Physicochemical characterization of biochars from *Eucalyptus maiden Entandrophragma cylindricum* (Liboyo)*, Milicia excelsa* (Muvula.) and *Ocotea michelsonie* (licheche) used in Goma city, DR Congo for water treatment potentials

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Abstract

In recent years, research on biochar as an eco-friendly material and cost-effective means for water treatment, soil amendment and carbon sequestration has gained more attention due to the availability of feedstock, the simplicity of the preparation methods, and their enhanced physico-chemical properties. Given that pyrolysis temperature and resident time amongst other factors have significant effect on biochar's pollutant removal efficacy, this study focused on the evaluation of some properties of biochars produced by pyrolysis (500 - 600 °C) from *Entandrophragma cylindricum* (ECB)*, Eucalyptus maiden* (EMB)*, Milicia excelsa* (MEB) and *Ocotea michelsonie* (OMB) sawdusts for their water treatment potentials by using standardized methods. Biomass generated from wood mill in the city of Goma (DR Congo) is valorised for the first time as potential water purifier. The highest yield was obtained from OMB (36.6%) which was found to be significantly greater that those of ECB and EMB ($p<0.05$). All the biochars had neutral to weakly alkaline pH (7.10-7.90), very high porosity (92-94%, with EMB having the highest value) and ash content between 9.40 and 18.40%, with the highest value attributed to OMB). Most physical and chemical characteristics of biochars varied significantly due to different wood species. Potential toxic elements were far below environmental threshold values and exchangeable cations were equally detected in the biochars. The obtained biochars are therefore seen as good media for water treatment.

Keywords: Water treatment, pyrolysis, removal efficacy, ash content, contaminants, porosity

1. Introduction

Water quality is continuously deteriorating due to rapid industrialization, population expansion and climate change (Lumnwi *et al.*, 2018). The resulting pollution caused by chemicals and/or pathogenic microorganisms poses a serious threat to the well-being of the earth and its people. According to (UNICEF and WHO, 2015; Biosengazeh *et al*., 2020, 663 million people with 319 million in Sub-Saharan Africa, of which 80% live in rural areas, still lack improved drinking water sources. These figures show the failure of the United Nations millennium Development Goals (MDGs) in Sub-Saharan African countries. Recently, with the adoption of Sustainable Development Goals (SDGs), the United Nations through SDG 6 intend to ensure the availability and sustainable management of water and sanitation for the entire world population by 2030 (UN, 2015, Nchofua *et al.,* 2020). However, SDG 6 may only be achieved in Sub-Saharan African countries if community-based water treatment methods are elaborated and practically put in use. In this regard, biochar technology appears to be promising as it relies on locally readily available materials and uses local skills, making it appropriate for small and low-income communities (Niwagaba *et al*., 2014).

The origin of biochar is traced to the ancient populations of Indian America in the Amazon region, known locally as "Terra Preta de Indio", where black earth was created using coal, traditionally produced (Lehmann*,* 2009). Research on Terra Preta soils (anthroposols) in the Amazon revealed the effects of biochar on soil by the improvement of its fertility in a sustainable way. Biochar is defined as char produced by pyrolysis for use in agriculture (and other non-thermal applications) in an environmentally sustainable manner (EBC, 2013). As a stable carbon-rich material Biochar shows incredible potential to handle water/wastewater contaminants (Enaime *et al*., 2020). It is generally produced by pyrolysis from agricultural residues, manure or wood, and characterised by having a large surface area ($200–1000$ m^2/g), low density and high porosity which makes it an efficient adsorbent and good biofilm carrier (Downie *et al*., 2009; Enaime *et al*., 2020). Due to its unique properties, there is growing interest in using it as a filter medium to enhance water and wastewater quality (Perez-Mercado *et al.*, 2018). Many studies have also demonstrated its efficiency as an adsorbent and biofilm carrier for removing organic matter, surfactants, phosphorus (P) and nitrogen (N) from onsite wastewater and greywater treatment systems (Rasheed *et* al., 2017; Gwenzi, 2018; Ye *et al*., 2019). However, its physical, chemical and structural properties can vary greatly depending on the type of organic material used and the biochar production conditions (temperature, heating rate and oxidation) (Perez-Mercado *et al.*, 2018).

The Democratic Republic of Congo (DRC) has one of the highest available water resources in Africa. However, potable water supply remains a major concern in many urban and rural communities in this country. The DRC is one of the Sub-Saharan African countries which have recorded the highest number of cases of cholera. In 2017, 55 000 people fell sick that is 28% more compared to the previous year and 1190 died in 24 of the 26 provinces of the country (RDC, 2018). The country is also very rich in forestry species which are generally used for wood works generating by-products such as sawdust that causes serious environmental problems as they are not managed in a suitable way. Some authors in DRC have worked on biochar as part of soil amendment (Peltier *et al*., 2010; Lele, 2016; Schure *et al*., 2019). However, no critical work has focused on the potential use of wood biochar for water purification in this country.

The main objective of the study was to assess the potential of wood species in Goma for water treatment as a contribution to enhancing the access to portable water of the population.
Specifically, the study focused on the:
- Determination of wood species widely used in Goma from which biochar can be obtain
- Determination of the biochar yield of the identified wood species.
- Evaluation of the biochars physicochemical characteristics for their potentials in the purification of water.

This research focuses on the valorisation of biomass generated from wood mill in the city of Goma which may serve as potential water purifier thus, mitigating environmental pollution as well as providing sustainable water supply to households in the city in particular and the entire DRC in general.

2. Identification of wood species widely used in the city of Goma

A questionnaire was administered to eight sawmills in the city to identify the most used lumber in the city. Two selection criteria (quality and the availability of species) guided the investigation. The non-parametric test was applied to identify the most commonly used wood species and the species that produces the highest quantity of sawdust.

3. Biochar production and determination of biochar yield

Variation in pyrolytic temperatures and feedstocks affects the yield and nutrient composition of biochar (Naeem *et al*., 2014). During pyrolysis, biomass undergoes a variety of physical, chemical and molecular changes. Volatilization during pyrolysis causes significant loss in mass and therefore volume reduction and shrinking without causing much change to the original structure of the feedstock (Laine *et al*., 1991). In addition, pyrolysis affects cation exchange capacity (CEC), pH and carbon content of biochar (Wu *et al*., 2012; Naeem *et al*., 2014). Yield determination is thus of prime importance to determining optimum condition and the best feedstock for the production of biochar for a particular use. The raw materials were collected in bags and transported to the biochar production site. Pyrolysis was carried out in a traditional Adam Retolt Kiln oven. Pyrolysis temperatures were taken every

20 minutes using a chromel alumel thermocouple until completion. Each sample was pyrolyzed in ten cycles and the masses were taken before and at the end of each production, using a salsa balance and the production yields calculated by using formula 1 (Naeem *et al*., 2014; Boris *et al*., 2018).

$$B_y(\%) = \left(\frac{R_W}{B_W}\right) \times 100 \tag{1}$$

where B_y = yield of biochar (% on a mass basis), R_w = weight of raw dry material (kg), and B_w = weight of biochar (kg).

4. Physical characterization of biochars

Three samples from a mixture of 10 pyrolyses of each type of biochar were analysed.

4.1 Particle size analysis

The shape and size of external biochar pores is a function of particle size and particle morphology (Lim *et al*., 2017). Particle size and particle morphology are often expressed in terms of Uniformity Coefficient (UC) and have a significant impact on biochar's ability to retain or eliminate contaminant from water. The particle size analysis was carried out following method prescribed by NF P 94-054 (1991). The process consisted of weighing 200 g of the sample, sieving it on a Controlab brand electronic sieve with a battery of sieves ranging from 0.2 to 10 mm for 10 minutes and weighing the residue from each sieve. The uniformity coefficient (UC) was determined by formula 2:

$$U.C = \left(\frac{D_{60}}{D_{10}}\right) \times 100 \tag{2}$$

where
    D_{60} = size (in mm) of screen opening where 60% of a sample passes and 40% is retained
    D_{10} = size (in mm) of screen opening where 10% of a sample passes and 90% is retained.

4.2 Biochar porosity

The biochar porosity (Φ) was determined using bulk and particle densities using formula 3 (Boris *et al*., 2018).

$$\Phi = 1 - \left(\frac{\rho_a}{\rho_s}\right) \tag{3}$$

where ρ_a = bulk density (g/cm^3), and ρ_s = particle density (g/cm^3).

4.3 Bulk and particle densities

Density and porosity are fundamental physical properties that control how biochar interacts with water. The bulk density (ρ_a) was determined according to NF X 31-503 (1992) by dividing the difference between the mass of the crucible filled with the sample and the mass of empty crucible with the crucible's volume. Particle density (ρ_s) was determined according to NF P 94-054 (1991). The principle was to measure the volume of grains excluding voids and to calculate the ratio between their weight and their volume.

4.4 Water content

The water content was determined by the gravimetric method at 105 °C (Pauwels *et al.,* 1992). Five grams of wet sample in a crucible and heated in a Carbolite Eurotherm furnace at 105 ° C for 5 hours. After cooling, water content was calculated using formula 4.

$$W\% = \left(\frac{m_w - m_d}{m_d}\right) \times 100 \tag{4}$$

where m_w = mass of the wet sample in grams (g) and m_d = mass of dry sample in grams (g).

5. Chemical characterization of biochars

5.1 Determination of ash content, electrical conductivity and pH(H₂O)

Ash refers to the inorganic residue remaining after either ignition or complete oxidation of organic matter in biomass. The ash content is a measure of the mineral content and other inorganic matter in biomass (National Renewable Energy Laboratory, 2008). Biochar's ash content can play a positive or negative role for the removal of heavy metals in water depending on its dominant ions (cations or anions). Ash content was determined using the dry incineration method (Pauwels *et al.,* 1992). Five grams of sample were placed in a crucible and heated in a Carbolite Eurotherm furnace at 450 ° C for 5 hours. After cooling, the dried sample was weighed. The ash content was calculated using formula 5:

$$\% Ash = \frac{m_1 - m_2}{m} \times 100 \tag{5}$$

where m_1 = mass (g) after incineration (sample + crucible), m_2 = mass (g) of the crucible and m = the original mass (g) of the sample.

The electrical conductivity and the pH (H_2O) were determined by the method described by Rajkovich *et al*. (2011).

5.2 Determination of total nitrogen

Total nitrogen content was determined according to the Kjeldahl method (Bremner, 1965). Organic nitrogen was completely mineralized with a mixture of concentrated sulfuric acid and hot salicylic acid. The mixture was distilled off and the ammonia generated was trapped by a solution of boric acid and later on titrated with a solution of sulfuric acid (0.01 N).

5.3 Organic Carbon (%)

Organic Carbon (%) was determined as described by Rodier *et al*. (2009). The organic carbon of biochar was oxidized by a solution of excess potassium dichromate ($K_2Cr_2O_7$), in a sulfuric acid medium. The excess potassium dichromate not reduced by organic carbon was then titrated with a solution of ferrous sulphate ($FeSO_4.7H_2O$) in the presence of diphenylamine which turns from purple to green at the equivalence point. Total carbon was determined by the sum of organic carbon and graphitic carbon. Nitrate was determined according to the Kjeldahl method (Bremner, 1965) by mixing 25 mL of sample with devada alloy followed by distillation and lastly titration with 0.01 N H_2SO_4 solution. The nitrite content was then determined from nitrate by using formula 6.

$$\left[NO_2^-\right] = 0.742 \times \left[NO_3^-\right] \tag{6}$$

5.4 Determination of chloride and phosphorus contents

Chloride content was determined by argentometric titration with silver nitrate in the presence of potassium chromate as an indicator as described by CEAEQ (2015) after extracting the sample with distilled water. The extraction of phosphorus was analogously to that described for chloride above. The determination of phosphorus in the filtrate was done by molecular absorption spectrophotometric method as described by Rodier *et al*. (2009).

5.5 Determination of metals

Calcium, Magnesium, Potassium, Sodium, Manganese, Lead, Iron, Copper, Aluminum and Zinc were determined by flame atomic absorption spectrophotometry using the brand Perkin-Elmer device, after mineralization of samples in the oven and their solubilization with acid. The samples were crushed and 10 g to 1/10 mg each was weighed and placed in an oven at 450 °C followed by dissolution in hydrochloric acid (0.05N HCl) and filtration of the solution. Calibration curves were then plotted with a range of different known concentrations for each metal. The wave length of each element was read and its concentration was calculated.

6. Statistical analysis

GenStat software was used for statistical analysis which consisted of comparing the means by analysis of variance (ANOVA). In cases of significant differences, the means were separated using the Smallest Significant Difference. The significance level for all analysis was 1%.

7. Results and discussion

7.1 Identification of wood species

Following the survey results, four species were selected: *Eucalyptus maideni*, *Milicia excelsa*, *Entandrophragma cylindricum* and *Ocotea michelsonie*. These were the four species commonly used (Figure 1). Thus, generating the highest quantities of sawdust in all the sawmills visited. From Figure 1, all the sawmills (100%) use the above-mentioned species and only 25, 15, 14 and 13% of them use other woods such as *Julbernardia Sarreti, Strombosia Schefflera, Abizia Girmmifera* and *Alstonia Boonni*

respectively. The choice for the high use of *Eucalyptus maideni*, *Milicia excelsa*, *Entandrophragma cylindricum* and *Ocotea michelsonie* was related to their high abundance and the high quality of timber resulting from these woods as testified by users.
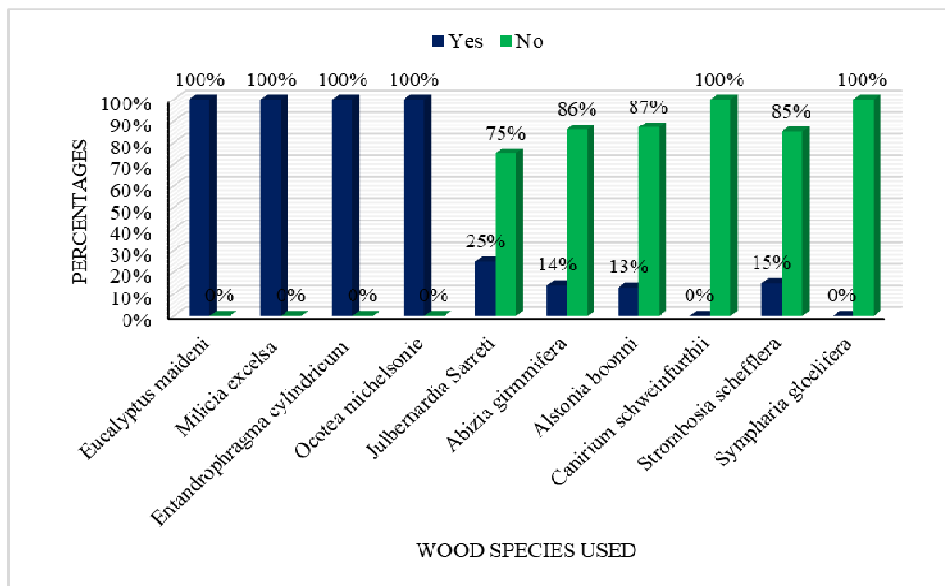

Figure 1: Percentages of wood species commonly used in Goma

7.2 Biochar production and yield

The parameters related to the production of biochar are summarized in Table 1

Table 1. Parameters related to biochar production

	Unit	*Eucalyptus maideni*	*Milicia excelsa*	*Entandrophragma Cylindricum*	*Ocotea michelsonie*
Temperature (T)	°C	524.28	519.67	529.11	522.67
Pyrolysis time(t)	H	3h30	3h30	3h30	3h30
Yield	%	31.4[a]	34.3[ab]	33.3[a]	36.6[ab]

Whenever the values of the characteristics of the biochars carry at least two different letters, there is significant difference between the averages.

All the biochars were obtained between 500 and 550 °C after 3h 30 minutes (Table 1). This was in order to optimize biochar's porosity and ash content. Temperatures were moderately high due to the thermal insulation of the furnace and the steady maintenance of the fire during the process. The process was thus a fast pyrolysis (Tripathiet *al*., 2016; Lee *et al.,* 2017). The yield values were 31.4% for EMB, 34.3% for MEB, 33.3% for ECB and 36.6% for OMB. The yield of EMB was significantly different from that of MEB and OMB and *Ocotea michelsonie* had the highest yield. The differences could be due to difference in density between the biochars (Boris *et al*., 2018). Also, higher yield for OMB could be due to a higher lignin content in *Ocotea michelsonie* wood as compared to others (Maga, 1986; Brewer *et al*., 2014). The yields were similar to those found by Niazi *et al.* (2017) for two biochars produced at 300 ° C and 700 °C but far below that reported by Lau *et al*. (2017). This was due to pyrolysis temperature which caused the loss of condensable materials and led to high ash content (Zama *et al*., 2017). Brewer *et al*. (2014) equally reported decrease in biochar with increasing biochar's production temperature. OH *et al.*(2012) argued that decrease in biochar yield with increasing pyrolysis temperature might be due to the destruction of chemical constituents, such as cellulose and hemicelluloses, included in the biomass.

7.3 Characteristics of biochars

Results of all the analysed physical and chemical parameters are summarized in Table 2

Table 2: Physical and chemical properties of biochar from some selected wood species

Parameter	Unit	*Eucalyptus Maideni* Biochar(EMB)	*Milicia excelsa* Biochar(MEB)	*Entandrophragma cylindricum* Biochar(ECB)	*Ocotea Michelsonie* Biochar(OMB)
		Physical properties			
Bulk density(ρ_a)	g.cm^{-3}	0.19[a]	0.18[a]	0.16[a]	0.17[a]
Particle density(ρ_s)	g.cm^{-3}	0.32[a]	0.50[c]	0.39[ab]	0.47[bc]
Porosity($_\Theta$)	%	94[a]	93[a]	92[a]	92[a]
uniformity Coefficient (UC)		3.2[a]	3.9[b]	4.3[c]	3.6[b]
Water content (W)	%	6[b]	8[c]	7[bc]	4[a]
		Chemical properties			
pH		7.3[b]	7.40[b]	7.70[a]	7.90[a]
Electrical Conductivity (EC)		0,04[a]	0,01[b]	0,07[a]	0,14[c]
Ash content	%	9.40[a]	12.30[b]	16.8[c]	18.40[d]
Total Carbon (C_{Tot})	%	54.30[a]	55.85[b]	54.80[ab]	55.60[b]
Organic Carbon (C_{org})	%	39.65[a]	39.10[a]	40.03[a]	32.72[c]
Total Nitrogen (TN)	mg/kg	3786.6	946.6	1437	3254
Chloride	mg/kg	792[a]	643[a]	703[b]	589[c]
Total Phosphorus	mg/kg	250.7[a]	280.3[b]	260.9[c]	230.1[d]
Ammonium	mg/kg	11.08[a]	5.50[b]	11.08[c]	12.94[d]
Nitrate	mg/kg	12.4[a]	9.92[b]	2.48[c]	7.44[d]
Nitrite	mg/kg	9.20[a]	7.41[b]	1.84[c]	5.52[d]
		Heavy metals			
Aluminium	mg/kg	<0,01	<0,01	<0,01	<0,01
Arsenic	mg/kg	<0,01	<0,01	<0,01	<0,01
Copper	mg/kg	<0,01	<0,01	<0,01	<0,01
Iron	mg/kg	642[a]	504[b]	680[c]	594[d]
Nickel	mg/kg	<0,025	<0,025	<0,025	<0,025
Lead	mg/kg	<0,01	<0,01	<0,01	<0,01
Zinc	mg/kg	3,21[a]	4,32[a]	4,41[b]	2,70[c]
		Exchangeable Bases			
Potassium	Cmol/kg	2,99[a]	6,860[b]	7,50a[b]	3,80[a]
Calcium	Cmol/kg	81,03[a]	81,79[b]	96,41[c]	108,87[d]
Magnésium	Cmol/kg	2,52[a]	2,70[b]	1,52[c]	1,80[d]
Sodium	Cmol/kg	0,52[a]	0,48[a]	0,44[ab]	0,56[b]

Whenever the values of the characteristics of the biochars carry at least two different letters, there is significant difference between the averages.

7.3.1 Physical properties

Bulk density ranged from 0.16 to 0.19 g.cm^{-3} with EMB having the highest value without any significant difference, while particle density ranged from 0.39 to 0.50 g.cm^{-3} with MEB having the highest value, significantly greater than those of EMB and ECB (Table 2). These values are lower than those reported in the literature by (Niazi *et al.,* 2017) and (Boris *et al*., 2018) and seemed to have been influenced by pyrolysis temperature and the type of wood material (Kaudal *et al*., 2015).

All the biochars had very high porosities ranging between 92 and 94% with EMB having the highest value. No significant difference in porosity was observed between biochars (p>0.05). High porosity was due to high temperature reached during the pyrolysis of biochars which led to the release of volatile matter and thus, creating more pores [Niazi *et al.,* 2017; Kaudal *et al*., 2015; NF EN 933-8, 2012). The high porosities suggest increased capacity of biochar to absorb certain contaminants from water implying that if the biochars obtained in this study are used as filter media, they will have better capacity to hold water in macropores as well as better capacity to grow biofilm in the pores, which is important for water treatment (Tomczyk *et al*., 2020).

The uniformity coefficient of MEB (4.3%) was greater than 4 suggesting that this biochar had a well-spread particle size without the predominance of a particular fraction. Those of ECB, EMB and OMB were 3.9%, 3.2% and 3.6% respectively Their grain sizes were poorly graded given that all the UC values were below 4 (Dalahmeh, 2016). These values are similar to those found by

(Boris *et al*., 2018). Water content also differed from one biochar to the other attributed to wood species (8% for MEB, 7% for ECB, 6% for EMB and 4% for OMB).

7.3.2 Chemical properties

The ash contents (9.4% for MEB, 12.3% for ECB, 16.8% for EMB and 18.40% for OMB) significantly differ from one biochar to the other and were very low compared to others encountered in literature (Yargicoglu *et al*., 2015; Zama *t al*., 2017). This proves a good characteristic of these biochars to purify water. These results are in accordance with the observations of Ronsse *et al*.(2013) and Li *et al*.(2017) who found that wood-based biochar particularly has high surface area and lower ash compared to other biochars, which might be due to the higher proportions of lignin, cellulose, and hemicellulose in the former than the latter.

pH ranged from 7.3 to 7.9 with OMB having the highest value and the biochars ranged from neutral to weakly alkaline without any significant difference. The values were similar to those of biochars produced at high temperatures reported by Rehrah *et al*.(2017). pH values obtained in this study were surely dependent on pyrolysis temperature. This is because biochar pH generally increases with pyrolysis temperature as a result of the loss volatile organic compounds and an increase in the basic cations in the biochar (NF EN 933-8, 2012; Mohan *et al*., 2014). The obtained biochars are thus suitable for water treatment as high pH promotes the deprotonation of functional groups on the surface of biochar. This creates negative charges that increase the attraction between the absorbent surface and adsorbing cations resulting in high adsorption capacity for some metals present in water (Sabry *et al*., 2019).

The values of electrical conductivities (0.07 for MEB, 0.01 for ECB, 0.04 for EMB and 0.14 for OMB) reflect the amounts of salts dissolved in the biochars. Electrical conductivities significantly differ between biochars and the highest value obtained for OMB could be attributed to the large amount of ash it contained (Dalahmeh, 2016; Boris *et al*., 2018). Total carbon (C_{Tot}) content of all biochars was greater than 50%. The large percentage of carbon was attributed to pyrolysis temperature due to organic matter (cellulose, lignin) decomposition that led to the formation of turbostratic and graphite carbons (NF EN 933-8, 2012). Organic carbon (C_{org}) ranged between 32.72 and 40.03% with ECB recording the highest value. The values were all below 50% suggesting the decomposition of organic matter due to pyrolysis temperature. However, these values are all above that obtained in Eucalyptus back under similar conditions by (Boris *et al*., 2018).

High total nitrogen (946.6-3786.6 mg/kg), Chloride (589-792 mg/kg), nitrate (2.48-12.40 mg/kg), ammonium (5.50-12.94 mg/kg), and nitrite (1.84-9.20 mg/kg) were also obtained in the biochars with EMB recording highest values (Table 2). High total nitrogen found in this study could be due to the fact that all nitrogen containing compounds were not completely carbonized. These results are in disagreement with the prediction of (Tomczyk *et al*., 2020) who stipulated that nitrogen is generally lost during pyrolysis, resulting to less than 6% nitrogen in biochars. Most heavy metals were below environmental threshold values except iron (504-680 mg/kg) with ECB having the highest value.

The presence of exchangeable bases in the biochars such as potassium (2.99 Cmol/kg for EMB, 6.860 Cmol/kg for MEB, 7.5 Cmol/kg for ECB, and 3.8/kg for OMB), Calcium (81.79 Cmol/kg for EMB, 81.71 Cmol/kg for MEB, 96.41 Cmol/kg for ECB, and 108.41 Cmol // kg for OMB), Magnesium (2.52 Cmol/kg for EMB, 2.7 Cmol/kg for MEB, 1.52 Cmol/kg for ECB, and 1.8 Cmol/kg for OMB), Sodium (0.58 Cmol/kg for EMB, 0.44 Cmol/kg for MEB, 0.48 Cmol/kg for ECB, and 0.56 Cmol/kg for OMB) could be explained by the percentage of ash contained in the biochars. This suggests that in contact with water containing heavy metals these bases will be exchanged for the latter, thus increasing biochars capacities in purifying the water. However, except calcium, the concentrations of the other bases were low due to the fact that they are temperature dependent, thus decreasing with increasing temperature (Mukherjee *et al*., 2011).

8. Conclusion

This research valorizes biomass generated from wood mill in the city of Goma which for the production of biochar which may serve as a potential water purifier thus, mitigating environmental pollution as well as providing sustainable portable water supply to households in Goma. Four wood species (*Eucalyptus maideni*, *Milicia excelsa*, *Entandrophragma cylindricum* and *Ocotea michelsonie*) are commonly used in Goma. The yield values were 34.3% for MEB, 33.3% for ECB, 31.4% for EMB and 36.6% for OMB. The values were slightly different from each other with *Ocotea michelsonie* having the highest yield. The high porosity of the biochars gives them a good capacity to absorb contaminants from water. In addition, the ability to remove pollutants in water by biochar from wood could be attributed to its characteristics such as high porosity, large area, pH, high ash content, and alkalinity. Results obtained may help to determine the appropriate conditions to have a good efficiency of removing contaminants from water using biochars obtained from wood sawdust in the city of Goma.

Acknowledgements

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