



Characteristics of Effluent from Formic Acid and Sodium Hydroxide Pulping of Kenaf Stem

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Authors' contributions

This work was carried out in collaboration between both authors. Both authors read and approved the final manuscript.

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ABSTRACT

The pulp and paper industry is considered as one of the major potential sources of pollution in the environment and a consumer of wood. Environmental effects have been attributed to chemicals introduced during the manufacturing process. This paper investigated the influence of cooking chemicals, concentration and time on the properties of effluent generated during pulping of agricultural residue. A stem of kenaf which is an agricultural residue was pulped with 20%, 60% and 90% concentrations of formic acid and sodium hydroxide at 1 hour, 2 hours and 3 hours intervals to determine the characteristics of their effluents. The lowest Chemical Oxygen Demand (COD) obtained from formic acid effluent for the 3 hours cooking at 20%, 60% and 90% concentrations was 324mg/l at 60% concentration after cooking for 2 hours while sodium hydroxide effluent has 3050mg/l at 20% concentration after 1hour cooking as its lowest. Formic acid effluent showed lowest Biological Oxygen Demand (BOD) of 10.63mg/l at 60% concentration after cooking for 2 hours while sodium hydroxide has 13.75mg/l at 90% after 1 hour cooking. The value of Total Solid (TS) from formic acid effluent was lowest (16890mg/l) at 60% concentration after cooking for 2 hours while sodium hydroxide lowest value (15524mg/l) was recorded at 20% after 3 hours cooking. Sodium hydroxide effluent has lowest Total Suspended Solid (TSS) of 3165mg/l while formic acid has 2245mg/l both at 90% concentrations after 2 and 3 hours cooking.

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1. INTRODUCTION

There is a high demand for pulp and paper products globally due to an increase in population, industrialization, and urbanization [1]. This demand has led to the massive cutting down of trees as this sector is heavily reliant on fibrous wood for the production of paper and pulp which are primarily sourced from the forests. There is a concern that in the coming years the demand for paper will increase tremendously and ultimately surpass the resource regeneration [2]. Agricultural residues therefore offer an alternative source of raw material in the production of pulp and paper whilst supporting the concept of a sustainable manufacturing through the use of abundant agricultural residue resources [3]. Most of the agricultural residues are obtained from the three major sources namely; the agricultural by-products, industrial crops, and other naturally growing plants. In the past, these nonwood fibres and other agricultural waste were either burnt or naturally converted into compost [4]. Agricultural residue utilization will help in the maintenance and regeneration of the forests, as healthy forests are considered as one of the solutions to the emerging climate and biodiversity crisis [5]. The biomass is left to rot or openly burned in the fields, especially in developing countries like Nigeria that do not have strong regulatory instruments to control such pollutive practices [6]. As a common practice, direct combustion of agricultural residue results in air pollution thereby posing risk to human and ecological health. It also leads to soil crusting and reduced biological activity causing low yield. Its utilization in this sector will help in the reduction of carbon emissions and other greenhouse gases, species loss, perturbation of the water cycle, and soil erosion [7].

Looking for new raw materials suitable for production of pulp and paper industry with minimal cost and application of environmentally friendly cooking methods is core of attention for most of the researchers of this field [8,9,10,11]. Pulping is the process of converting plant fiber into smaller chips. The pulping process reduces the fibrous mass of the non-wood and breaks down the interlinking bonds of the biomass hence making them more suitable for the papermaking process. The most widely used pulping methods are mechanical pulping, chemical pulping, semi-chemical pulping or bio pulping [12,13,14,15]. The chemical pulping

method involves the addition of chemicals that eventually separates the fibres through degradation of the lignin and hemicelluloses into small water-soluble molecules. The most commonly used chemical methods are the Kraft pulping, sulphite pulping, soda pulping, and organosolv pulping processes [16]. The soda pulping process involves the cooking of the biomass with either sodium carbonate or both sodium carbonate and sodium hydroxide [17]. Organosolv pulping involves the hydrolysis and removal of the lignin through the use of organic solvent. At the end of the process, the dissolved lignin together with other dissolved components is recovered by the distillation of the solvent [18]. Conventionally, agricultural residues are pulped by alkaline processes. But most of the crops residues are high in ash content. In alkali processes, pulp yield and properties are good, but the main drawback is the dissolution of silica in the black liquor, which causes problem during recovery of the cooking reagents. A pulp mill cannot be environmentally friendly without chemical recovery system. Another factor for consideration is that the crops residues are bulky in nature, making transportation of these types of raw materials difficult. Therefore, organic solvent-based delignification has been exhaustively studied in recent years as an alternative to the traditional processes of chemical pulp production because of strict regulations on environmental discharges [19].

The pulping process affects the strength, appearance and intended use characteristics of the resultant paper product. Pulping processes are the major source of environmental impacts in the pulp and paper industry, each pulping process has its own set of process inputs, outputs and resultant environmental impacts [20]. Waste water quality is commonly judged on the basis of such aggregate characteristics as biochemical oxygen demand (BOD), chemical oxygen demand (COD), total suspended solids (TSS), total solids (TS), turbidity, pH, color etc. Biological oxygen demand is the amount of oxygen required for microbial degradation of organic matter while chemical oxygen demand is the amount of oxygen required to breakdown both organic and inorganic matter [21]. Total suspended solid (TSS) represents the solid particles mixed in water or effluent. Total dissolved solids (TDS) are measured as the mass of residue remaining when a measured volume of filtered water is evaporated. Total

solids (TS) are the amount of solid present in dissolved and suspended form [22]. In the recent past due to the advancement of analytical facilities, some researchers have detected a complex mixture of organic and inorganic compounds as residual recalcitrant pollutants present in pulp paper mill effluent after secondary treatment [23,24]. These compounds not only contribute the toxicity and increase COD, but some are even also carcinogenic, mutagenic and endocrine-disrupting properties (EDCs) along with metabolic constituents which disturb the food chain and adversely affect human health also [25,26,27].

They are parameters that indicate the pollution load of effluent. The effluent physicochemical characteristics vary because of the different pulping, bleaching processes, and additives. But from every literature review, we observed that many physicochemical parameters exceed the permissible limit [28].

In Nigeria with little or no wood reserves, there is need to develop environmental friendly processes to use straw and different crops residues for papermaking. Sustainable process will enable the organic acid to be recovered by distillation and reused in the process while the wastewater can be used for secondary purposes such as agricultural needs for irrigating the crops to address the declining freshwater sources for the expanding population(With the above explanation, it must not have a table). This paper investigated the influence of cooking chemicals, concentration and time on the properties of the generated effluents(It has been corrected above)

2. MATERIALS AND METHODS

Kenaf stem was 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%. Five grams of kenaf stem was taken in 400ml of pulping mixture in 1000 ml flask at atmospheric pressure and pulped at 20, 60 and 90% concentrations of formic acid and sodium hydroxide, cooking time was varied from 1, 2 h and 3 h at 95°C as shown in Figure.1. At the end of each period, the sample was filtered with a fine mesh sieve of size 0.027 mm to get the effluent used in the analyses. The tests were carried out in triplicate and each value is an average of three samples(Author's lab work).

The effluent was analysed using the Standard Method for Examination of Water and Wastewater [29].

THREE-WAY ANOVA was used for analysis of the data.

The parameters determined were COD BOD, TSS, and TS.

Calculation done to determine the BOD₅;

$$\frac{(DO_1 - DO_5)}{p} = BOD_5 \text{ mg/l}$$

DO₁ = initial dissolved oxygen

DO₅ = final dissolved oxygen (dissolved oxygen after five days)

P = fraction of sample (volume of sample/volume of sample bottle)

Calculation done to determine the COD;

$$\frac{V_t \times N \times 8000}{V_s} = COD \text{ mg/l}$$

Where V_t is the volume of titrant, N is normality of the standard Ferrous Ammonium Sulphate and V_s is volume of sample used.

$$\text{Calculation of TSS (mg/l)} = \frac{A-B}{V_s} * 1000^2 \text{ mg/l}$$

Where A = weight of filter + dried residue

B = weight of filter

V_s = volume of sample used

$$\text{Calculation of TS (mg/l)} = \frac{A-B}{V_s} \times 1000^2$$

Where A is weight of porcelain dish and residue after oven dried,

B is weight of empty porcelain dish and Vs is volume of sample used.

3. RESULTS AND DISCUSSION

Table 1 reported the values of COD, BOD, TSS and TS from effluent obtained from pulping kenaf stem with 20%, 60%, and 90%(fixed by the author) concentrations of sodium hydroxide and formic acid at 3 hours interval. With 20% concentration of sodium hydroxide, the COD value in the effluent was at lowest (3050mg/L) after 1 hour and highest (7300mg/L) after 2 hours. Formic acid at the same concentration has lowest (1252mg/L) COD after 2 hours cooking and highest (2000mg/L) after 3 hours. The values of BOD reported with 20% concentration of sodium hydroxide decreased with increase in cooking time having highest

(80.63mg/l) after 1 hour and lowest (45.63mg/l) after 3 hours. This showed that less oxygen is used which may be due to reduced degradation. In formic acid cooking, the lowest (21.88mg/l) BOD was obtained after 2 hours having highest (34.38mg/l) after the first hour. TS in effluent from 20% sodium hydroxide cooking decreased with increase in cooking time having highest (99240 mg/l) after 1 hour and lowest (15524 mg/l) after 3 hours which agrees with [30]. In formic acid pulping effluent, TS increased with cooking time while [31] reported a decrease with cooking time which may be due to difference in raw materials.

These findings showed increase in TSS with time in sodium hydroxide effluent (4637.5 to 9660 mg/L) and also in formic acid which did not follow a particular pattern at 20% concentrations.

At 60% concentrations, COD values in sodium hydroxide effluent increased (6250 mg/l – 18750mg/l) with increase in cooking time (1hr – 3hrs) which indicated that more oxygen was used for more degradation. Formic acid effluent showed highest COD value (1684mg/l) after 1hr and lowest (324mg/l) after 2 hrs. BOD in sodium hydroxide effluent has its maximum value (37.5mg/l after 1 hour and minimum (17.5mg/l)

after 2 hours and BOD in formic acid effluent followed the same pattern having its maximum value (31.25mg/l) after 1hour and minimum (10.63 mg/l) after 2 hours. The TSS of both effluents (sodium hydroxide and formic acid) decreased with increase in cooking time showing more degradation of particles. The result showed increment in TS from sodium hydroxide effluent when the cooking time increased but effluent from formic acid showed little variation between 1 hour and 3 hours.

With 90% of the chemicals, the COD values in both effluents increased with increase in cooking time having lowest after 1 hour and highest after 3 hours. This may be due to increase in oxidative activities and oxygen usage. Sodium hydroxide effluent showed increase in BOD values (13.75 mg/l – 46.88mg/l) which showed that more oxygen was used in microbial degradation while formic acid recorded a reduction in BOD. TS reduced (127,945mg/l – 72,860mg/l) in sodium hydroxide effluent but formic acid effluent did not show appreciable reduction. Effluent from formic acid pulping showed reduction in TSS value (5272.5mg/l – 2245mg/l) with increase in cooking time while reduction in sodium hydroxide effluent did not follow a particular pattern.



Fig. 1. Kenaf stem Shredded stem Pulping effluent

Table 1. Parameters over the concentration, time and chemical factors for pulping

Concentration	Time	Chemical	COD (mg/l)	BOD (mg/l)	TS (mg/l)	TSS (mg/l)
			Mean + SE	Mean + SE	Mean + SE	Mean + SE
20% ^a	1 Hour	NaOH	3050 + 2561.78 ^a	80.63 + 9.48	99240 + 16781.29	4637.5 + 2532.81
		Formic Acid	1332 + 2561.78 ^a	34.38 + 9.48	59855 + 16781.29	12167.5 + 2532.81
	2 Hours	NaOH	7300 + 2561.78 ^a	51.88 + 9.48	45680 + 16781.29	6052.5 + 2532.81
		Formic Acid	1252 + 2561.78 ^a	21.88 + 9.48	60942.5 + 16781.29	18177.5 + 2532.81
	3 Hours	NaOH	5900 + 2561.78 ^a	45.63 + 9.48	15524 + 16781.29	9660 + 2532.81
		Formic Acid	2000 + 2561.78 ^a	30 + 9.48	70167.5 + 16781.29	17192.5 + 2532.81
60% ^a	1 Hour	NaOH	6250 + 2561.78 ^a	37.5 + 9.48	132232.5 + 16781.29	9902.5 + 2532.81
		Formic Acid	1684 + 2561.78 ^a	31.25 + 9.48	51405 + 16781.29	11185 + 2532.81

2 Hours	NaOH	8000 + 2561.78 ^a	17.5 + 9.48	293340 + 16781.29	7385 + 2532.81
	Formic Acid	324 + 2561.78 ^a	10.63 + 9.48	16890 + 16781.29	5932.5 + 2532.81
3 Hours	NaOH	18750 + 2561.78 ^a	32.5 + 9.48	216510 + 16781.29	8147.5 + 2532.81
	Formic Acid	748 + 2561.78 ^a	26.88 + 9.48	50297.5 + 16781.29	7767.5 + 2532.81
90% ^b	1 Hour	NaOH	5650 + 2561.78 ^a	13.75 + 9.48	127945 + 16781.29
		Formic Acid	400 + 2561.78 ^a	51.25 + 9.48	59927.5 + 16781.29
	2 Hours	NaOH	7000 + 2561.78 ^a	14.38 + 9.48	102680 + 16781.29
		Formic Acid	710 + 2561.78	45 + 9.48	88145 + 16781.29
	3 Hours	NaOH	14350 + 2561.78 ^a	46.88 + 9.48	72860 + 16781.29
		Formic Acid	922 + 2561.78	48.13 + 9.48	57047.5 + 16781.29

Table 2. COD (mg/l) over the concentrations, time and chemicals for pulping

Concentration	Time	Chemical	Mean + SE
20% ^a	1 Hour	NaOH	3050 + 2561.78 ^a
		Formic Acid	1332 + 2561.78 ^b
	2 Hours	NaOH	7300 + 2561.78 ^a
		Formic Acid	1252 + 2561.78 ^b
	3 Hours	NaOH	5900 + 2561.78 ^a
		Formic Acid	2000 + 2561.78 ^b
60% ^a	1 Hour	NaOH	6250 + 2561.78 ^a
		Formic Acid	1684 + 2561.78 ^b
	2 Hours	NaOH	8000 + 2561.78 ^a
		Formic Acid	324 + 2561.78 ^b
	3 Hours	NaOH	18750 + 2561.78 ^a
		Formic Acid	748 + 2561.78 ^b
90% ^a	1 Hour	NaOH	5650 + 2561.78 ^a
		Formic Acid	400 + 2561.78 ^b
	2 Hours	NaOH	7000 + 2561.78 ^a
		Formic Acid	710 + 2561.78 ^b
	3 Hours	NaOH	14350 + 2561.78 ^a
		Formic Acid	922 + 2561.78 ^b

ANOVA Table

Source	Sum of Squares	df	Mean Square	F	Sig.
Chemical	496962987.111	1	496962987.111	37.86 3	.000
Time	106267096.889	2	53133548.444	4.048	.035
Conc.	37231718.222	2	18615859.111	1.418	.268
Chemical * Time	96971096.889	2	48485548.444	3.694	.045
Chemical * Conc.	61102384.889	2	30551192.444	2.328	.126
Time * Conc.	38370079.111	4	9592519.778	.731	.583
Chemical * Time * Concentration	50982479.111	4	12745619.778	.971	.448
Error	236257144.000	18	13125396.889		
Corrected Total	1124144986.222	35			

R Squared = .790 (Adjusted R Squared = .591)

The ANOVA table reveal that of the 3 factors, only Chemicals and time were significant ($p < 0.05$) while concentration is not ($p > 0.05$). Also, the interaction between chemical and time was also significant ($p < 0.05$)

Table 3. BOD (mg/l) over the concentrations, time and chemicals for pulping

Concentration	Time	Chemical	Mean + SE
20% ^a	1 Hour	NaOH	80.63 + 9.48 ^a
		Formic Acid	34.38 + 9.48 ^a
	2 Hours	NaOH	51.88 + 9.48 ^a
		Formic Acid	21.88 + 9.48 ^a
	3 Hours	NaOH	45.63 + 9.48 ^a
		Formic Acid	30 + 9.48 ^a
60% ^b	1 Hour	NaOH	37.5 + 9.48 ^a
		Formic Acid	31.25 + 9.48 ^a
	2 Hours	NaOH	17.5 + 9.48 ^a
		Formic Acid	10.63 + 9.48 ^a
	3 Hours	NaOH	32.5 + 9.48 ^a
		Formic Acid	26.88 + 9.48 ^a
90% ^{ab}	1 Hour	NaOH	13.75 + 9.48 ^a
		Formic Acid	51.25 + 9.48 ^a
	2 Hours	NaOH	14.38 + 9.48 ^a
		Formic Acid	45 + 9.48 ^a
	3 Hours	NaOH	46.88 + 9.48 ^a
		Formic Acid	48.13 + 9.48 ^a

ANOVA Table

Source	Sum of Squares	df	Mean Square	F	Sig.
Chemical	189.063	1	189.063	1.052	.319
Time	1414.931	2	707.465	3.935	.038
Conc	1966.753	2	983.377	5.470	.014
Chemical * Time	32.292	2	16.146	.090	.915
Chemical * Conc	4346.094	2	2173.047	12.088	.000
Time * Conc	1308.767	4	327.192	1.820	.169
Chemical * Time * Concentration	1179.427	4	294.857	1.640	.208
Error	3235.938	18	179.774		
Corrected Total	13673.264	35			

b. R Squared = .763 (Adjusted R Squared = .540)

Table above shows that while only time, concentrations and chemical – concentration interaction are significant (P < 0.05), the rest are not significant (P > 0.05)

Table 4. TS (mg/l) over the concentrations, time and chemicals for pulping

Concentration	Time	Chemical	Mean + SE
20% ^a	1 Hour	NaOH	99240 + 16781.29 ^a
		Formic Acid	59855 + 16781.29 ^b
	2 Hours	NaOH	45680 + 16781.29 ^a
		Formic Acid	60942.5 + 16781.29 ^a
	3 Hours	NaOH	15524 + 16781.29 ^a
		Formic Acid	70167.5 + 16781.29 ^b
60% ^a	1 Hour	NaOH	132232.5 + 16781.29 ^a
		Formic Acid	51405 + 16781.29 ^b
	2 Hours	NaOH	293340 + 16781.29 ^a
		Formic Acid	16890 + 16781.29 ^b
	3 Hours	NaOH	216510 + 16781.29 ^a
		Formic Acid	50297.5 + 16781.29 ^b
90% ^c	1 Hour	NaOH	127945 + 16781.29 ^a
		Formic Acid	59927.5 + 16781.29 ^b
	2 Hours	NaOH	102680 + 16781.29 ^a
		Formic Acid	88145 + 16781.29 ^b
	3 Hours	NaOH	72860 + 16781.29 ^a
		Formic Acid	57047.5 + 16781.29 ^a

ANOVA Table

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Chemical	38852877728.444	1	38852877728.444	68.983	.000
Time	2661787677.722	2	1330893838.861	2.363	.123
Conc	28416502896.889	2	14208251448.444	25.227	.000
Chemical * Time	3706947754.389	2	1853473877.194	3.291	.061
Chemical * Conc	56030117193.556	2	28015058596.778	49.741	.000
Time * Conc	10834068182.111	4	2708517045.528	4.809	.008
Chemical * Time * Concentration	21852055647.111	4	5463013911.778	9.700	.000
Error	10138015159.000	18	563223064.389		
Total	464340464986.000	36			
Corrected Total	172492372239.223	35			

R Squared = .941 (Adjusted R Squared = .886)

The Anova table reveals that all the factors except time are significant ($P < 0.05$) and all interactions are significant ($P < 0.05$) except chemical – time

Table 5. TSS (mg/l) over the concentrations, time and chemicals for pulping

Concentration	Time	Chemical	Mean + SE
20% ^a	1 Hour	NaOH	4637.5 + 2532.81
		Formic Acid	12167.5 + 2532.81
	2 Hours	NaOH	6052.5 + 2532.81
		Formic Acid	18177.5 + 2532.81
	3 Hours	NaOH	9660 + 2532.81
		Formic Acid	17192.5 + 2532.81
60% ^a	1 Hour	NaOH	9902.5 + 2532.81
		Formic Acid	11185 + 2532.81
	2 Hours	NaOH	7385 + 2532.81
		Formic Acid	5932.5 + 2532.81
	3 Hours	NaOH	8147.5 + 2532.81
		Formic Acid	7767.5 + 2532.81
90% ^b	1 Hour	NaOH	7352.5 + 2532.81
		Formic Acid	5272.5 + 2532.81
	2 Hours	NaOH	3165 + 2532.81
		Formic Acid	3220 + 2532.81
	3 Hours	NaOH	6105 + 2532.81
		Formic Acid	2245 + 2532.81

ANOVA Table

Source	Sum of Squares	df	Mean Square	F	Sig.
Chemical	47851806.250	1	47851806.250	3.730	.069
Time	10594050.000	2	5297025.000	.413	.668
Conc	275361879.167	2	137680939.583	10.731	.001
Chemical * Time	9230316.667	2	4615158.333	.360	.703
Chemical * Conc	210180154.167	2	105090077.083	8.191	.003
Time * Conc	95375783.333	4	23843945.833	1.858	.162
Chemical * Time * Concentration	16320791.667	4	4080197.917	.318	.862
Error	230944337.500	18	12830240.972		
Corrected Total	895859118.750	35			

b. R Squared = .742 (Adjusted R Squared = .499)

The Anova table above shows that only concentration factor and chemical – concentration interaction are significant (P < 0.05)

4. CONCLUSION

The characteristics of effluents from pulping kenaf stem with sodium hydroxide and formic acid with reference to COD, BOD, TSS and TS were studied. From the characteristics of the effluent, it was observed that formic acid pulping has lower values in COD, BOD and TS. Also the formic acid can be recovered by evaporation and condensation for reuse making the process sustainable. The pulp appearance showed that 60% concentration for 2 hours has a better pulp, though; more analysis is needed to ascertain the pulp properties. From the present study, formic acid pulping effluent has lower COD and BOD and the acid can easily be recovered through evaporation, therefore, it is recommended over sodium hydroxide pulping. Countries with little or no wood reserves should develop interest in using straw and crop residues with appropriate process for pulp and paper production. There is need for further research to check other characteristics of the effluents e.g AOX, turbidity, TDS etc.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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