Research Article

A Comparative Study of Fluoride Removal Using Bovine and Goat Bone Chars

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Abstract

Overexposure to fluorides causes dental, skeletal, and crippling fluorosis. Population growth and droughts in Kenya recently necessitated the sinking of boreholes to supply drinking water. The water fetched from the boreholes contains a high concentration of fluorides, consequently increasing the prevalence of dental fluorosis. Bone char remains a sustainable adsorbent to remove fluorides from drinking water as it is environmentally friendly, has high efficiency, is easy to use, and is low cost compared to other adsorbents or fluoride removal methods that may be technically non-feasible in rural communities. This study compared the fluoride removal using bovine and goat bone chars. Bovine and goat bone chars were prepared by calcining in a muffle furnace at 400 ℃ and 600 ℃. Bone chars made at 400 ℃ appeared gray while those made at 600 ℃ appeared darker. Bone char was activated using 1 M H_2SO_4 at a ratio of 40 ml per 2 g of bone char and a contact time of 24 hours. The activated bone chars also appeared grey. The surface functional groups were examined by infrared spectroscopy (FT-IR), which revealed similar functional groups in both bovine and goat bone char and their activated parts, with notable differences in peak depths. The amount of inorganic materials was determined by portable X-ray fluorescence (XRF) which showed high concentrations of Ca, P, Mg, Si, Al, Fe, S, K, Mn and Ti in decreasing concentrations before and after using H_2SO_4. The prepared bone chars adsorbed fluorides from a concentration of 2 ppm to below WHO-recommended fluoride limits in 30 minutes, with bovine bone chars removing 66.8% and goat bone chars 61.8% of the initial fluoride concentrations.

1. Introduction

The effects of high water fluoride concentrations remain an endemic problem in the East African Rift Valley, Japan, the region between Turkey and China, Australia, the United States of America, and South America [1]. Various minerals such as rock phosphate, cryolite, apatite, fluorspar, and topaz contain fluorides which dissolve in groundwater resulting in high concentrations [2]. Nearby volcanic activity and fumarole gasses from hot springs amplify groundwater fluoride concentrations in these regions. The large water consumption of industry results in the co-production of heterogeneous wastewater streams that often contain a wide range of chemical pollutants, including fluorides, which dissolve and enter groundwater through weathering processes and water circulation [3]. High concentrations of aluminum, low concentrations of magnesium and calcium oxides, and soil conditions such as alkalinity increase subsoil fluoride ion leaching [4]

which also increase the availability of fluorides in water sources [5, 6].

Although fluoride is important for developing and maintaining dental health, it poses health risks when ingested in high concentrations. Chronic exposure to fluoride at concentrations >1.5 mg/L is associated with the development of dental fluorosis with symptoms ranging from tiny white streaks to dark brown spots and rough, pitted enamel that is difficult to clean [7]. Prolonged water consumption >4 mg/L causes dental and skeletal fluorosis with symptoms such as lower back pain, stiffness and pain in the joints, and bone fractures. Exceeding concentrations > 10 ppm leads to debilitating skeletal fluorosis with symptoms such as restricted joint movement, muscle wasting, abnormal spinal flexion and neurological defects due to spinal cord compression [8]. Other effects include a delay in neurological development in children, changes in the immune system and human reproductive system, and health problems associated with the kidneys and gastrointestinal tract [9]. Almost 3.5 billion people worldwide are affected throughout their lives, causing pain, discomfort, disfigurement and even death. Unfortunately, oral health treatment is expensive and usually not covered by general health insurance (Universal Health Coverage). This makes low- and middle-income countries vulnerable as the prevalence of these health problems increases due to urbanization and lifestyle changes [10]. This calls for technical and scientific interventions to help solve the problem.

Various technologies have subsequently been developed to remove fluoride from water systems to reduce its concentrations to below 1.5 mg/l, which is the current WHO recommended limit [11]. The different technologies used include co-precipitation [12], precipitation coagulation [13], electrocoagulation [14], adsorption [15], ion exchange [16] and membrane processes [17] or a combination of the above technologies [18]. Notwithstanding, these methods pose problems such as high operating costs, waste production,

stringent pH and other experimental conditions, and the use of toxic chemicals that limit their use in water defluoridation [11].
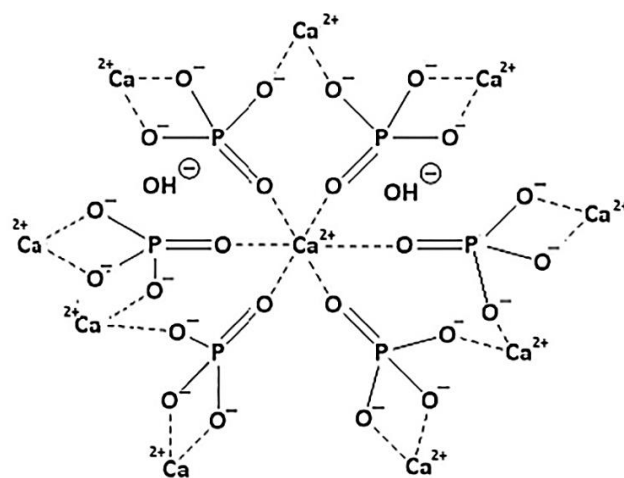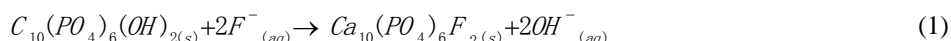

***Figure 1.** Structure of hydroxyapatite.*

It has been reported that adsorption/ion exchange processes are the ideal method for fluoride removal due to their excellent adsorption rate, wide range of adsorbents, ease of operation as well as high removal efficiency [1] Various adsorbents are used including bone char [1, 19], nanomaterials [20, 21] zeolites [22, 23], activated carbon [24, 25], aluminium [26] and biochar [27, 28]. Bone char contains calcium hydroxyapatite, which can exchange hydroxyl ions for fluoride ions in the water, making it safe. The structure of the hydroxyapatite is shown in Figure 1 while the equation showing the adsorption of fluoride onto the hydroxyapatite is shown in equation (1).

$$C_{10}(PO_4)_6(OH)_{2(s)} + 2F^-_{(aq)} \rightarrow Ca_{10}(PO_4)_6F_{2(s)} + 2OH^-_{(aq)} \tag{1}$$

This work performed a comparative study of fluoride removal by adsorption on goat and bovine bone char. Bone char is inexpensive, biocompatible and easy to produce adsorbent [29]. In particular, the main focus of this work was to give a comprehensive comparative overview of bone char production, activation by chemical treatment and characterization of the functional groups and elemental composition, particle characterization and the efficiency of fluoride removal. The outlined work was completed using a muffle furnace to produce the bone char at 400 ℃ and 600 ℃, activation with H_2SO_4, characterization of the functional groups by infrared spectroscopy, elemental analysis by X-ray fluorescence (XRF), and particle characterization by light microscopy. The adsorption was monitored on continuous columns and the decrease in fluoride concentration was monitored with Low Range Fluoride Meter HI 96729.

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2. Materials and Methods

2.1. Materials

The bones used in this work are beef and chevon (goat) bones sourced from slaughterhouses in the city of Nairobi, Kenya. Sulfuric acid of molecular weight = 98 and pH strips were obtained from Science Lab Kenya Limited. Deionized water was produced at the Technical University of Kenya's School of Chemistry and Materials Science Labs. Potassium bromide of molecular weight =119 used in the acquisition of IR spectra was supplied by Sigma Aldrich Chemical Co. Sodium fluoride with molecular weight = 41.98817 obtained

from Science Lab Kenya Limited was used to prepare pilot water samples.

2.2. Bone Char Preparation

Bone char was prepared following previous work by [1, 30]. The bones were degreased with boiling water in a saucepan for 1 hour 30 minutes and dried in an oven at 110 ℃ for 6 hours. The bones were weighed and placed in an aluminium pot and sealed using a ceramic tile to prevent ingress of excess oxygen and to provide the limited oxygen environment required for charring. The charring was done in a muffle furnace set at 400 ℃ and 600 ℃. The chars were recovered after 1 hour of carbonization.

The bone chars were stored in air-tight containers to prevent any interaction with air. The chars were then ground using a milling machine and graded using 125 μm, 250 μm, 500 μm and 1000 μm pore size sieves. Each sample produced five samples; <125 μm, 125-250 μm, 250-500 μm, 500-1000 μm and >1000 μm. 250-500 μm char were selected for the study.

2.3. Bone Char Activation

Milled samples of both bovine and goat bone chars of 250-500 μm were separately activated using 1 M H_2SO_4 at a ratio of 40 ml per 2 g bone char and a contact time of 24 hours [30, 31] The chars were washed with deionized water until a neutral pH (pH=7) was achieved. The chars were then dried in an oven at 105 ℃ for 2 hours and stored in airtight containers for adsorption studies.

2.4. Instrumentation

2.4.1. Analysis of Surface Functional Groups by Fourier Transform Infra-Red Spectroscopy

Bone char sample spectra were recorded with the FTIR-600 spectrophotometer (School of Chemistry and Material Science Lab, Technical University of Kenya), model WQF-520, in the range of wavenumbers 4000-400 cm^{-1}. 20 scans were collected with a resolution of 4 cm^{-1}. Neat KBr pellets were prepared using a hydraulic press and a load of 25 psi was applied for 3 minutes. A background spectrum was previously recorded, followed by the analysis of the samples dusted into the KBr pellets.

2.4.2. Elemental Analysis

The elemental analysis was performed with a portable Bruker XRF, model number S1 Titan 800. A small amount of each sample was placed in the sample holder and scanned with X-rays for 60 seconds for the range of Mg to U. The results were obtained as percentage oxide compositions which were converted to give the corresponding elemental compositions of the total inorganics present. The data were presented as a table and a stacked chart of major elements in the prepared bone chars.

2.4.3. Adsorption Studies Monitored by Low Range Fluoride Meter HI 96729

Dynamic columns were used for the adsorption studies. Burettes were used to represent the columns. A piece of cotton was placed on the bottom of the burette and covered with acid-washed sand, and 5 g of each of the char produced was packed on the columns. Pi-lot water containing 2 ppm F^- was prepared by dissolving 0.02889 g of NaF in 5 L of deionized water.

The pilot water was passed through the columns at a rate of 1L/hour and fluoride concentrations in the treated waters were measured using up to 500mL of water treated with 5g of bone char. The removal efficiency was calculated based on the concentrations of adsorbed fluorides relative to the initial concentration and expressed as a percentage.

3. Results and Discussion

3.1. Prepared Bone Chars

Figures 2 (a) and (b) show the cleaned bones of cattle and goats, respectively. Figures 2 (c) and (d) show the charred bones of cattle and goats respectively. Chars appearing grey are attributed to incomplete calcination, with proportions of organic compounds present [32].
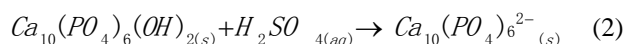
The studies described the chemistry of activation as involving the following reactions [33, 34];

Figure 2. Different forms of bone char obtained.
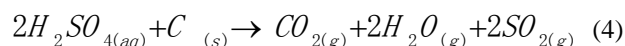
Treatment of bone with H_2SO_4 leads to

1. A partial dissolution of hydroxyapatite forming $CaSO_4$ and anion deficient hydroxyapatite. (HAP) as shown in equation (2).

$$Ca_{10}(PO_4)_6(OH)_{2(s)} + H_2SO_{4(aq)} \rightarrow Ca_{10}(PO_4)_6{}^{2-}{}_{(s)} \quad (2)$$

2. A higher release of CH_4 due to methanation of CO_2 as shown in equation (3).

$$CO_{2(g)} + 4H_{2(g)}) \rightarrow CH_{4(g)} + 2H_2O_{(1)} \quad (3)$$

3. Formation of high levels of CO_2, SO_2, and steam as shown in equation (4).

$$2H_2SO_{4(aq)} + C_{(s)} \rightarrow CO_{2(g)} + 2H_2O_{(g)} + 2SO_{2(g)} \quad (4)$$

These chemical reactions result in a highly micro-porous and meso-porous structure with excellent surface adsorption properties.

3.2. Surface Functional Groups

The IR spectra of activated and non-activated bovine and goat bone chars are shown in Figures 3-6 below. Analysis of the IR spectra of the activated and non-activated bone chars showed that all samples have similar functional groups related to band positions. Notable differences exist in the intensity of the bands. The surface functional groups in both activated and non-activated bovine and goat bone char are the same; therefore, adsorption would occur similarly.
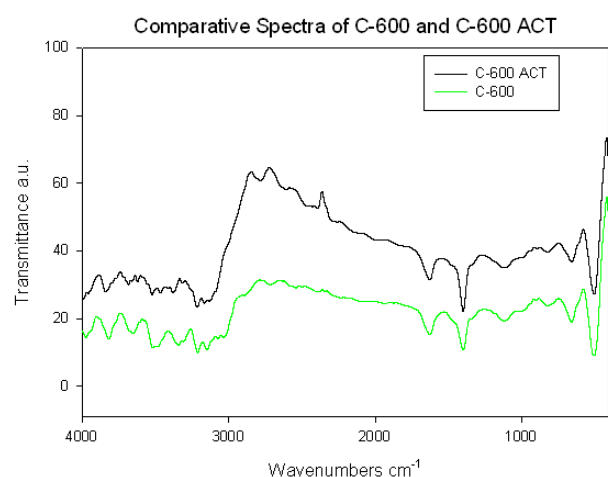

Figure 3. Comparative Spectra of bovine bone charred at 600 ℃ (C-600) and activated bovine bone char charred at 600 ℃ (C-600 ACT).
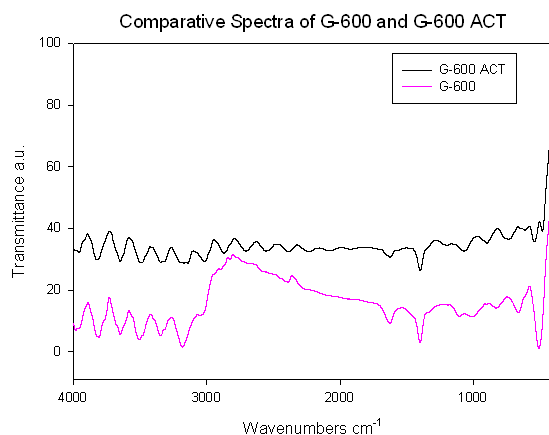

Figure 4. Comparative Spectra of goat bone charred at 600 ℃ (G-600) and activated goat bone char charred at 400 ℃ (G-600 ACT).
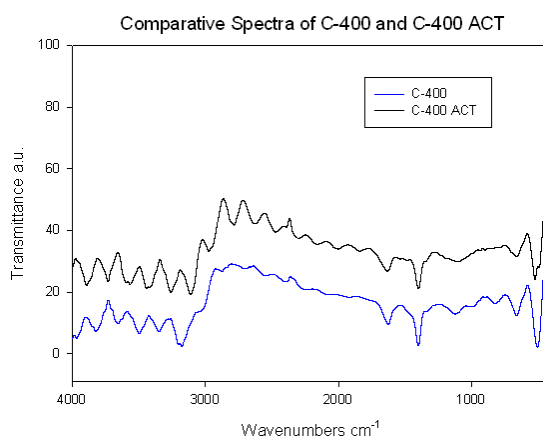

Figure 5. Comparative Spectra of bovine bone charred at 400 ℃ (C-400) and activated bone char charred at 400 ℃ (C-400 ACT).
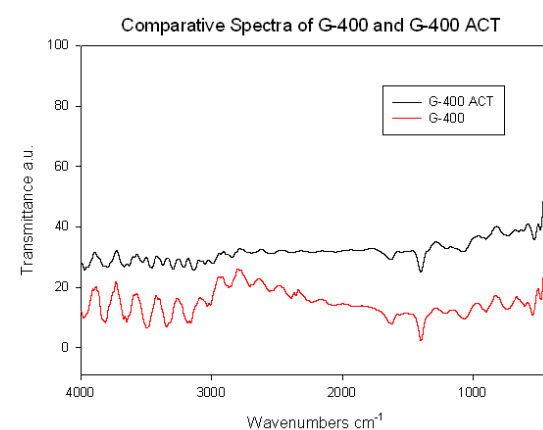

Figure 6. Comparative Spectra of goat bone charred at 400 ℃ (G-400) and activated goat bone char charred at 400 ℃ (G-400 ACT).

The various peaks represented on the spectra are interpreted as per the Table 1 below.

Table 1. *Functional group interpretation of the IR spectra.*

Species	Peak position for non-activated char	Peak position for activated char	Functional groups
Goat	480	485	
Bovine	507	507	PO_4^{3-}
Goat	663	663	
Bovine	659	659	
Goat	824	833	
Bovine	824	824	Aromatic rings
Goat	1062	1072	
Bovine	1108	1108	P-O; PO_4^{3-}
Goat	1398	1403	C=O;
Bovine	1400	1401	C=C;
Goat	1627	1635	O-H,
Bovine	1625	1627	H-O-H
Goat	>3200	>3200	
Bovine	>3200	>3200	O-H*

*The number of bands increases with an increase in the calcination temperature.

3.3. Elemental Analysis

The elemental composition from X-ray fluorescence analysis is presented in Table 2 below. The increase and decrease in concentrations of elements is attributed to the formation of soluble sulfates that are washed off during cleaning with distilled water. This leads to a corresponding increase in the concentration of insoluble sulfates. [35]

Table 2. *Elemental composition of inorganics as shown by handheld XRF (%).*

Sample Element	C-400	C-400 ACT	C-600	C-600 ACT	G-400	G-400 ACT	G-600	G-600 ACT
Ca	21.041	19.961	20.801	19.257	21.213	21.642	20.963	19.855
S	16.838	15.974	16.643	15.407	16.972	17.316	16.772	15.885
P	19.76	21.687	20.221	24.348	19.102	17.386	19.661	22.761
Mg	0.736	1.243	0.778	0.555	0.9829	1.315	0.983	0.693
Si	0.261	0.313	0.233	0.322	0.234	0.280	0.233	0.280
Al	0.228	0.301	0.291	0.297	0.254	0.227	0.254	0.291
Fe	0.0350	0.063	0.035	0.070	0.035	0.112	0.035	0.049
Ti	0.006	0.012	0.006	0.012	0.006	0.006	0.006	0.012
Mn	0.008	0.016	0.008	0.016	0.008	0.016	0.016	0.008
K	0.017	0.008	0.100	0.008	0.025	0.033	0.017	0.008

The major elements in the prepared bone chars are illustrated in Figure 7 as shown below.
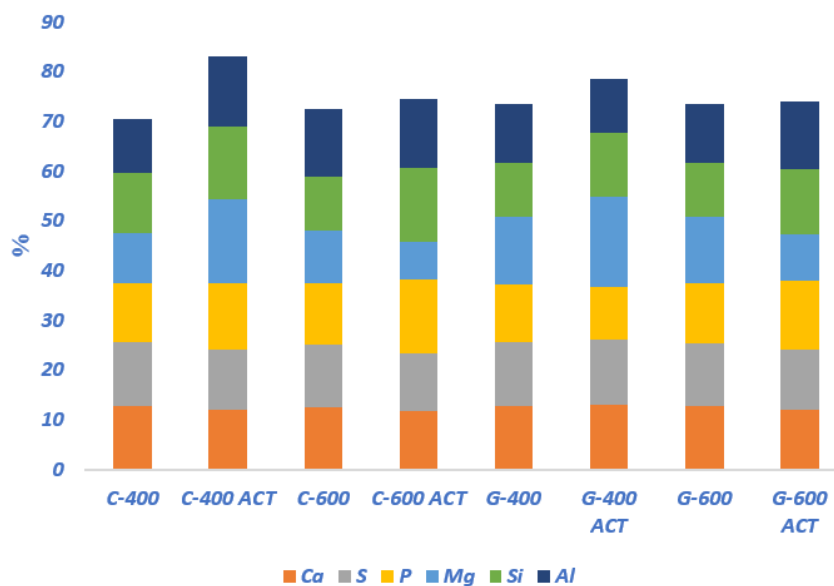

Figure 7. A stacked column chart showing the concentrations of major elements found in bone chars.

3.4. Adsorption Studies

Adsorption studies were only performed using bone char activated at 600 ℃ because that prepared at 400 ℃ produced yellow-coloured water upon treatment. This is due to the dissolution of organic compounds in bone char due to incomplete calcination [32, 35, 36]. The water treated at 600 ℃ appeared clear. The adsorption of fluorides in hydroxyapatite is illustrated in Equation 1.
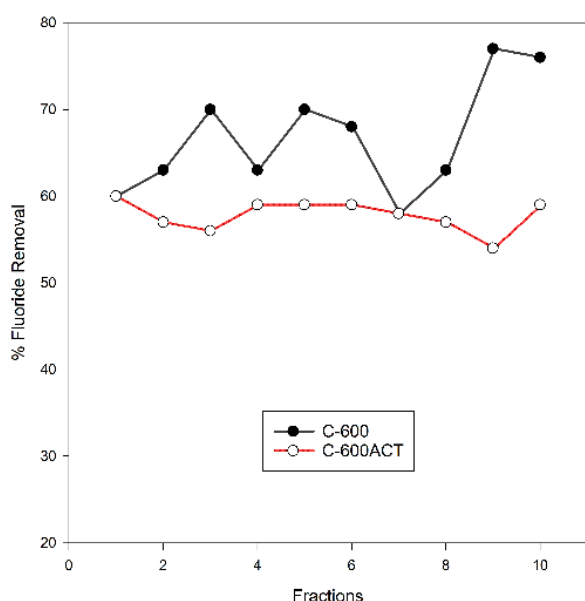

Figure 8. A Comparative line graph showing fluoride removal using bovine bone chars.

Figure 9. A Comparative line graph showing fluoride removal using bovine bone chars.

Fluoride concentrations in the pilot water after elution from the columns were measured to determine the adsorption potential of the char produced. From 2 ppm, fluoride concentrations were reduced to below the WHO recommended limit of 1.5 ppm, as shown in the comparative line graph in Figures 8-9.

On average, C-600 reduced fluoride concentrations from 2 ppm to 0.664 ppm, while G-600 reduced concentrations of fluorides from 2 ppm to 0.764 ppm, representing a 66.8% and

61.8% reduction, respectively. In contrast, activated bone char fractions reduced fluoride concentrations from 2 ppm to 0.844 ppm and 0.806 ppm for C-600 ACT and G-600 ACT, respectively. This shows a reduction of 57.8% and 59.7% respectively. Change in pH during adsorption using activated chars increases electrostatic repulsion resulting in a rapid decline in the adsorption capacity of the functional groups. The activated chars therefore recorded decreased performance. [1, 37]

4. Conclusion

This study has shown that the properties of goat bone char are similar to those of cattle bone char, indicating a promising alternative for use in water defluoridation. The prepared bone chars have a slightly higher fluoride removal potential compared to the activated bone chars. Bovine chat at 600 ℃ recorded a 66.8% fluoride removal while goat bone charred at 600 ℃ recorded 61.8% fluoride removal. Activated bovine char at 600 ℃ had a 57.8% fluoride removal while activated goat bone char had a 59.7% fluoride removal. 5 g of the char treated 500ml of contaminated water to WHO recommended levels in 30 minutes. Therefore, this work recommends the use of goat bone chars as an alternative to bovine bone chars in water defluoridation.

Further studies should focus on (1) the characterization of specific surface areas, microstructure and crystallinity of the bone char adsorbent materials. (2) Optimization and modelling of adsorption mechanisms and kinetics.

Abbreviations

C-400	Bovine Char Calcined at 400 ℃
C-400 ACT	Activated Bovine Char Calcined at 400 ℃
C-600	Bovine Char Calcined at 600 ℃
C-600 ACT	Activated Bovine Char Calcined at 600 ℃
G-400	Goat Char Calcined at 400 ℃
G-400 ACT	Activated Goat Char Calcined at 400 ℃
G-600	Goat Char Calcined at 600 ℃
G-600 ACT	Activated Goat Char Calcined at 600 ℃
WHO	World Health Organization

Author Contributions

Geoffrey Otieno: Conceptualization, Methodology, Project Supervision, Resources, Validation, Writing – review and editing.

Joab Otieno Onyango: Resources, Writing – review and editing.

Lucas Odhiambo Sije: Conceptualization, Data curation, Formal Analysis, Investigation, Methodology, Software, Validation, Visualization, Writing – original draft.

Data Availability Statement

The data supporting the outcome of this research work has been reported in this manuscript.

Conflicts of Interest

The authors declare no conflicts of interest.

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Biography

Geoffrey Otieno Geoffrey is currently the Director of The School of Chemistry and Material Science at The Technical University of Kenya. Geoffrey obtained a PhD in Material Science from the University of Oxford, UK in 2012; and a Master's in Advanced Materials Engineering from Kangwon National University, South Korea (2007). He is a member of the Kenya National Academy of Sciences. He has been a member of the Governing Council of Kenya Chemical Society (KCS) since 2015. He is a fellow of the Organization of the Prohibition of Chemical Weapons (OPCW) having completed the Associate Program in 2017. Geoffrey's career as a professional Chemist and Material Scientist spans over 20 years. These years have been spent as a researcher, and advisor to government, international organizations and private organizations. As an academic, Dr Otieno worked at Kangwon National University, South Korea as a researcher on the development of bipolar plate materials for fuel cells supported by Samsung and as a post-doctoral researcher at Oxford Materials on novel nanostructures for applications in electronics and body armor. His research interests are in the area of nanomaterials with applications in water treatment and renewable energy. Recent key projects include leading research with Nestle Waters (France) in formulating biodegradable polymers and the University of Oxford on Solar concentrators for bone charring.

Joab Otieno Onyango is a Senior Lecturer in the School of Chemistry and Material Science at the Technical University of Kenya, Nairobi. He holds a PhD in chemistry from the State University of New York at Binghamton, New York. Since joining Technical University of Kenya, Joab has been active in research in the fields of synthetic modification of polymer for biodegradable plastics for various applications; malaria treatment and management using plant-based vector control as well as nano-based multidrug agents for plasmodial control. Joab has also ventured in natural products research including phytochemical screening, isolation and characterization of bioactive components for various applications such as water purification and disease management frontiers. These research trajectories have benefitted both undergraduate degree research projects and postgraduate students resulting in theses and publications in peer-reviewed journals.

Lucas Odhiambo Sije is a research and teaching assistant in the School of Chemistry and Material Science at the Technical University of Kenya, Nairobi, Kenya. Lucas completed his Bachelor of Technology in Industrial Chemistry from the Technical University of Kenya in 2022 with First Class Honours and was privileged to be the University Valedictorian during the university's 11th graduation ceremony held in Nairobi, Kenya. This work was his undergraduate thesis, submitted to the School of Chemistry and Materials Science at the Technical University of Kenya. He has participated in multiple international trainings and conferences including the 2022 OPCW Chemical Safety Training for the Youth in Kenitra, Morocco; the 2023 International Summer Student programme at the Helmholtz Zentrum Berlin, Germany and the 2024 Global Challenge Lab organized by the Imperial College London.

Research Fields

Geoffrey Otieno: Water treatment, Biodegradable plastics, Nanomaterials, Fuel cell materials, Adsorption mechanisms and kinetics.

Joab Otieno Onyango: Biodegradable Polymers, Water treatment, Natural products, Medicinal products, Anti-cancer drugs.

Lucas Odhiambo Sije: Water treatment, Energy materials, Perovskite solar cells, Hydrogen production and transport, Fuel cell materials.