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Water-pH phytoremediation: An entrance to exploiting the potential of plant biomass ash in water defluoridation

Hezron Timothy Mwakabona[a]*, Karoli N. Njau[b] and Bart Van der Bruggen[c]

[a] *Department of Applied Sciences, Mbeya University of Science and Technology, Mbeya, Tanzania;* *hezron.mwakabona@must.ac.tz*

[b] *Department of Waterand Environmental Science and Engineering, Nelson Mandela African Institution of Science and Technology, Arusha, Tanzania.;* *karoli.njau@nm-aist.ac.tz*

[c] *Department of Chemical Engineering, KU Leuven, Leuven, Belgium;* *bart.vanderbruggen@kuleuven.be*

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ABSTRACT

Field application of low-cost biomass ash in water defluoridation is hindered by media-induced contamination in the treated water including pH elevation. This study explored the method for adjusting pH of water using Cyperus plants. Findings reveal that used ash material is calcium rich with concentration of 537.9 gKg-1. The ash water extract was found to be rich in K with concentration of 642.6 gKg-1. The ash material used was found to have faster and sustained fluoride removal, attaining 76% removal efficiency in 30 minutes with slow increase throughout the experimental time of 24 hours. The pH of the ash-defluoridated water was found to increase from 8.3 to an average of 12.2. Live Cyperus plants showed ability to lower the pH of the treated water from 12.2 to 7.3 and elevate the pH of acid treated water from 3.5 to 6.8. This implies that these plants exude some form of buffer compounds with a constant pH of around 7. This was confirmed by concentration of hydrogencarbonates was found to increase while that of the carbonates decreased in the Cyperus-treated water. This could further imply that the Cyperus-assisted pH adjustment is effected by a buffer compound released by these plants.

Keywords: Defluoridation*; Cyperus spp* ; Biomass ash.

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1. Introduction

Plant biomass ash consists of (hydr)oxides, carbonates and hydrogencarbonates of such elements as, Ca, Si, K, P, Al, Mg, S, Fe and Na [1-6] and residues of partially burnt biomass. While some of these chemical components are partially soluble, others are highly soluble in aqueous media. As a result, when biomass ash is used in water defluoridation [7-9], there is great chance of causing media induced contamination, namely, leaching their water-soluble components and elevating the pH of the treated water, necessitating post-treatment remediation.

Considering the cheap availability of biomass ash as fireplace waste and its great potential in water defluoridation [7,8], efforts to develop methods to contain post-treatment contamination are worthwhile. Extracting the water soluble contaminants from ash material with the aim of reducing water soluble components may compromise their defluoridation properties, thence, rendering them inefficient [4,5,10], Alternatively, the ash-induced elevation of pH can conventionally be addressed by post-treatment neutralisation by addition of acids to the treated water. This, however, will require predetermination of the levels of pH contamination in the treated water, to establish the required concentrations of acids, every time neutralisation is required. Moreover, addition of mineral acids into the treated water may result into additional cost. This, therefore, implies that, wide application of this material for water treatment would require developing a low-cost method to address the pH elevation. This study, therefore, investigated the plant-assisted pH remediation method for adjusting pH in the ash-defluoridated water. The design in the column experiments is made to mimic the constructed wetland, where plants are used in adjusting the pH of the treated water. While the constructed wetland is a well-studied technology for treatment of wastewaters from various sources [11-13], its application for fluoride treatment is however, scantly studied with a few studies pointing to a combination of plants and adsorbents for enhancing fluoride adsorption on the substrate (adsorbent) [14,15].

Studies exploring the capabilities of plants to adjust water pH in the constructed wetland are scanty. Therefore, in this study the capabilities of Cyperus plants in adjusting the pH of the ash treated water are presented.

2. Materials and Methods

2.1. Materials and material preparation

The ash used in this study was collected from fireplaces in kitchens of selected households in Morogoro, Tanzania. It was then mixed and sieved through 0.1 mm sieve to remove unburnt and other dirty particles and characterised to obtain its chemical composition as further explained in the next section. The plant used for constructed wetland was *Cyperus esculentus* locally found in swampy areas and riverbanks. This plant was selected because of its ability to survive in water logging conditions. The plants were immediately washed in fluoride free tap water to remove soil particles from both the roots and leaves. Washed plants were then inserted in an alkaline solution prepared by mixing the prepared ash and fluoride free tap water to acclimatise the experimental pH conditions. Natural water with fluoride concentration of around 20.8 mgL^{-1} (measured using ion selective electrode (HI 98402 supplied by Hanna Instruments Company)) was obtained from a natural spring in the Usa river area in Arusha, Tanzania.

2.2. Material characterization

The elemental analysis of the raw ash used and ash extract (evaporated to dryness) was done using X-ray Fluorescence spectrometer (S8 Tiger, Bruker AXS GmbH, Germany) to establish the chemical composition of the ash and its water extract. The levels of Ca and K in the water before and after treatment with ash and plants (in the batch and continuous flow experiments) were determined by Atomic Absorption Spectrometer (919 SOLAAR, UNICAM, UK) and Digital Flame Analyzer (2655-00 Cole-Parmer Instrument Company, Chicago, IL), respectively. The carbonates and hydrogencarbonates of the untreated and treated water in the batch experiments were determined by a standard titrimetric method and the pH was estimated using a pH tester pHep (HI 98128, Hanna Instruments, USA).

In the batch experiments, the leaching properties of the ash used in aqueous media was established as follows; 3 kg of the sieved ash was added into 6 L distilled water in the 10 L plastic bucket, stirred manually by stirring rod for 30 minutes and left to settle for another 30 minutes. The supernatant solution was siphoned into a 1 L glass beaker and boiled to obtain the solid residues of the ash extract for establishing the solubility of different chemical components of the ash. In the continuous flow, 1 kg of ash was packed in a 5 L capacity column and distilled water was allowed to flow in the downwards flow through the ash and the flow control valve by gravity at the rate of 0.18 Lh^{-1} for a total of 24 hours. The concentration of the most soluble components (as established in the batch experiment) was monitored at regular intervals (as explained above) to establish the effect of time on the leached concentration in treated water. This was expected to inform the chemical composition of the treated water in the continuous flow design. The most soluble component was selected to be a good marker for contaminant levels and thus a representative of the levels of the media-introduced contaminants in the treated water.

2.3. Determination of fluoride removal efficiency of biomass ash used in batch experiments

10 g of sieved ash was added in 250 ml of the 20.8 mgL^{-1} natural fluoride water in a 1 L plastic bottle and allowed to equilibrate for a total of 24 hours. During this time the mixture was manually shaken vigorously for the first five minutes and then left to settle for the rest of the time. The residual fluoride concentration was determined after 0.5, 1, 4, 8, 12 and 24 hours. Different selected physico-chemical parameters (turbidity, total dissolved solids (TDS) and solution pH of the water before and after treatment were established to understand the influence of ash on the physical and chemical properties of the ash used. Then, the effect of variation of water pH and initial fluoride concentration was determined by using the fluoride water with pH of 2, 4, 6, 8, and 10 and initial fluoride concentration of 5, 10, 15, and 20 mgL^{-1} respectively for a total of 2 hours. The pH was adjusted by using 0.1 M HCl and NaOH solution and the solutions with lower fluoride concentrations were obtained by standard dilution of the natural 20.8 mgL^{-1} fluoride water with distilled water. All experiments were duplicated and averages reported.

2.4. The Cyperus-assisted pH adjustment in the batch experiments

Exactly 1 L of the ash-treated water from the batch reactors was transferred into two separate 1.5 L plastic vessels where, (350 g) approximately 35 live *Cyperus esculentus* plants were immersed in an upright position with half of the plant height in water. To further understand the pH neutralisation mechanism, parallel experiments were conducted using equal volume of water with low pH of 3.5 (adjusted using 0.1 M HCl). All the vessels were left undisturbed throughout the experiment. About 10 ml of the treated water was drawn from all vessels with the aid of the 10 ml plastic syringe after 0.5, 1, 2, 3, 4 and 5 hours and their pH estimated using a portable pH tester. The effect of number of plants on the neutralization of the ash-water was determined by varying the number of plants from 7, 14, 21, 28, 35 to 42 plants in the ash-treated water for a total of 2 hours and pH of the water determined by the pH meter.

2.5 Column experiments

To determine the performance of the continuous flow system, two columns with diameter of 9 cm and height of 40 cm were packed with coarse sand occupying 6 cm thick section in the lower part of the column, followed by ash occupying 7 cm thick section in the middle and then fine sand occupying 10 cm thick section in the uppermost part anchoring the *Cyperus* plants as shown in Fig. 1. The column was set such that water driven by gravity flowed vertically upwards through coarse sand, ash, fine sand and plants in that order with influent water flowrate maintained at 0.18 Lh^{-1} throughout the experiment. The lower coarse sand and the upper fine sand zones were meant to facilitate water flow and settling of the ash material and potentially formed precipitate of CaF_2 and MgF_2. The actual fluoride removal and pH adjustment were meant to take place in the ash zone and plant zone, respectively. Another set of two columns packed with sand and plants without ash were used as control. The levels of fluoride, potassium and pH in the effluent water were monitored for 14 days with measurements taken every after 24 hours.

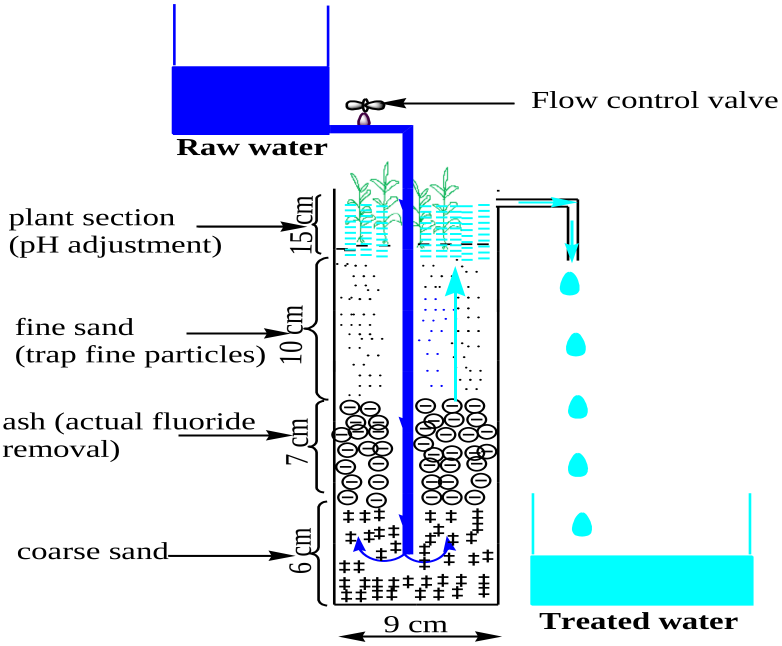

Fig 1. A vertical flow design of the constructed wetland for fluoride and pH adjustment.

3. Results and Discussion

3.1. Characterisation of the ash and ash extract

XRF results of the raw ash used revealed that it was rich in Ca, O, Mg, K and Si with concentrations of 537.9, 301.9, 39.1, and 12.8 gKg^{-1}, respectively, as shown in Fig. 2a. In comparison with other reported wood ashes [3,16], this ash is evidently very rich in oxides of calcium. Its Ca concentration is nearly two times higher than the average concentration of Ca in most reported wood ashes [2,3,16]. Although calcium rich wood ashes have been widely reported for soil fertilisation and liming, their abundance in oxides of Ca and Mg is an advantage in its application as water defluoridation material [9,15]. The chemical composition of water extract revealed that K, S, Si, Na, Cl, Ca, Al, and Mg are highly soluble in water, thus, likely to be present in the treated water when ash is used for water defluoridation (Fig. 2b). It further indicates that K, having highest solubility, can be used as a marker of post-treatment contamination levels in ash treated water. The presence of oxygen in the raw ash and loss on ignition (LOI ranging from 38.81 to 40.12 %) values suggests that the metals in this material occur in their oxide, carbonates and hydrogencarbonates forms, which could account for the low solubility of Ca and Mg [17]. It can thus be asserted that the fluoride removal process in this ash is largely by two processes, namely, adsorption onto the insoluble compounds of hydrated CaO, MgO and Al_2O_3 and precipitation of CaF_2 and MgF_2 salts [4,5,7].

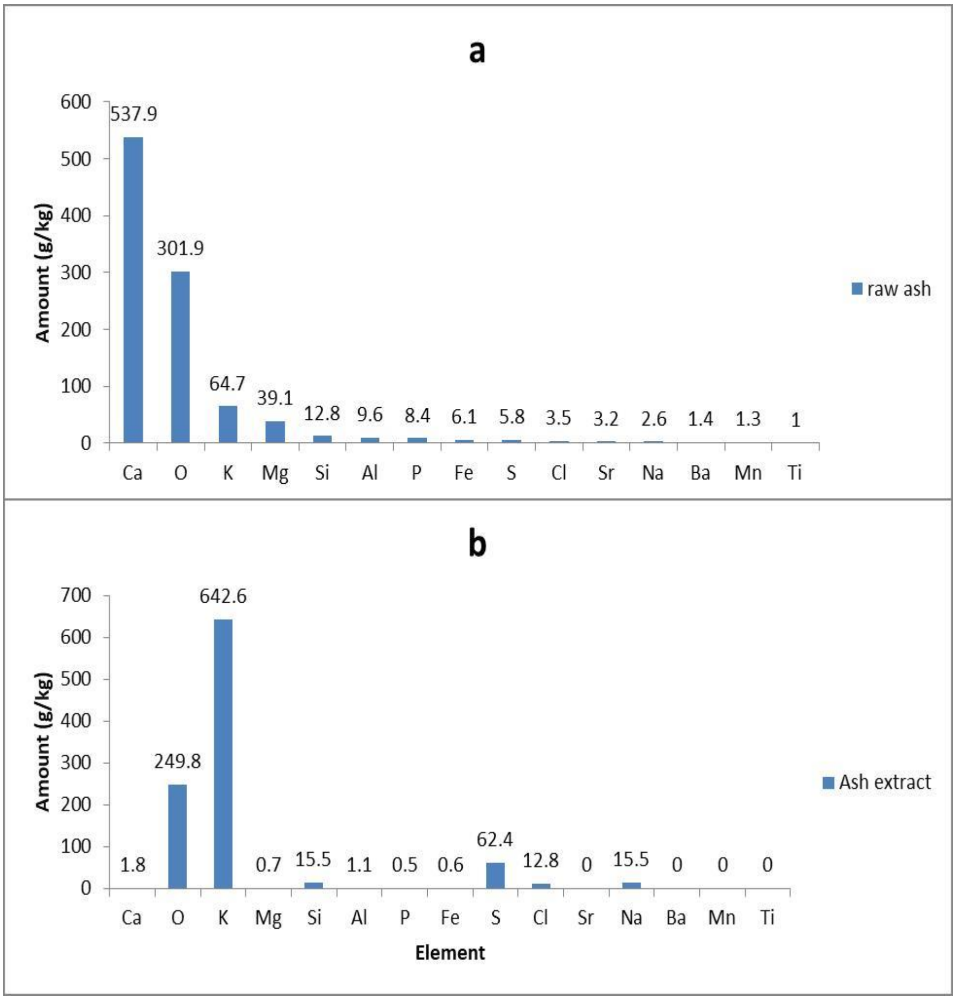

Fig 2. The chemical composition of the waste wood ash and ash-extract in batch experiments.

In the continuous flow experiment, findings reveal that the concentration of the media-introduced contaminants in the treated water declines steadily with time. In the first three hours the concentration of the K was found to increase sharply from 2,578 to 5,612 mgL^{-1} as shown in Fig. 3. This increase could imply that the material had not reached its maximum dissolution. A further increase in time resulted in the continuous decline in the concentration of K down to below 500 mg/L after 24 hours (Fig. 3). Since K^+ ion was the most soluble component in the ash material, it can reasonably be used to indicate the quality of the treated water with respect to the ash-introduced contaminants. It therefore, implied that the treated water will be suitable for consumption after discarding a few first aliquots. However, this makes sense if and only if its defluoridation capacity is sustained beyond this contaminant's leaching time. This is further explained in the subsequent sections when the defluoridation properties of the material was tested in batch and column experiments.

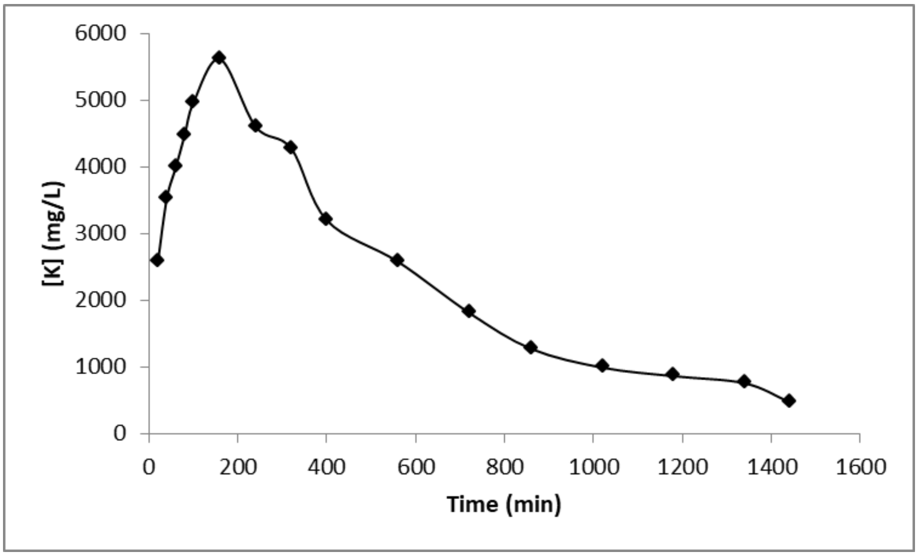

Fig 3. The effect of time on the concentration of K in the treated water at flowrate = 0.18 L/h

3.2. Fluoride removal efficiency of the biomass ash in the batch experiments

When 10 g of ash was added into 250 mL of fluoride water and hand shaken for 5 minutes and allowing 30 minutes for settling, a reduction of fluoride concentration from 20.8 to 4.5 mg/L was observed as shown in Fig. 4. The removal efficiency was found to increase with increase of settling time from 80% at 0.5 hours to 97% at 24 hours, when the residual fluoride was below 0.5 mg/L, suggesting removal by adsorption and co-precipitation [5,15].

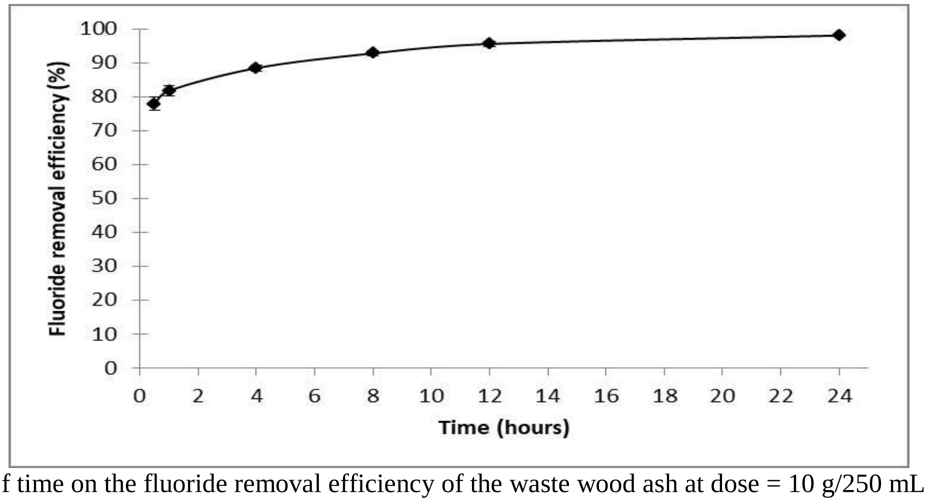

Fig 4. The effect of time on the fluoride removal efficiency of the waste wood ash at dose = 10 g/250 mL, F = 20.08 mg/L, pH = 8.3

This increase in fluoride removal efficiency with increasing settling time could be due to settling of the adsorbent particles which have already trapped fluoride ions and slow precipitation of the fluorides of Ca and Mg [4,5,15] as further confirmed by the parallel decline in turbidity of the treated water (Table 1). These results, therefore, indicate that the reaction between fluoride ion and adsorbent used is fast [8] and that additional time is required to effect settling of the adsorbent particles which have already trapped the fluoride ions. Therefore, a low flowrate is necessary in the column design to ensure that no adsorbent particles escape into the treated water [7]. The pH of the treated water was found to increase from 8.3 to an average of 12.9 in the treated water. The observed increase in the pH of the ash-treated water can be attributed to the dissolution of oxides and carbonates of metals present in the ash.

Table 1. The effect of time (h) on the total dissolved solid and turbidity of the ash treated water.

S/N	**Parameter**	**Before treatment**	**t_4**	**t_8**	**t_{12}**	**t_{24}**
1	Turbidity (NTU)	0.00	79	16	2	0
2	TDS (ppm)	130	6860	7105	7230	7920
3	pH	8.3-8.5	12.1	12.4	12.9	12.9

3.3 The effect of initial solution pH on fluoride removal efficiency ash material

Increasing the solution pH resulted in a decrease in the fluoride removal efficiency of the biomass ash, as shown in Fig. 5. The higher fluoride removal efficiency of the biomass ash at acidic conditions could be attributed to increased generation of ions of Ca and Mg from the ash matrix which are responsible for the formation of MgF_2 and CaF_2 precipitate. Alternatively, acid-induced positive charges on the surface of the partially burnt biomass ash particles in acidic conditions could have resulted into the increase in the number of active sites. A similar trend was reported when fly ash, potato peel and rice husk ash were used in water defluoridation experiments [8,18]. The low fluoride removal efficiency in higher pH could be due to increased competition between OH^- and F^- ions for active sites.

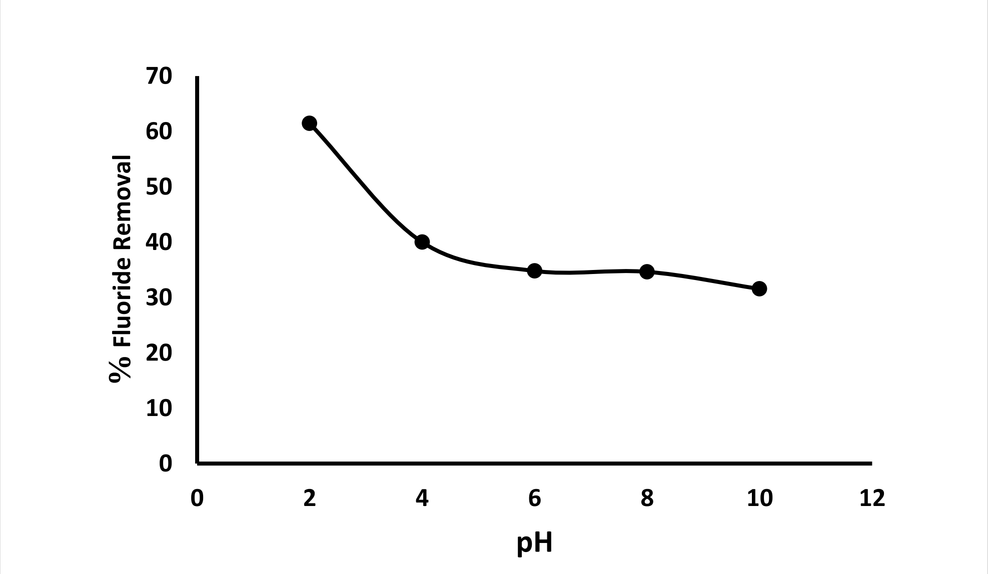

Fig 5. The effect of pH on the fluoride removal efficiency of the wood ash at F = 20.8 mg/L, dose = 10 g/250 mL, time = 2 h.

3.4 The effect of initial fluoride concentration on fluoride removal efficiency

An increase in the initial fluoride concentration resulted in an increase in the fluoride removal efficiency by biomass ash as shown in Fig. 6. This increase in fluoride removal efficiency when the initial fluoride concentration was varied from 5 to 15 mg/L could be an indication that the maximum removal capacity of the ash used was not attained. A similar trend was reported when fly ash was used for water defluoridation [7]. This suggests that biomass ash is a potential material for fluoride removal in water with high concentrations of fluoride.

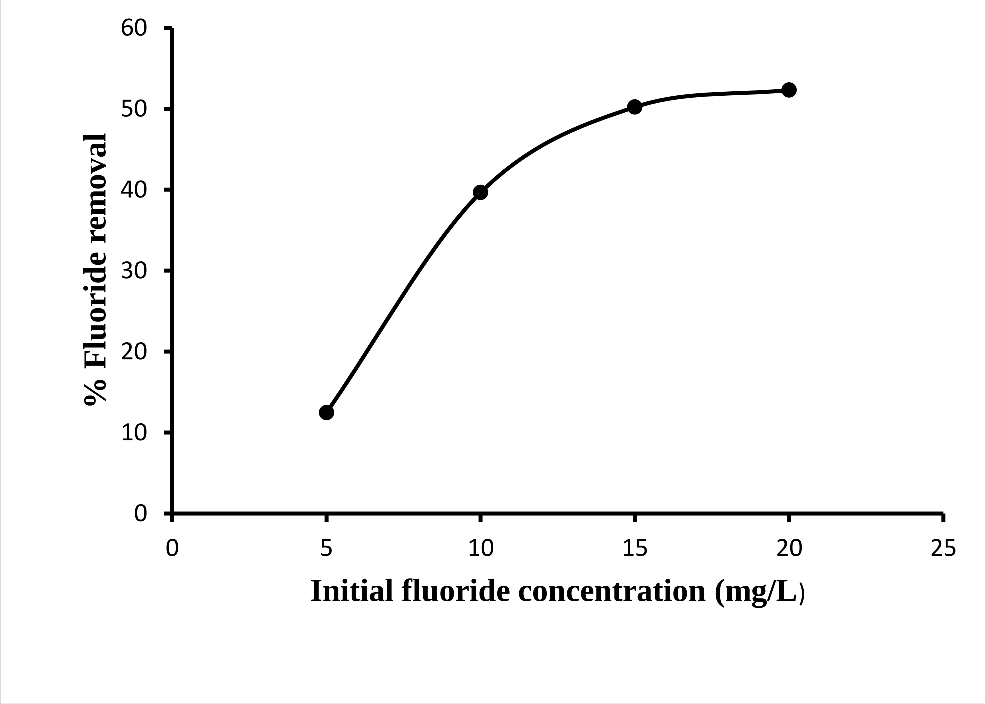

Fig 6. The effect of the initial fluoride concentration on the fluoride removal efficiency of the waste wood ash at dose = 10 g/250 mL, time = 2 h, pH = 8.3

3.5 Plant-assisted pH adjustment efficiency in the batch experiments

When ash treated water was added in a container with 35 *Cyperus* plants (each plant weighing on average 10.0 g), such that half of the plant height was immersed in water, its pH declined steadily with time from pH 12.2 to pH 7.3, after which no further decrease was observed with further increase in time. This is shown in Fig. 7. A parallel experiment using HCl-spiked acidic water with of pH 3.5, showed a steady increase in pH with time from 3.5-6.8 (Fig. 7). This effect was found to depend on the number of plants used, where the greater the number of plants the faster the pH change as further explained in the next section.

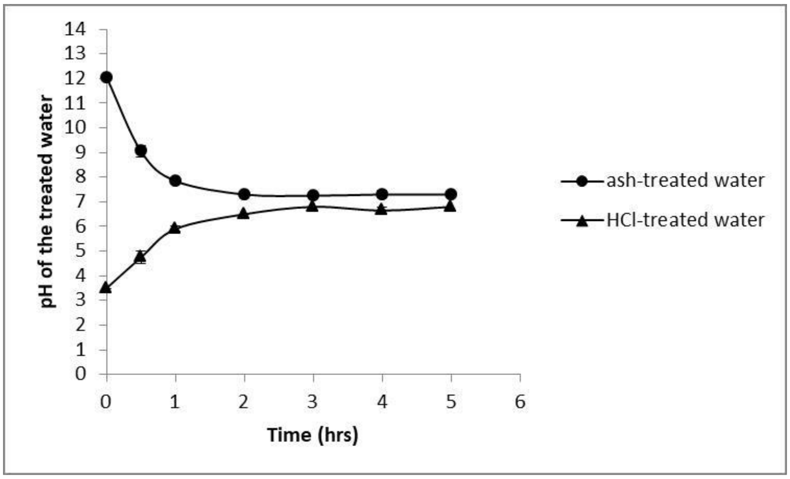

Fig 7. The effect of Cyperus plants on the pH of the treated water at dose = 350 g/L (plants per litre of water).

3.6 The effect of number of Cyperus plants on buffering effect of ash-treated water

The increase in the neutralising power of the *Cyperus* plant with increasing number of plants is attributable to the increasing concentration of the secreted natural buffering exudates resulting from the increased number of plants (Fig. 8). The attainment of a constant pH from both pH extremes (acidic and alkaline) in the pH scale suggests that, the pH adjustment mechanism involved the secretion of some form of the natural buffers by *Cyperus* plants whose constant pH is around 7. These natural buffers could be natural flavonoids, which these plants are rich in [19,20]. Samples of treated water collected from different height in the vessel revealed no significant difference in rate of neutralisation. This could indicate that this natural buffer is released from both the roots and the leaves of a plant.

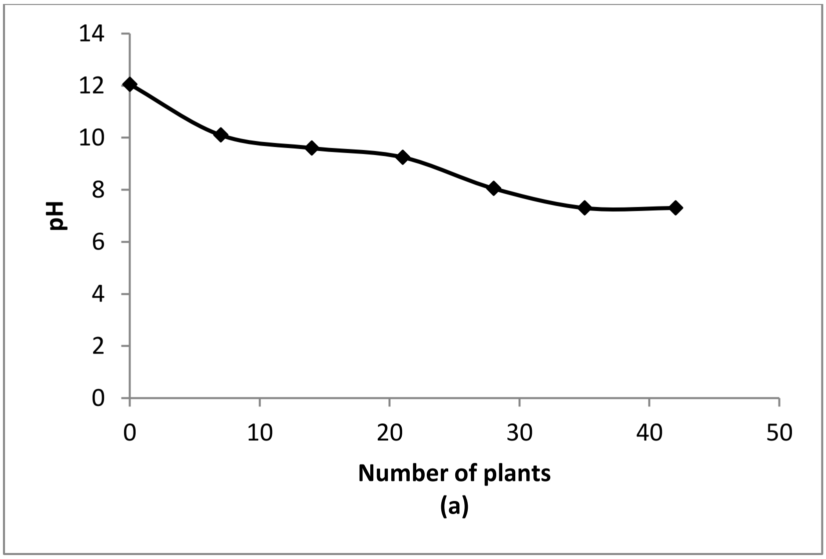

Fig 8. The effect of the plant dose on the pH of the treated water at initial pH = 12.

The concentration of carbonates decreased from 18.0 to 10.8 mg/L, whereas that of hydrogencarbonates increased from 427 to 562 mg/L in the *Cyperus*-treated water. The concentrations of Ca and K did not change significantly (Table 2). This observation indicated that the *Cyperus* exudates or their metabolites release H^+ to the aqueous solution, which combines with the carbonates and hydroxyl ions to form hydrogencarbonates and water, respectively, as shown in equation 1 and 2. This is likely due to the reported instability of the natural flavonoids in the varying solution pH [21]. This study, therefore, presents a new application of these plants in the field of water treatment. It further suggests the potential application of these plants in soil pH remediation and as natural buffers in chemistry laboratories.

$$CO_3^{2-} + FLV\text{-}H \rightarrow HCO_3^- + FLV^- \qquad (1)$$

$$OH^- + FLV\text{-}H \rightarrow H\text{-}OH + FLV^- \qquad (2)$$

Table 2. The effect of *Cyperus* plants on the concentration of K, Ca, CO_3^{2-} and HCO_3^- (mg/L) in the ash-treated water.

S/N	Chemical species	Before plant treatment (mg/L)	After plant treatment (mg/L)
1	K	1198.76	1190.30
2	Ca	43.11	40.02
3	CO_3^{2-}	18.0	10.8
4	HCO_3^-	427	562

3.7 Performance of the constructed wetland

When coarse sand, ash and fine sand were packed in a vertical flow constructed wetland as shown in Fig. 1, 120 g of ash was able to produce up to 10 L of the treated water, with a fluoride concentration below the WHO guideline of 1.5 mgL^{-1} at a flowrate of 0.18 Lh^{-1}, as shown in Fig. 9. The low flowrate was important for enhancing the probability of involvement of the second fluoride removal mechanism, namely, co-precipitation of fluorides ions with Ca and Mg ions, which is a slow process [4,5,22]. A comparison of the experimental and control columns revealed that fluoride remediation in this system is generally insignificant outside the ash zone and takes only a short time. Equally, the pH of the treated water was adjusted in the plant zone as columns without plants did not show any adjustment of pH of the ash treated water.

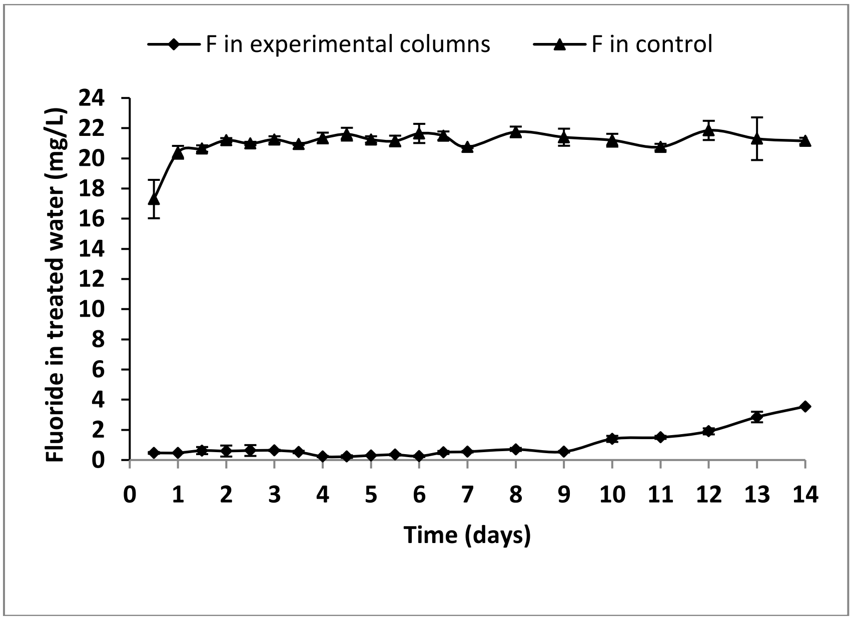

Fig 9. The variation of the residual fluoride concentration in the effluent water from the designed constructed wetland at flowrate = 0.018 L/h.

The concentration of potassium as the highly soluble component in the ash (Fig. 2b) was found to decrease steadily with time from 1653 in day 1 to below 50 mg/L in day 14. Findings reveal that the concentration of potassium falls steadily with time as the water washes through the ash as shown in Fig. 10. When this trend is viewed parallel with the trend in defluoridation efficiency presented in Fig. 4, three litres on average need to be discarded, after which the average concentration of the K falls well below 100 mg/L which is agreeable [23]. Considering the total amount of defluoridated water produced, and the pH remediation potential of the *Cyperus* plants, biomass ash can be applied as a low-cost material for water defluoridation in remote areas where provision of other defluoridation facilities is a challenge.

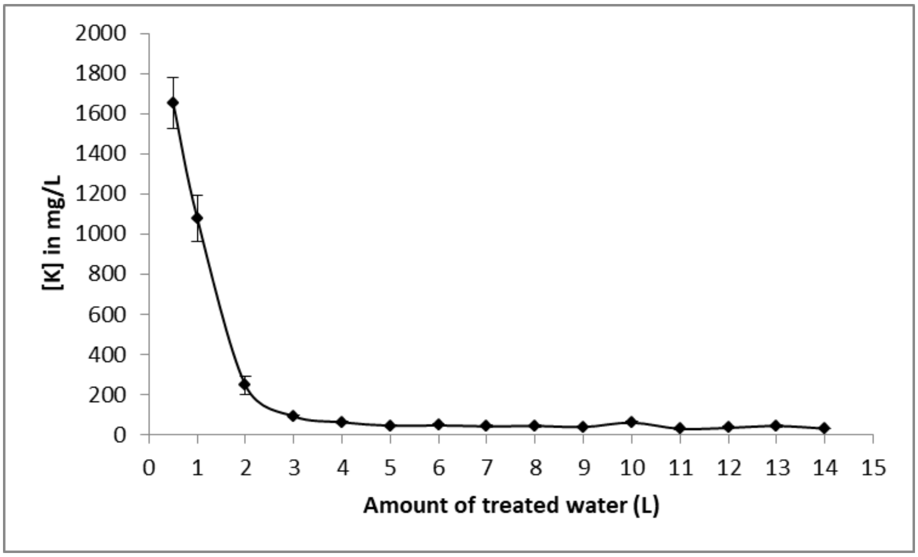

Fig 10. The variation of the concentration of K in the effluent water from the designed constructed wetland.

4. Conclusion

Waste biomass ash (wood ash) produced from kitchens in rural areas can be used for water defluoridation. The fluoride removal mechanism is likely by adsorption and slow co-precipitation. This therefore calls for slow flowrate in column designs. The pH elevation of the treated water resulting from application of ash could be addressed naturally and inventively by *Cyperus esculentus* plants in a constructed wetland. Findings show that these plants release a natural buffer that maintains the pH of the treated water at neutral. Therefore, these plants could be applied in pH adjustment of the water and soil. Furthermore, these findings unlock the applicability of wood ash as defluoridation material. Waste wood ash coupled with *Cyperus* plants in a constructed wetland provide a low-cost defluoridation and pH adjustment method applicable by communities in rural areas where these materials are available plenty.

Abbreviations and Acronyms

Abbreviation and acronyms should be defined the first time they appear in the text, even after the have already been defined in the abstract. Do not use abbreviations in the title unless they are unavoidable.

Appendix

Appendixes, if needed, appear before the acknowledgment.

Acknowledgements

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Ethical Statement

This study does not contain any studies with human or animal subjects performed by any of the authors.

Conflict of Interest

The authors declare that they have no conflict of interest.

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