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Contents of Sludge from Kenaf Pulp Bleaching with Potassium Hydroxide and Calcium Hypochlorite

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Abstract:
The content of pulp and paper sludge depends on the raw materials, bleaching chemicals, and the manufacturing process. In this research, kenaf pulp was bleached with 20%, 60%, and 90% concentrations of potassium hydroxide and calcium hypochlorite at 3hr intervals at room temperature. The effluents from the bleaching processes were filtered to get the sludge. The sludge was analyzed for ash, phosphorus, carbon, and nitrogen. The sludge showed high content of carbon (17.77%/11.73% and phosphorus (12.53mg/l/12.6mg/l) from both potassium hydroxide and calcium hydroxide bleaching. Carbon contents were 17.77% after 2hr bleaching with 60% concentration of potassium hydroxide and 17.73% after 2hr bleaching with 90% concentration of calcium hypochlorite. Phosphorus contents were 12.53mg/l and 12.6mg/l after bleaching with a 90% concentration of potassium hydroxide and calcium hypochlorite for 3hrs. Nitrogen and ash showed low values with the lowest (0.12% and 0.23%) from bleaching with a 90% concentration of potassium hydroxide. The sludge can be used to amend soils that their nutrients have been eroded either by over-farming or logging and replenish it, making them fertile again.

Keywords*: Bleaching, Calcium hypochlorite, Potassium hydroxide, Pulp, Sludge.*

INTRODUCTION

The rapid increase in population and the increasing demand for industrial establishments, and the overexploitation of available resources to meet human requirements have created problems such as pollution of the land, air, and water environments. Humanity is suffering from a phenomenon called global warming, which is a natural phenomenon that, unfortunately, without a doubt, has been accelerated immensely by human activity [1]. Pulp and paper mills generate a significant amount of biodegradable sludge during the papermaking and pulp-making stages [2]. A huge quantity of the sludge produced by paper mills with large usage of paper is considered as one of the most serious environmental problems [3]. The increasing amount of sludge and its consequent treatments are very sensitive environmental problems [4]. The economics and environmental impacts of the pulp and paper industry all depend on the efficient treatment of wastewater produced in the processes [5]. The disposal of industrial sludge from the effluent treatment plant is a global concern to the industries, not a unique problem for the pulp and paper industry. Among the four major modes of sludge disposal; sea discharge, landfill, incineration, and land application, nowadays, most of the industries disposing of the sludge by only land application. The final disposal route for excess sludge generated by wastewater treatment is becoming a serious issue mainly due to the growth of population and sludge accumulation in large cities and growth in the amount and complexity of the related industrial activities [6]. The sludge" is a generic term for the residue that results from pulp and papermaking. Generally, it is the solid residue recovered from the wastewater stream of the pulping and papermaking process [7].

Sustainable use and management of natural resources that are byproducts of industrial production can play an important role in support of the raw material demand [8]. The large amount of waste paper involved in production processes in paper mills necessitates the generation of fibrous sludge waste (FSW). FSW contains lignocellulosic fibers, and various inorganic materials such as kaolin, calcium carbonate, titanium dioxide are considerably produced [9]. These sludge wastes from paper-recycling factories are either burned or used as landfills. However, the incineration of these wastes, which contain a large number of inorganic materials, generates gases with a low calorific value from chimneys, thereby polluting the atmosphere. Further, the use of FSW as landfills drastically reduces the fertility of the soil and causes environmental pollution. Environmental concerns and regulations have forced paper mills to search for an efficient strategy for recycling the FSW generated as a byproduct in the manufacturing of paper [10]. Some wastes are rich in nutrients that can be returned to the environment, reducing the amount disposed of in landfills. In general, these wastes are considered non-hazardous wastes [11], [12] and. [13]. The wood, fresh process water, and pulping (including recovery make-up), bleaching, and papermaking chemicals introduce certain elements into the pulp and paper system. Corrosion and wear, as well as surfactants and polymers, added to control scales and to aid dewatering all supplement the elemental input. These elements leave the system in the pulp and paper products, the effluents, air emissions, and solid wastes [14]. Those in the effluents appear in the sludges and in the water from the effluent treatment. The levels of the individual elements depend on the amounts entering and leaving the system. Pulp and paper mill sludges contain cellulosic fibers and chemical compounds of nitrogen, phosphorus, potassium, and other elements. They can serve to increase the water-holding capacity of soils (especially sandy soils), improve soil structure, and supply nutrients for plant growth. The nitrogen content of the sludge is most important inland spreading. An important measure of the suitability of sludge for land spreading agriculturally is its ratio of carbon to nitrogen (C/N). [15]. This ratio is an indication of the tendency of the material to release nitrogen, on the one hand, or, on the other, to immobilize it. Economic combustion of pulp and paper sludges for disposal or energy recovery is dependent on their solids and organic contents. The latter determines the heating value of the sludge on a dry solids basis, as influenced by the inorganic or ash contents [16]

Although there is worldwide shrinking in the graphic paper market due to digitization, there is overall continual growth in the pulp and paper industry because of the increasing market demand for several other applications, such as cardboard and packaging paper, tissue paper, pulp for personal hygiene products, and textile applications [17] To obtain whiter and non-degradable pulp, bleaching is a necessary process in pulping [18]. Chlorine has been widely used for bleaching; however, its use is limited because it pollutes the environment and is toxic [19]
This paper considers the effects of time. Concentration and chemical interactions under laboratory conditions on the ash, phosphorus, nitrogen, and carbon content of sludges generated during two chemical bleaching processes.

MATERIALS AND METHODS

Kenaf stem was manually chopped into 1 to 4 cm long, washed with warm water to remove dirt and dust. The washed kenaf was dewatered to a solid content of 40% to 45%. The dewatered kenaf was pulped with different concentrations of sodium hydroxide and formic acid at different time intervals. In the end, the pulp from each cooking was bleached with 20%, 60% and
90% concentrations of potassium hydroxide (KOH) and calcium hypochlorite (ca(CIO)2 and the bleaching time were varied from 1hr, 2 hrs, and 3 hrs at room temperature. At the end of each period, the sample was filtered with a fine-mesh sieve of size 0.027 mm to get effluent, and the effluent was filtered with a filter paper to get the sludge used in the analyses. The sludge samples were then air-dried and screened to remove other contaminating materials. The screened raw materials were ground and placed in an airtight container to balance the moisture content and then used for chemical analysis. The tests were carried out in triplicate, and each value is an average of three samples.

Determination of Nitrogen content

The nitrogen content of the sample was determined by using Kjedahl technique [20]. The method involves digestion of samples, the distillation of digests, and titration of distillate.

Calculation: $\% \text{ N} = \frac{T \times 14.01 \times Molarity\ of\ HCl\ (0.1) \times 100 \times 10}{Weight\ of\ sample\ taken\ (2g) \times 1000}$

Where T = (sample titre – blank titre)

Determination of Carbon content

The colorimetric method of [21] was used to calculate the total carbon

$$\% \text{ Ash (wet basis)} = \frac{M_{ASH}}{M_{WET}} \times 100$$

M_{ASH} = Weight of ash
M_{WET} = Original weight

The phosphorus content is calculated according to following formular [22]

$$W_P = \frac{C \text{ x } V}{m \text{ x } mt}$$

W_P = content of phosphorus in g/l

C = concentration of phosphorus measured in the extraction solution, mg/l

V = Volume of the volumetric flask, ml

m = mass of the test sample

mt = dry mass of the testsample

Fig. 1: Pulp bleaching

Results and Discussion

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

			Nitrogen (%)	Carbon(%)	Ash (%)	Phosphorus (mg/100ml)
Concentration	Time	Chemical	Mean + SE	Mean + SE	Mean + SE	Mean + SE
20%	1 Hour	Ca(CIO)2	1.83 + 0.11	0.11 + 0.09	0.98 + 0.02	0.97 + 0.07
		KOH				
	2 Hours	Ca(CIO)2	2.67 + 0.11	0.07 + 0.09	0.66 + 0.02	1.1 + 0.07
		KOH				
	3 Hours	Ca(CIO)2	8.67 + 0.11	0.07 + 0.09	0.87 + 0.02	10.2 + 0.07
		KOH				
60%	1 Hour	Ca(CIO)2	5.3+0.11	0.02 + 0.09	0.97 + 0.02	2.73 + 0.07
		KOH	0.13 + 0.11	23.2 + 0.09	0.97 + 0.02	12.57 + 0.07
	2 Hours	Ca(CIO)2	5.1 + 0.11	0.02 + 0.09	1.26 + 0.02	11.23 + 0.07
		KOH	0.12 + 0.11	17.77 + 0.09	0.99 + 0.02	2.03 + 0.07
	3 Hours	Ca(CIO)2	2.83 + 0.11	0.03 + 0.09	1.29 + 0.02	3.37 + 0.07
		KOH	0.13 + 0.11	10.47 + 0.09	0.36 + 0.02	10.53 + 0.07
90%	1 Hour	Ca(CIO)2	0.29 + 0.11	4.7 + 0.09	1.05 + 0.02	9.3 + 0.07
		KOH	0.17 + 0.11	9.6 + 0.09	0.86 + 0.02	1.77 + 0.07
	2 Hours	Ca(CIO)2	0.16 + 0.11	17.73 + 0.09	2.78 + 0.02	9.43 + 0.07
		KOH	0.17 + 0.11	11.77 + 0.09	0.24 + 0.02	8.5 + 0.07
	3 Hours	Ca(CIO)2	0.15 + 0.11	12.57 + 0.09	0.27 + 0.02	12.6 + 0.07
		KOH	0.12 + 0.11	5.2 + 0.09	0.77 + 0.02	12.53 + 0.07

Table 1 presents the values of ash, phosphorus, carbon, and nitrogen from sludge obtained from bleaching kenaf pulp with 20%, 60%, and 90% concentrations of potassium hydroxide (KOH) and calcium hypochlorite ($Ca(CIO)^2$). The result from the analysis of the sludge from KOH bleaching using 20% concentration did not show the presence of ash, phosphorus, carbon, and nitrogen throughout the three-hour interval. This was in agreement with [23] using formic acid. This may be attributed to incomplete breakdown due to a low concentration of potassium hydroxide. With calcium hypochlorite, the four minerals were present at varying percentages as in [25] with sodium hydroxide. Nitrogen and phosphorus increased with increase in bleaching time from 1hr to 3hrs (N= 1.83%/8.67%, P=0.97mg/100ml/10.2mg/100ml) while carbon and ash decreased with increase in bleaching time (C= 0.11%/0.07%, A=0.98%/0.87%)

The result of sludge analysis from 60% concentrations of potassium hydroxide and calcium hypochlorite showed that some minerals increased with an increase in bleaching time while some decreased with an increase in bleaching time. With calcium hypochlorite bleaching, nitrogen decreased with an increase in bleaching time, having its highest value (5.3%) after 1hr bleaching and lowest (2,83%) after 3hr bleaching but almost remained constant with potassium hydroxide bleaching. The reported value of carbon from analyzing sludge obtained from using 60% concentrations of potassium hydroxide and calcium hypochlorite showed a sharp decrease in the content from 1hr to 3hr bleaching with 23.2% as highest and 10.47% as the lowest with potassium hydroxide. Bleaching with calcium hypochlorite did not show any noticeable change in the value of carbon. Ash content of sludges from bleaching with potassium hydroxide and calcium hypochlorite only showed marginal increment with close values. Sludges from potassium hydroxide and calcium hypochlorite bleaching showed variations of phosphorus content without a particular pattern, with potassium hydroxide bleaching sludge having the highest content of phosphorus (12.57mg/100ml) after 1hr bleaching.

At 90% concentrations of potassium hydroxide and calcium hypochlorite, the result showed that carbon and phosphorus increased with an increase in bleaching time while nitrogen and ash have minimal downward or upward changes. Nitrogen has its highest value (0.29%) after 1hr bleaching and lowest (0.15%) after 3hr bleaching with calcium hypochlorite, which is not far from the value obtained from potassium hydroxide bleaching during the 3hr period. The carbon content of sludge from 90% concentration bleaching increased with an increase in bleaching time with the highest values of 17.73% from calcium hypochlorite and 11.77% from potassium hydroxide after 2hr bleaching. Ash content increased or decreased minimally with the two chemicals during the 3hr bleaching period. The result showed an increase of phosphorus content in the sludges obtained from potassium hydroxide and calcium hypochlorite bleaching along the 3hr period with the highest values (12.53mg/100ml and 12.6mg/100ml) after 3hr bleaching.

Table 2: Mean and SE of Nitrogen (%) Sludge over the concentration, time, and chemical factors for Bleaching

Concentration	Time	Chemical	Mean + SE
20%[a]	1 Hour	Ca(CIO)2	1.83 + 0.11
		KOH	
	2 Hours	Ca(CIO)2	2.67 + 0.11
		KOH	
	3 Hours	Ca(CIO)2	8.67 + 0.11
		KOH	
60%[b]	1 Hour	Ca(CIO)2	
		KOH	0.13 + 0.11
	2 Hours	Ca(CIO)2	5.1 + 0.11[a]
		KOH	0.12 + 0.11[b]
	3 Hours	Ca(CIO)2	2.83 + 0.11[a]
		KOH	0.13 + 0.11[b]
90%[c]	1 Hour	Ca(CIO)2	0.29 + 0.11[a]
		KOH	0.17 + 0.11[a]
	2 Hours	Ca(CIO)2	0.16 + 0.11[a]
		KOH	0.17 + 0.11[a]
	3 Hours	Ca(CIO)2	0.15 + 0.11[a]
		KOH	0.12 + 0.11[a]

ANOVA Table

Source	Sum of Squares	df	Mean Square	F	Sig.
conc	63.165	2	31.583	952.716	.000
Time	19.969	2	9.985	301.196	.000
Chem	22.642	1	22.642	683.028	.000
conc * Time	68.519	4	17.130	516.734	.000
conc * chem	22.042	1	22.042	664.907	.000
Time * chem	1.904	2	.952	28.721	.000
conc * Time * chem	2.007	1	2.007	60.537	.000
Error	.928	28	.033		
Corrected Total	251.677	41			

b. R Squared = .996 (Adjusted R Squared = .995)

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

Table 3: Mean and SE of Carbon (%) Sludge over the concentration, time, and chemical factors for Bleaching

Concentration	Time	Chemical	Mean + SE
20%[a]	1 Hour	Ca(CIO)2	0.11 + 0.09
		KOH	
	2 Hours	Ca(CIO)2	0.07 + 0.09
		KOH	
	3 Hours	Ca(CIO)2	0.07 + 0.09
		KOH	
60%[b]	1 Hour	Ca(CIO)2	0.02 + 0.09[a]
		KOH	23.2 + 0.09[b]
	2 Hours	Ca(CIO)2	0.02 + 0.09[a]
		KOH	17.77 + 0.09[b]
	3 Hours	Ca(CIO)2	0.03 + 0.09[a]
		KOH	10.47 + 0.09[b]
90%[c]	1 Hour	Ca(CIO)2	4.7 + 0.09[a]
		KOH	9.6 + 0.09[b]
	2 Hours	Ca(CIO)2	17.73 + 0.09[a]
		KOH	11.77 + 0.09[b]
	3 Hours	Ca(CIO)2	12.57 + 0.09[a]
		KOH	5.2 + 0.09[b]

ANOVA Table

Source	Sum of Squares	df	Mean Square	F	Sig.
Conc	224.225	2	112.112	4237.765	.000
Time	115.079	2	57.539	2174.946	.000
Chem	460.889	1	460.889	17421.268	.000
conc * Time	220.520	4	55.130	2083.874	.000
conc * chem	894.110	1	894.110	33796.670	.000
Time * chem	241.776	2	120.888	4569.479	.000
conc * Time * chem	16.150	2	8.075	305.233	.000
Error	.794	30	.026		
Corrected Total	2579.180	44			

R Squared = 1.000 (Adjusted R Squared = 1.000)

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

Table 4: Mean and SE of Ash (%) Sludge over the concentration, time, and chemical factors for Bleaching

Concentration	Time	Chemical	Mean + SE
20%a	1 Hour	Ca(CIO)2	0.98 + 0.02
		KOH	
	2 Hours	Ca(CIO)2	0.66 + 0.02
		KOH	
	3 Hours	Ca(CIO)2	0.87 + 0.02
		KOH	
60%b	1 Hour	Ca(CIO)2	0.97 + 0.02a
		KOH	0.97 + 0.02a
	2 Hours	Ca(CIO)2	1.26 + 0.02a
		KOH	0.99 + 0.02b
	3 Hours	Ca(CIO)2	1.29 + 0.02a
		KOH	0.36 + 0.02b
90%b	1 Hour	Ca(CIO)2	1.05 + 0.02a
		KOH	0.86 + 0.02b
	2 Hours	Ca(CIO)2	2.78 + 0.02a
		KOH	0.24 + 0.02b
	3 Hours	Ca(CIO)2	0.27 + 0.02a
		KOH	0.77 + 0.02b

ANOVA Table

Source	Sum of Squares	df	Mean Square	F	Sig.
Conc	1.137	2	.569	520.130	.000
Time	.811	2	.406	371.067	.000
Chem	2.936	1	2.936	2684.919	.000
Conc * Time	3.326	4	.831	760.500	.000
Conc * chem	.267	1	.267	244.157	.000
Time * chem	3.175	2	1.588	1452.091	.000
Conc * Time * chem	5.180	2	2.590	2369.027	.000
Error	.033	30	.001		
Corrected Total	15.134	44			

b. R Squared = .998 (Adjusted R Squared = .997)

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

Table 5: Mean and SE of Phosphorus (mg/100ml) Sludge over the concentration, time, and chemical factors for Bleaching

Concentration	Time	Chemical	Mean + SE
20%[a]	1 Hour	Ca(CIO)2	0.97 + 0.07
		KOH	
	2 Hours	Ca(CIO)2	1.1 + 0.07
		KOH	
	3 Hours	Ca(CIO)2	10.2 + 0.07
		KOH	
60%[b]	1 Hour	Ca(CIO)2	2.73 + 0.07[a]
		KOH	12.57 + 0.07[b]
	2 Hours	Ca(CIO)2	11.23 + 0.07[a]
		KOH	2.03 + 0.07[b]
	3 Hours	Ca(CIO)2	3.37 + 0.07[a]
		KOH	10.53 + 0.07[b]
90%[c]	1 Hour	Ca(CIO)2	9.3 + 0.07[a]
		KOH	1.77 + 0.07[b]
	2 Hours	Ca(CIO)2	9.43 + 0.07[a]
		KOH	8.5 + 0.07[b]
	3 Hours	Ca(CIO)2	12.6 + 0.07[a]
		KOH	12.53 + 0.07[a]

ANOVA Table

Source	Sum of Squares	df	Mean Square	F	Sig.
Conc	131.097	2	65.549	4402.525	.000
Time	213.021	2	106.511	7153.691	.000
chem	.134	1	.134	9.030	.005
Conc * Time	224.659	4	56.165	3772.254	.000
Conc * chem	66.694	1	66.694	4479.478	.000
Time * chem	118.654	2	59.327	3984.646	.000
Conc * Time * chem	250.001	2	125.000	8395.541	.000
Error	.447	30	.015		
Corrected Total	902.690	44			

b. R Squared = 1.000 (Adjusted R Squared = .999)

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

CONCLUSION

Looking at the results of the sludge analysis, 20% concentration of the chemicals may not be strong enough while 60% concentration was able to release all the elements analyzed for a while 90% concentrations have a higher content of some of the analyzed minerals. With respect to the bleached samples, 20% concentrations of the chemicals were not able to bleach the pulp properly while the sample from 60% concentration was properly bleached. Pulp from 90% concentration bleaching was over-bleached. This made 60% concentration for 2hr bleaching to look more acceptable. Looking at the chemicals, sludge can be used as compost to replenish soil nutrients and increase soil fertility and forest productivity as a better alternative to landfilling. Since potassium plays an important role in increasing the fertility of the soil, bleaching with 60% potassium hydroxide for 2hrs will be a better option for both bleached pulp and the sludge contents. There is also a need to look into other characteristics of this sludge for a well-informed conclusion.

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There is no conflict of interest

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