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

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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 1 hour cooking. Formic acid effluent showed lowest Biological Oxygen Demand (BOD) of 10.63mg/l after cooking for 2 hours while sodium hydroxide effluent has 13.75mg/l at 90% concentration 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.
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:

\[
(\text{DO}_1 - \text{DO}_5) = \text{BOD}_5 \text{ mg/l}
\]

\[
P = \text{fraction of sample (volume of sample/volume of sample bottle)}
\]

Calculation done to determine the COD:

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

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.

Calculation of TSS (mg/l) = \(\frac{A - B}{V_s}\) x 1000² mg/l

Where \(A\) = weight of filter + dried residue
\(B\) = weight of filter
\(V_s\) = volume of sample used

Calculation of TS (mg/l) = \(\frac{A - B}{V_s}\) x 1000²

Where \(A\) is weight of porcelain dish and residue after oven dried,
\(B\) is weight of empty porcelain dish and \(V_s\) 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 it 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.

| 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%            | 1 Hour | NaOH     | 3050 + 2561.78* | 80.63 + 9.48 | 99240 + 16781.29 | 4637.5 + 2532.81 |
|                |      | Formic Acid | 1332 + 2561.78* | 34.38 + 9.48 | 59855 + 16781.29 | 12167.5 + 2532.81 |
| 2 Hours        | NaOH | 7300 + 2561.78* | 51.88 + 9.48 | 45680 + 16781.29 | 6052.5 + 2532.81 |
|                | Formic Acid | 1252 + 2561.78* | 21.88 + 9.48 | 60942.5 + 16781.29 | 18177.5 + 2532.81 |
| 3 Hours        | NaOH | 5900 + 2561.78* | 45.63 + 9.48 | 15524 + 16781.29 | 9660 + 2532.81 |
|                | Formic Acid | 2000 + 2561.78* | 30 + 9.48 | 70167.5 + 16781.29 | 17192.5 + 2532.81 |
| 60%            | 1 Hour | NaOH     | 6250 + 2561.78* | 37.5 + 9.48 | 132232.5 + 16781.29 | 9902.5 + 2532.81 |
|                | Formic Acid | 1684 + 2561.78* | 31.25 + 9.48 | 51405 + 16781.29 | 11185 + 2532.81 |
Table 2. COD (mg/l) over the concentrations, time and chemicals for pulping

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

ANOVA Table

| Source                  | Sum of Squares | df  | Mean Square | F         | Sig. |
|-------------------------|----------------|-----|-------------|-----------|------|
| Chemical                | 496962987.111  | 1   | 496962987.111 | 37.86     | .000 |
| Time                    | 106267096.889  | 2   | 53133548.444 | 4.048     | .035 |
| Conc.                   | 3723178.222    | 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 * Conc. | 50982479.111   | 4   | 12745619.778 | .971      | .448 |
| Error                   | 236257144.000  | 18  | 13125396.889 |           |      |
| Corrected Total         | 112414498.222  | 35  |             |           |      |

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%<sup>a</sup> | 1 Hour | NaOH              | 80.63 + 9.48<sup>a</sup> |
|               |        | Formic Acid       | 34.38 + 9.48<sup>a</sup> |
|               | 2 Hours | NaOH              | 51.88 + 9.48<sup>a</sup> |
|               |        | Formic Acid       | 21.88 + 9.48<sup>a</sup> |
|               | 3 Hours | NaOH              | 45.63 + 9.48<sup>a</sup> |
|               |        | Formic Acid       | 30 + 9.48<sup>a</sup>   |
| 60%<sup>b</sup> | 1 Hour | NaOH              | 37.5 + 9.48<sup>a</sup>  |
|               |        | Formic Acid       | 31.25 + 9.48<sup>a</sup> |
|               | 2 Hours | NaOH              | 17.5 + 9.48<sup>a</sup>  |
|               |        | Formic Acid       | 10.63 + 9.48<sup>a</sup> |
|               | 3 Hours | NaOH              | 32.5 + 9.48<sup>a</sup>  |
|               |        | Formic Acid       | 26.88 + 9.48<sup>a</sup> |
| 90%<sup>ab</sup> | 1 Hour | NaOH              | 13.75 + 9.48<sup>a</sup> |
|               |        | Formic Acid       | 51.25 + 9.48<sup>a</sup> |
|               | 2 Hours | NaOH              | 14.38 + 9.48<sup>a</sup> |
|               |        | Formic Acid       | 45 + 9.48<sup>a</sup>    |
|               | 3 Hours | NaOH              | 46.88 + 9.48<sup>a</sup> |
|               |        | Formic Acid       | 48.13 + 9.48<sup>a</sup> |

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 * Conc  | 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 ± 16781.29a |
|               |        | Formic Acid| 59855 ± 16781.29b |
|               | 2 Hours | NaOH       | 45680 ± 16781.29a |
|               |        | Formic Acid| 60942.5 ± 16781.29a |
|               | 3 Hours | NaOH       | 15524 ± 16781.29a |
|               |        | Formic Acid| 70167.5 ± 16781.29b |
| 60%a          | 1 Hour | NaOH       | 132232.5 ± 16781.29a |
|               |        | Formic Acid| 51405 ± 16781.29b |
|               | 2 Hours | NaOH       | 293340 ± 16781.29a |
|               |        | Formic Acid| 16890 ± 16781.29b |
|               | 3 Hours | NaOH       | 216510 ± 16781.29a |
|               |        | Formic Acid| 50297.5 ± 16781.29b |
| 90%c          | 1 Hour | NaOH       | 127945 ± 16781.29a |
|               |        | Formic Acid| 59927.5 ± 16781.29b |
|               | 2 Hours | NaOH       | 102680 ± 16781.29a |
|               |        | Formic Acid| 88145 ± 16781.29b |
|               | 3 Hours | NaOH       | 72860 ± 16781.29a |
|               |        | Formic Acid| 57047.5 ± 16781.29b |
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%<sup>a</sup> |         |                   |                    |
| 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%<sup>b</sup> |        |                   |                    |
| 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%<sup>b</sup> |        |                   |                    |
| 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   | 10509077.083  | 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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