	19.247	.000
Time	25038179.167	2	12519089.583	1.215	0.320
Conc	173646037.500	2	86823018.750	8.425	0.003
Chemical * Time	34919926.389	2	17459963.194	1.694	0.212
Chemical * Conc	222381776.389	2	111190888.194	10.790	0.001
Time * Conc	36296145.833	4	9074036.458	0.881	0.495
Chemical * Time * Conc	24973431.944	4	6243357.986	0.606	0.663
Error	185493175.000	18	10305176.389		
Corrected Total	901088950.000	35			

R Squared = .794 (Adjusted R Squared = .600)

The table above shows that chemical, concentration and chemical – concentration interaction were significant ($p < 0.05$) while the remainder were not.

With 20% concentration of KOH during the 3 hours period, COD, BOD, TSS, TS and pH recorded values that were not actually consistent. COD which is the measure of amount of oxygen required to breakdown both organic and inorganic matters ranged between 388mg/L and 460mg/L while BOD which measures the organic loading of streams and thereby quantifies the dissolved oxygen levels ranged between 13.75mg/L and 17.5mg/L. TS values which is the total dissolved and un-dissolved matter with KOH were between 6037mg/L and 16252.5mg/L while TSS were between 936mg/L and 9094mg/L which did not follow a particular pattern of increase or decrease. The pH which is the hydrogen-ion concentration is a measure of acidity and basicity of an aqueous solution and is an important parameter to check the quality of effluent. The pH range of 12.43 to 13.7 was recorded with KOH at 20% concentration during the 3 hours period which showed highly alkaline effluent. The result of 20% concentration of $Ca(CIO)^2$ bleaching showed COD with values between 762mg/L and 4600mg/L with highest value of 4600mg/L after 2hrs while the BOD ranges between 18.13mg/L and 35mg/L with 2hrs also having the highest value of 35mg/L. TSS values recorded were between 330 and 367mg/L while TS decreased from 3832mg/L to 473 which may be as a result of decrease in total dissolved oxygen. The pH still maintained its alkalinity nature with hypochlorite.

The result of analyses of effluent from bleaching with 60% concentration of KOH showed that the value of COD decreased with increase in time from 996mg/L to 312mg/L unlike the BOD which its lowest value was recorded after 2hrs. TS and TSS from KOH effluent also recorded reduced values as the time increased having the lowest values after 3hrs which showed that more degradation occurred while the pH remains alkaline. With 60% concentration of $Ca(CIO)^2$ bleaching, both COD and BOD values increased with increase in time with both recording their highest values after 2hrs. The solids (TSS & TS) increased with time with their highest values being recorded after 2hrs. The pH of the effluent is still highly alkaline.

The effluent from bleaching with 90% concentration of KOH has COD values that decreased with increase in bleaching time while the BOD increased with increase in time of bleaching. The results of the solids showed that TSS did not have significant change during the 3-hour period while the TS recorded wide margin between the 3 hour periods which may be due to the influence of dissolved solids. The result of 90% concentration of $Ca(CIO)^2$ showed highest COD and BOD after 2 hours of bleaching. TS decreased with increase in bleaching time while TSS had its lowest value after 1 hour.

The effluent of $Ca(CIO)^2$ bleaching has its highest values of COD and BOD after 2 hours bleaching in all the three concentrations with 60% having the highest value of COD(6800mg/L) and 20% having the highest value of BOD(35mg/L). The result from KOH bleaching effluent showed highest values of COD after 1 hour bleaching in all the three concentrations with 90% having the highest value(6200mg/L) while the highest values of BOD were recorded after 3 hours in all the concentrations with 90% (27.5mg/L) having the highest value as well. The result of TSS from $Ca(CIO)^2$ effluent showed highest value(1970mg/L) at 90% after 3 hours bleaching and the highest value(18685mg/L) of TS also at 90% after 1 hour bleaching. Both TS and TSS of KOH effluent recorded highest values (16252.5 and 9094mg/L) from 20% concentration after 2 hours of bleaching.

Conclusion

The results of the analysed effluents showed that COD and BOD from KOH bleaching are lower than those from $Ca(CIO)^2$ during the 3 hour periods at the three different concentrations. The values of TSS and TS varied at 20%, 60% and 90% for both chemicals. The THREE-WAY ANOVA showed that chemical, time and concentration interactions were mostly significant ($p > 0.05$).

The KOH effluent is better than that of $Ca(CIO)^2$ despite not meeting the standard of effluent discharge but with more green

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technologies, its environmental quality will improve. The KOH will add to soil fertility as well as help in neutralizing the soil acidity. The result can also help in addressing the controversy of TCF and ECF bleaching of pulp. More research is also needed to check other effluent characteristics for better understanding of the process.

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