



## RESEARCH ARTICLE

**REVISED** **Physico-chemical properties of waste derived biochar from community scale faecal sludge treatment plants [version 2; peer review: 2 approved, 2 approved with reservations]**

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**Abstract**

**Background:** The dumping of untreated faecal sludge from non-sewered onsite sanitation facilities causes environmental pollution and exacerbates poor public health outcomes across developing nations. Long-term mechanisms to treat faecal sludge generated from these facilities are needed to resolve the global sanitation crisis and realize the Sustainable Development Goal (SDG) 6 “ensure availability and sustainable management of water and sanitation for all” by 2030. Pyrolysis of faecal sludge removes pathogens and generates biochar, which can be used as a soil enhancer.

**Methods:** The properties of faecal sludge biochars from three full-scale treatment plants in India were determined via Fourier transform infrared (FTIR) spectroscopy, scanning electron microscopy (SEM), energy dispersive x-ray (EDX) spectroscopy, crystal x-ray diffraction (XRD), proximate analyses, and BET surface area porosimetry.

**Results:** Results showed that all three biochars had low specific surface area, high alkaline pH values, high ash content, and negative surface charge. Fourier transform infrared spectra showed the same surface functional groups present in each biochar. X-ray diffraction analysis showed the mineral composition of each biochar differed slightly. Scanning electron microscopy analysis indicated a porous structure of each biochar with ash particles evident.

**Conclusions:** Slight differences in the ash content, surface area, pH and mineral content was observed between the three biochars.

**Keywords**

Biochar, Faecal Sludge, Fecal, Characterization, Properties, Pyrolysis, Sanitation

**Open Peer Review**

**Approval Status** ✓ ? ? ✓

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**REVISED Amendments from Version 1**

The article was accepted by the reviewer, only minor corrections have been made, some grammatical errors have been corrected, additional information in the introduction and conclusion has been supplied and methods section is clearer.

**Any further responses from the reviewers can be found at the end of the article**

**Article highlights**

- The physico-chemical properties of faecal sludge biochars from full-scale pyrolysis facilities were evaluated
- Biochars recorded very alkaline pH values, high ash content, low carbon content, low specific surface area
- Similar FTIR spectra indicated the same functional organic groups present on the biochars surface
- There were differences in ash content, pH and mineral content between the biochars

**Introduction**

Improving sanitation along with hygiene practices and access to safe water are essential for improving socioeconomic development and health globally. Inadequate sanitation facilities and lack of clean water are key factors in the contraction of diarrhoeal diseases world-wide leading to 1.6–2.5 million deaths every year (Kosek *et al.*, 2003).

In 2000 the international community set out eight Millennium Development Goals, a template aimed at tackling the needs of the world's most poverty stricken and underprivileged (Hutton & Bartram, 2008). Target 7C of the Environmental Sustainability Goal was to reduce by half the proportion of citizens “without sustainable access to safe drinking water and basic sanitation”. Since 2000, however, the proportion of the population in low and middle- income nations that use “unimproved” sanitation facilities has increased (WHO & UNICEF, 2017).

In 2015 the international community set out 17 new Sustainable Development Goals including Goal 6, to “ensure availability and sustainable management of water and sanitation for all” (UN, 2015). It is estimated that 3.6 billion people in the world still do not have access to safely managed sanitation facilities (UNICEF/WHO, 2021). Approximately 2.1 – 2.6 billion of these depend on onsite sanitation facilities (UNICEF & WHO, 2017) that generate vast quantities of untreated faecal sludge each day. In developing countries, faecal sludge (FS) collected from onsite sanitation facilities has been poorly managed, which has led to negative public and environmental health outcomes from eutrophication of surface water bodies, and contamination of groundwater and soils (Gwenzi & Munondo, 2008), and poor social and economic development (Haller *et al.*, 2007; Mara *et al.*, 2010). Long term and more sustainable solutions to deal with faecal sludge that don't involve expensive, water intensive and energy intensive sewer systems are needed.

Recent research has focused upon thermochemical treatment, with an emphasis on pyrolysis as a safe method of disposing of faecal sludge (Krueger *et al.*, 2020). Pyrolysis is the heating of biomass to temperatures of 350°C – 1000°C in an oxygen-free environment (European Biochar Foundation, 2016) which eliminates harmful pathogenic organisms within the sludge. Carbon-rich biochar produced from pyrolysis does not readily burn like charcoal (Crombie *et al.*, 2013), is safe to handle, and has been demonstrated to be an important soil amendment (Chan *et al.*, 2007). The original feedstock source, pyrolysis temperature, hold time, and heating rate are the main factors determining the characteristics of biochars (Chen *et al.*, 2008; Crombie *et al.*, 2015; Lehmann & Joseph, 2012; Tomczyk *et al.*, 2020; Weber & Quicker, 2018).

The theory behind the utilization of biochar to improve soil fertility and increase crop yield originated from observations made on the Amazonian Black Earth (*Terra Preta*). *Terra Preta* is a specific type of very dark, fertile soil discovered in the Amazon basin, containing higher nutrient levels and higher organic carbon content than the surrounding soils which are generally low in fertility (Glaser *et al.*, 2001).

There are multiple benefits to adding biochar to soil aside from improving carbon content and nutrient levels. Surface functional groups on the surface of biochar can lead to an increase in the cation exchange capacity of the soil (CEC) (Glaser *et al.*, 2001); the microporous structure of biochar can increase the water holding capacity of the soil (Gaskin *et al.*, 2007), and alkaline biochars can increase pH levels in acidic soil (Novak *et al.*, 2009).

Biochar addition to soil has also been shown to reduce the bio-availability of heavy metals in soils (Park *et al.*, 2011; Uchimiya *et al.*, 2011; Zhang *et al.*, 2013) including that of wastewater sludge biochar (Hossain *et al.*, 2010) and sewage sludge biochar (Méndez *et al.*, 2012). Biochar addition also reduces plant uptake of pesticides (Yu *et al.*, 2009) and reduces the leaching of applied pesticides which can impact underground water contamination (Ahmad *et al.*, 2014). Changes in soil microbial properties upon biochar addition have also been reported (Lehmann *et al.*, 2011), including alteration of soil microbial community structure (Farrell *et al.*, 2013), and an increase in microbial abundance (Awad *et al.*, 2018).

There is a considerable amount of research investigating characteristics of sewage sludge-derived biochar but less on faecal sludge biochar (Gold *et al.*, 2018). Many studies exist on the properties of faecal sludge itself (Awere *et al.*, 2020; Bassan *et al.*, 2013; Fanyin-Martin *et al.*, 2017; Lama *et al.*, 2022; Schoebitz *et al.*, 2014; Septien *et al.*, 2020). Only a handful of articles examine properties of faecal sludge biochar and these studies have a diverse range of objectives including biochar as a soil amendment to increase lettuce yields (Woldetsadik *et al.*, 2018), biochars solid fuel characteristics (Krueger *et al.*, 2020), cadmium adsorption by biochar (Koetlisi & Muchaonyerwa, 2017), recovery of ammonium in urine by biochar (Bai *et al.*, 2018) and energy balance analysis of slow pyrolysis of human manure (Liu *et al.*, 2014).

Most of the research into faecal sludge-derived biochar has focused on characterization of small-scale laboratory-produced biochar (Bleuler *et al.*, 2021; Gold *et al.*, 2018; Liu *et al.*, 2014; Woldetsadik *et al.*, 2018) while data from full-scale operations are very limited (Krueger *et al.*, 2020). Investigating the feasibility of resource recovery of operational up-scaled sludge treatment technologies and production of FS biochar with consistent properties is imperative to alleviate the sanitation crisis (Andriessen *et al.*, 2019; Strande *et al.*, 2014). Krueger *et al.* (2020) investigated the physico-chemical properties of full-scale faecal sludge biochars from treatment plants in Warangal and Narsapur, India. They focused on solid fuel properties of biochar, particle size distribution and heavy metal concentration. Heavy metal concentrations were found to be within the limits for land application set out by the EU (EEC, 1986) and the International Biochar Initiative (IBI, 2015) apart from the Narsapur biochar which contained concentrations of lead over the IBI stated threshold.

The objective of this investigation was to assess the uniformity of biochar characteristics produced from three full-scale faecal sludge treatment plants in Wai, Warangal and Narsapur, India. This study focused more on physico-chemical properties that would contribute to biochars end-use as a soil amendment. The biochar properties determined were ash content, pH, carbon content, organic surface groups, surface charge, mineral content, pore volume, and specific surface area.

## Methods

### Biochar preparation

The faecal feedstocks for the preparation of the biochars used in this study were sourced from three different faecal sludge and septage processors in India: Narsapur in Andhra Pradesh, Warangal in Telangana and Wai, Maharashtra (Figure 2). Warangal and Narsapur treatment plants currently have a capacity of 15m<sup>3</sup> per day, whereas the Wai treatment plant has a capacity of 70 m<sup>3</sup> per day. FS collected from septic tanks

is delivered to each processing plant where it is stored in holding tanks for the homogenization of the sludge. Tide Technocrats Private Limited have several community scale faecal sludges and septage processors which sanitize faecal waste and dewater the sludge (5–10% moisture content) using solar energy in drying beds. Solar drying was managed on-site and expedited by spreading the sludge in a 10 mm layer. The sludge was pyrolyzed into biochar using a flame temperature operating range of 550–750°C. The process relies on autothermal operation, thus a limited supply of oxygen flows through an air fan into the main reaction chamber to allow for partial oxidation. process at the community scale faecal sludge and septage processors are outlined in Figure 1. Three 5kg biochar samples were collected from each processor in September 2018.

### Characterization of biochars

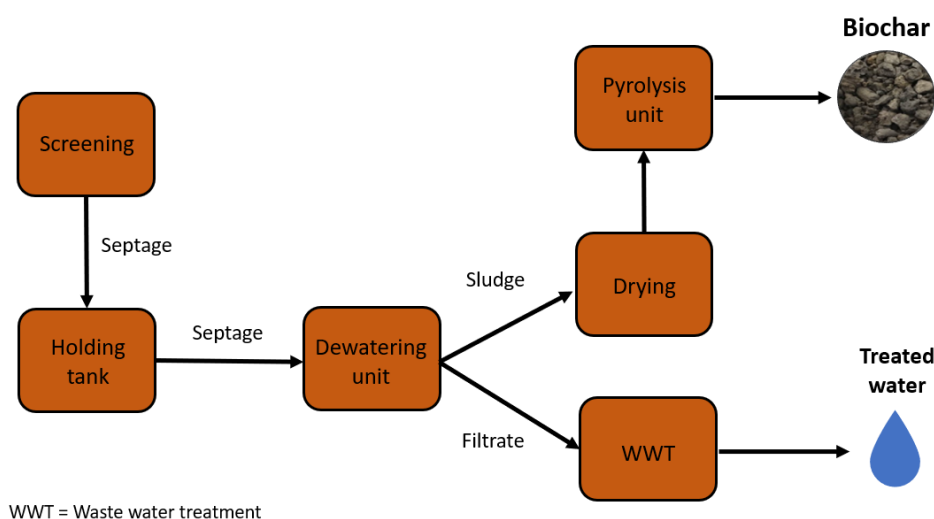
The biochars characterized were collected from the pyrolyzer.

**Chemical analysis.** Elemental C, N, S and H abundances were determined at Environmental Geosciences, University of Vienna, Austria using an elemental analyzer (Vario MACRO, Elementar).

The elemental composition (C, H, N, S and O) and ash content of the biochars were used to calculate the molar element ratios H/C, C/N, and O/C. The amount of oxygen in the samples was calculated from the subtraction of total percentage carbon, hydrogen, nitrogen, and sulphur and ash content from 100 (Castan *et al.*, 2019).

**Proximate analyses.** Moisture and ash content of the three biochars were determined in triplicate by methods adapted from the literature (ASTM D 1762-84, 2011; Enders *et al.*, 2012).

Crucibles and covers were cleaned by heating at 750°C for 6 hours and then cooling to 105°C. This procedure volatilized residual material on the crucibles. The crucibles were



**Figure 1.** Flow diagram of waste through a Tide Technocrats Community Scale Faecal Sludge and Septage Processor adapted from (Tide Technocrats, n.d).



**Figure 2.** Photo of the community scale faecal sludge and septage treatment plant in Wai, Maharashtra (Tide Technocrats, n.d.)

transferred to desiccators and cooled to ambient temperature. The mass of crucibles and crucible covers were recorded to 0.1mg and masses determined for all samples. Approximately 1.0g of biochar was added into each crucible. For the moisture determination the crucibles and covers were heated at 105°C for 18 hours and then transferred to desiccators whilst hot. The covers were removed briefly in order to safely remove the crucibles and covers from the oven. After cooling to ambient temperature, the mass of crucibles, covers and sample were recorded to 0.1mg for all samples.

For ash determination the covered crucibles with 105°C dry biochar was placed in the furnace. The covers were adjusted so that they were askew to allow air flow into the crucibles, while reducing the possibility of physical losses. The samples were heated from ambient to 750°C at a rate of 2°C per minute. The furnace was programmed to hold the temperature at 750°C for 6 hours then allowed to cool down to ~130°C. Crucible lids were adjusted to sit flush when the temperature of 105°C was reached. The crucibles were then removed from the furnace before placing in desiccators and left to cool to ambient temperature. The mass of each crucible and crucible cover with sample was recorded to 0.1mg.

Chars were ground to <850µm in a pestle and mortar to enhance representativeness of the sample and sieved to >149µm as this lessens physical losses upon rapid heating (Enders & Lehmann, 2015).

**pH and electrical conductivity.** The pH of biochar samples was measured by suspending 5.0g (ground to <2mm) biochar in deionised water in a 1:10 ratio (Singh *et al.*, 2017). After 1 hour

of shaking, suspensions were allowed to stand for 30 minutes before pH measurements were taken using a Voltcraft soil pH meter calibrated using pH 7 and pH 10 buffers. Electrical conductivity (EC) was measured on the same samples using a calibrated Whatman CDM 400 EC meter. The analyses of pH and EC were performed in triplicate.

**FT-IR analysis.** Fourier transform infrared (FTIR) spectra were used to identify the surface organic functional groups present in the biochar. High ash content in sludge-derived biochars leads to a high mineral content with bands in the infrared spectrum arising at similar wavenumbers to organic functional groups. To elucidate the different groups present, FTIR spectra of ashed biochars and de-ashed (acid washed) biochars were also generated.

Acid washing biochar to remove ash content (Klasson *et al.*, 2009; Lima *et al.*, 2016; Thomas Klasson *et al.*, 2014) was achieved with 0.1 M HCl at a ratio of approximately 50:1 (v/w). Samples were shaken in a Uniwist 400 at 180 rpm for 2 hours before being filtered and washed with deionised water until a pH of 7 was reached. Samples were oven-dried at 80°C overnight.

The samples were gently ground using a pestle and mortar and analyzed using a Perkin Elmer Spectrum 2 FTIR spectrophotometer applying the Attenuated Total Reflectance (ATR) method with a diamond crystal. The resulting spectra were an average of 16 scans obtained in the range from 400 to 4000 cm<sup>-1</sup> with a spectral resolution of 2 cm<sup>-1</sup> for biochars and 4 cm<sup>-1</sup> for acid washed and ashed biochars.

**Surface area.** The BET (Brunauer, Emmett, and Teller) method is frequently used to determine the total surface area and pore size of materials. The BET analysis was conducted using the NOVA 2200e surface area and pore size analyzer (Quantachrome Instruments). The BET specific surface area of the three biochar samples were determined using two methods: N<sub>2</sub> as adsorptive gas at 77 K and CO<sub>2</sub> at 273 K.

Prior to these measurements, 200mg – 300mg of biochar (<2mm) were heated to 130°C under vacuum for a minimum of 4 hours. Then the samples were transferred to the instrument and out-gassed at 105°C for a minimum of 4 hours following standard protocols. Samples were analyzed in triplicates.

For N<sub>2</sub> isotherms and CO<sub>2</sub> isotherms the BET equation was used to determine the specific surface areas from six points in the pressure region  $P/P_0 = 0.01-0.30$  (Brunauer *et al.*, 1938). For N<sub>2</sub> the pore size-distributions in the pressure region  $P/P_0 = 0.01-0.98$  were ascertained using the built-in Density Functional Theory (DFT) model assuming slit-like pores. DFT considers micropore filling process, the development of the adsorbed film thickness, and importantly capillary condensation and evaporation, thus it can model hysteresis in the adsorption/desorption mesopore region of the isotherm.

For CO<sub>2</sub> isotherms the pore size-distribution, the cumulative pore volume (μPV) and the cumulative surface area (μSSA) in the pressure region  $P/P_0 = 0.001-0.030$  were determined using the built-in Grand Canonical Monte Carlo (GCMC) simulation, again, assuming pores were slit-shaped.

The benefit of using of DFT and Monte Carlo simulation methods is that they provide a combined micro-mesopore analysis.

**Measurement of zeta potential.** Zeta potential measurements were undertaken according to methods reported in the literature (Samsuri *et al.*, 2013; Yuan *et al.*, 2011). The zeta potential values were determined by weighing 0.045g of 63μm sieved biochar into a 250ml conical flask and adding 180 mL of 0.1M NaCl solution to each flask. Five suspensions were prepared for each biochar at pH values between 5.0 – 9.0 with the pH of each suspension adjusted using HCl. Suspensions were then dispersed ultrasonically for 30 minutes at  $30 \pm 1$  °C in a bath-type sonicator at a frequency of 40 kHz and a power of 300 W. The samples were then left to stand for 72 hours before being measured with a Malvern Zeta Sizer Nano. In total 15 suspensions were prepared and each suspension was measured a minimum of three times.

**X-ray diffraction (XRD).** The X-ray diffraction (XRD) analysis of the chars was conducted on a Bruker D8 Discover XRD. This was operated at 40 kV and 40 mA and the data collected over a  $2\theta$  range of 20–70° using the Cu-K $\alpha$  radiation at a scan rate of 2° min<sup>-1</sup>. The main phase peaks were identified by comparing the observed XRD patterns to the standards compiled by the Crystallography Open Database (COD) (Downs & Hall-Wallace, 2003; Gražulis *et al.*, 2009; Gražulis *et al.*, 2015; Gražulis *et al.*, 2012; Merkys *et al.*, 2016; Quirós *et al.*, 2018).

**SEM/EDX.** SEM–EDX analysis offers detailed imaging data about the morphology and surface texture of individual particles, with characterization of the elemental composition of the analyzed volume. Scanning electron microscope (SEM) analysis was performed using a Hitachi TM3000 SEM fitted with a Bruker X-ray energy dispersive spectrometry (EDS). The two modes of operation in SEM analysis utilized here were backscattered electron imaging (BSE) and energy dispersive x-ray EDX. Biochar particles used were in the size range 150 μm– 850 μm. Prior to analysis samples were spread onto double-sided carbon tape and mounted on a SEM stub.

**Cation exchange capacity.** Cation exchange capacity measurements were performed in University of Santiago de Compostela, Spain, by the summation method of the exchangeable base cations of Ca, Mg, Na, K, and Al. NH<sub>4</sub>Cl 1M (25ml) was added to the biochar sample (5g) and shaken manually before being left to stand overnight (16 hours). The following day, 75 ml of NH<sub>4</sub>Cl 1M was added and then filtered using quantitative, low ash filter paper. (Peech *et al.*, 1947). Ca, Mg and Al were measured by PerkinElmer PinAAcle 500 Atomic Absorption Spectrometer and Na, K, were measured by Atomic Absorption Spectrophotometer with an Emission Flame.

### Statistical analysis

Electrical conductivity, pH, moisture content, ash content, CEC, surface area and pore volume measurements were all performed in triplicate and experimental uncertainty given by standard deviation.

## Results

### Proximate analyses EC, pH and elemental analyses

All biochars collected from the pyrolyzer had high ash contents (Nicholas, 2022). Warangal biochar (WGL\_BC) recorded the highest ash content at 88.3% and Narsapur biochar (NSP\_BC) and Wai biochar (WAI\_BC) had lower ash contents at 67.0% and 62.3% respectively (Table 1). Warangal biochar (WGL\_BC) also had the lowest moisture content at 0.98% in comparison with 2.15% and 3.08% for Narsapur biochar (NSP\_BC) and Wai biochar (WAI\_BC) respectively. Measured pH values were high for all three biochars (11.86 – 12.45). The measured high ash content is consistent with the literature (Gold *et al.*, 2018; Koetlisi & Muchaonyerwa, 2017; Liu *et al.*, 2014). The initial feedstock of sewage sludge is high in ash and sewage sludges have been found to contain very high concentrations of Si (19–58%), Ca (5.1–7.4%), and P (3.4–4.9%) (Zielińska *et al.*, 2015). Ash content of faecal sludge is also high and has been measured at 17.0 wt.%, significantly higher than measured ash content of sawdust at 0.8% (Liu *et al.*, 2014). Ash content of faecal sludge has been found to be higher than that of sewage sludge (Koetlisi & Muchaonyerwa, 2017). Several reasons for this high ash content have been suggested including loss of volatile solids from latrine waste due to long storage times in the latrine (Zuma *et al.*, 2015), ingress of grit and sand from poorly lined containment structures (Niwagaba *et al.*, 2014) and in community toilets a higher rate of disposal of polluting waste can lead to higher ash contents (Barani *et al.*, 2018).

Increases in pH due to increases in ash content in biochars derived from sewage sludge feedstocks have been previously reported (Hossain *et al.*, 2011; Liu *et al.*, 2014). The general alkaline character of biochar likely results from the increase in quantities of alkali salts (Na, K) and salts of alkaline elements (Ca, Mg) during the pyrolysis process (Singh *et al.*, 2010).

WGL\_BC recorded the most alkaline pH value (12.45) and the largest EC value (Table 1.) which is due to the higher ash content recorded for WGL\_BC (Rehrah *et al.*, 2014). There could be several reasons why the WGL biochar had a significantly higher ash content. Digestion during storage in onsite sanitation technologies can play a part in the high ash content of FS biochar (Gold *et al.*, 2018) as well as contamination of FS by sand and grit caused by poorly lined containment structures (Niwegaba *et al.*, 2014). A recent study investigating biochar from the same treatment facilities in India observed that sintered mineral depositions had to be removed from the reactor on a weekly basis (Krueger *et al.*, 2020). Therefore, it is likely that ash concentrations from biochars from these types of treatment plants will fluctuate over time.

**Table 1. Proximate analyses, elemental analyses, pH, EC and surface area measurements of faecal sludge biochars.**

(EC = Electrical Conductivity, C= Carbon, N= Nitrogen, S= Sulphur, Oxygen, SBET = Surface area measured by BET, TPV = Total pore volume, SSA = Specific Surface area, CEC=Cation Exchange Capacity).

Parameter	Unit	WAI BC	NSP BC	WGL BC
pH	[ ]	11.81 ± 0.01	11.82 ± 0.01	12.45 ± 0.01
EC	[mS.cm <sup>-1</sup> ]	2.70 ± 0.09	1.79 ± 0.17	9.00 ± 0.02
Moisture	[%]	3.08 ± 0.01	2.15 ± 0.31	0.98 ± 0.05
Ash	[%]	62.3 ± 0.32	67.0 ± 2.68	88.3 ± 0.21
C	[%]	21.11	23.79	8.06
N	[%]	1.32	1.13	0.37
H	[%]	1.55	0.73	1.15
S	[%]	0.03	0.27	0.03
O	[%]	13.69	7.08	2.09
H/C	[ ]	0.9	0.4	1.7
C/N	[ ]	18.7	24.6	25.4
O/C	[ ]	0.5	0.2	0.2
S <sub>BET</sub> N <sub>2</sub>	[m <sup>2</sup> .g <sup>-1</sup> ]	3.52 ± 0.78	3.69 ± 0.36	12.07 ± 4.12
N <sub>2</sub> TPV	[cm <sup>3</sup> .g <sup>-1</sup> ]	0.011	0.011	0.019
S <sub>BET</sub> CO <sub>2</sub>	[m <sup>2</sup> .g <sup>-1</sup> ]	46.72 ± 7.0	74.20 ± 4.0	26.11 ± 2.6
CO <sub>2</sub> μSSA	[m <sup>2</sup> .g <sup>-1</sup> ]	63.49 ± 8.3	99.62 ± 4.5	36.76 ± 3.0
CO <sub>2</sub> μPV	[cm <sup>3</sup> .g <sup>-1</sup> ]	0.017	0.027	0.010
CEC	[cmol.kg <sup>-1</sup> ]	90.0 ± 6.5	41.9 ± 2.2	129.3 ± 2.3

A high ash content of biochar could be useful with regards to its end-use a soil amendment. Increased crop growth with a highly alkaline (12.1), high ash biochar treatment of acidic soil has been previously reported (Smider & Singh, 2014) The authors deemed this was a result of the release of nutrients from the ash in the biochar itself and the biochar's liming effect. It has been proposed that this liming effect is one of the main processes influencing the enhanced plant growth seen on biochar addition to soils (Jeffery *et al.*, 2011). Altering soil pH is one of several mechanisms by which biochar can improve soils and increase agricultural productivity. Therefore, highly alkaline biochars could be of benefit to acidic soils are responsible for the severe limitation of crop agriculture worldwide. Currently only a small fraction of acidic soil is used for arable crops globally but approximately 50% of the earth's potential arable lands are acidic (von Uexküll & Mutert, 1995).

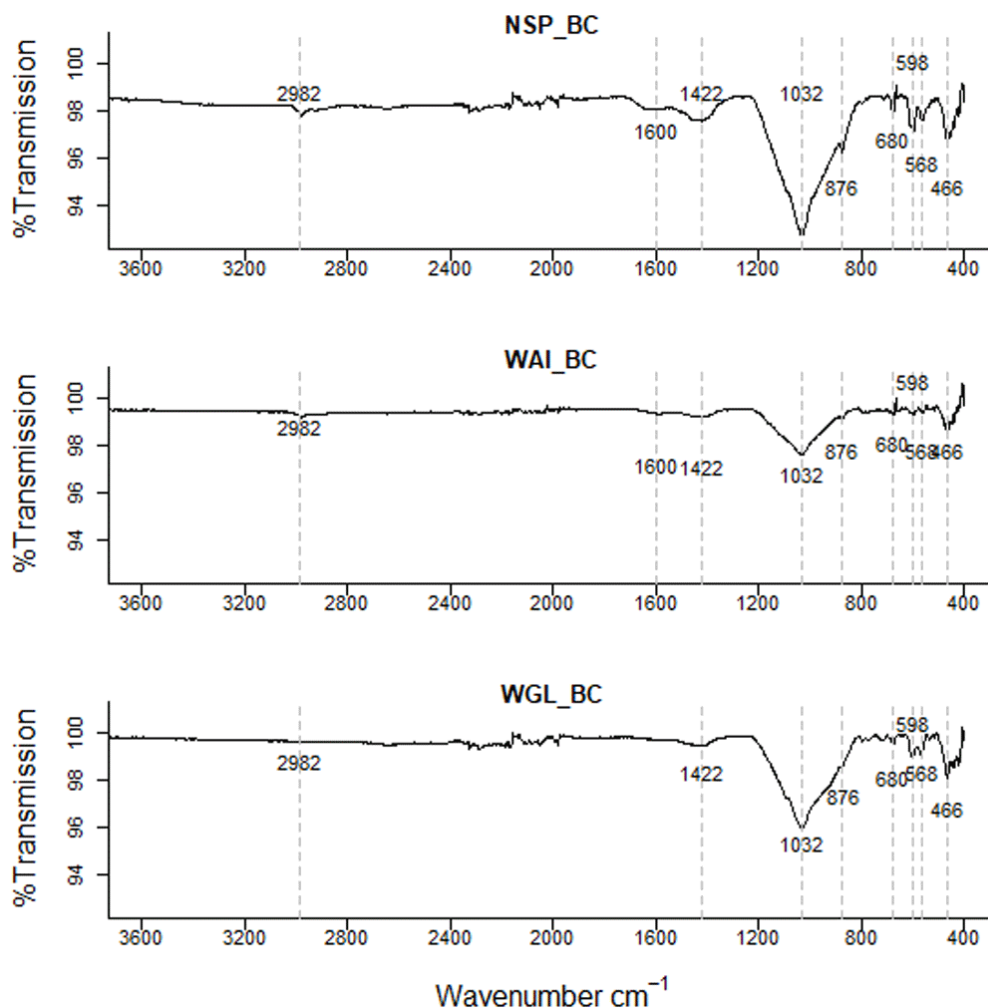
The elemental composition (Table 1.) shows a relatively low percentage of carbon within the samples, 21–23% for NSP\_BC and WAI\_BC and a very low 8% for WGL\_BC which is consistent with the measured ash content. Pyrolysis generally concentrates carbon in the biochar with an increase in C content relative to the feedstock frequently reported. However, most studies on sewage sludge (SS) –derived biochar show a decrease in the percentage of C in the final product relative to the feedstock (Agrafioti *et al.*, 2013; Khan *et al.*, 2013). FS- and SS-derived biochars generally have low total C concentrations in comparison with cellulose derived biochars (Tomczyk *et al.*, 2020). This is due to the high ash content in the original feedstock of faecal and sewage sludge. The measured carbon concentrations in these biochars are consistent with carbon contents reported in the literature for faecal sludge biochar 27.4– 34.9% (Gold *et al.*, 2018), 17.2 – 34.1% (Krueger *et al.*, 2020), 6.5 – 11.1% (Koetlisi & Muchaonyerwa, 2017), and 19.5% (Woldetsadik *et al.*, 2018).

#### Fourier transform infrared (FTIR) spectroscopy

FTIR spectra indicated that all three sludge biochars have a complex chemical bond structure with both organic matter and mineral compounds evident within the biochar. The FTIR spectra of all three biochars were similar with the same functional groups present on the surface; an indication of the homogenous nature of faecal sludge (Figure 3).

High ash content in sludge-derived biochars leads to a high mineral content with bands arising on the spectrum at similar wavenumbers to organic functional groups. For example, a broad peak in the 1000–1200cm<sup>-1</sup> region can arise due to several functional groups such as inorganic and organic silicon, phosphorus compounds, as well as C-O stretching and sulphate groups (Coates, 2004)

Low intensity peaks evident in the 3800cm<sup>-1</sup> –3600cm<sup>-1</sup> region relate to OH group vibrations within mineral matter (Hossain *et al.*, 2011) which indicates the presence of clay type compounds within the biochar (Table 2). Two peaks at 2980cm<sup>-1</sup> and 2890cm<sup>-1</sup> indicate asymmetric and symmetric aliphatic ν(CH) from terminal –CH<sub>3</sub> groups respectively (Socrates, 2001).



**Figure 3.** Fourier-transform infrared spectra (FTIR) of the three biochars NSP, WGL and WAI with dotted lines representing the main absorption (cm<sup>-1</sup>) peaks.

However, these CH bands disappear at high temperatures due to demethylation and dehydration (Zhang *et al.*, 2015) therefore in biochar pyrolyzed at 550 – 750°C the peaks are negligible.

Small peaks in the 2700–2100 region could be due to P-OH groups in phosphorus acids and esters which produce one or two broad bands (Stuart, 2004).

A peak at 1424cm<sup>-1</sup> in the biochar spectra (Figure 3.) corresponds to asymmetric stretches of carbonate groups, which correlates with the small peak at 874 cm<sup>-1</sup> due to the out-of-plane bending for CO<sub>3</sub><sup>2-</sup> (Zhao *et al.*, 2013). This could indicate the presence of calcite (calcium carbonate) in the sample. The presence of carbonate was verified as the FTIR spectrum of the acid washed biochar showed no clear peaks at 1424 cm<sup>-1</sup> or 874 cm<sup>-1</sup> confirming that acid washing removed carbonates from the sample (Figure 5.).

Another interesting difference between the ashed (Figure 4.) biochars and deashed biochars is a broad trough between 3400cm<sup>-1</sup> and 2500 cm<sup>-1</sup>. This implies the presence of O-H in

carboxylic acids however there is only a very weak intensity peak at ~1700 cm<sup>-1</sup> which could correspond to C=O in carboxylic acids. Other possible groups responsible for peaks within the 3400cm<sup>-1</sup> and 2500 cm<sup>-1</sup> region include ν(OH) from sorbed water and hydrogen bonded OH (Keiluweit *et al.*, 2010). The low intensity peak in biochar between 1540 and 1650 could be indicative of C=O stretching vibrations for amides (Calderón *et al.*, 2006), aromatic C=C stretching and carboxylate anion vibrations (Deacon & Phillips, 1980). The peak in the deashed biochar at 1580 cm<sup>-1</sup> to 1600 cm<sup>-1</sup> is indicative of a carboxylate ion, the conjugate base of a carboxylic acid (Deacon & Phillips, 1980; Ellerbrock & Gerke, 2021).

This peak was not evident in the ashed biochar (Figure 4.). It's been suggested that a reduction in inorganics by acid demineralization allows previously hidden carbon to emerge so increasing the amount of acidic functional groups (Lou *et al.*, 2011). In the ashed biochar there are very visible peaks ~1450cm<sup>-1</sup> indicative of a carbonate stretch (CO<sub>3</sub><sup>2-</sup>) whereas as the peaks in the acid washed samples are much less visible indicating some carbonate salts within the ash content have been successfully removed by acid demineralization.



**Table 2. Proposed band assignments of the FTIR spectra of biochar.**

Wavenumbers (cm <sup>-1</sup> )	Characteristic vibrations	Reference
3670 - 3650	v(OH) from non-hydrogen bonded O-H groups	(Sharma <i>et al.</i> , 2004)
3600 - 3200	v(OH) from sorbed water and hydrogen-bonded biochar O-H groups	(Keiluweit <i>et al.</i> , 2010)
~2980	2990-2950 cm <sup>-1</sup> asymmetric aliphatic v(CH) from terminal -CH <sub>3</sub> groups	(Socrates, 2001)
~2890	2870-2890 cm <sup>-1</sup> symmetric aliphatic v(CH) from terminal -CH <sub>3</sub> groups	(Socrates, 2001)
2700-2100	P-OH groups produce one or two broad bands in the 2700 -2100 region 2100 - 2250cm <sup>-1</sup> C≡C bonds 2100 - 2360cm <sup>-1</sup> Silane Si-H 2100 - 2270cm <sup>-1</sup> Dimides, Azides and Ketenes	(Stuart, 2004)
1700	v(C=O) from carboxylic acids amides, esters and ketones 1740- 1650	(Socrates, 2001)
1540 - 1650	C=O stretching vibrations for amides, aromatic C=C stretching and carboxylate anion vibrations.	(Calderón <i>et al.</i> , 2006)
1580 - 1600	vibration of C=C bonds	(Davis <i>et al.</i> , 1999)
1424	Carbonate (ν <sub>3</sub> ; asymmetric stretch)	(Socrates, 2001)
1200- 950	P-O (asymmetric and symmetric stretching of PO <sub>2</sub> and P(OH) <sub>2</sub> in phosphate)	(Jiang <i>et al.</i> , 2004)
1100-1000	Si-O-Si asymmetric stretching	(Falaras, 1999)
1020 - 1030	C-O stretching of ethers and primary amine C-N stretches	(Keiluweit <i>et al.</i> , 2010) (Claoston <i>et al.</i> , 2014)
~875	Out-of-plane bending for CO <sub>3</sub> <sup>2-</sup> and - v(M-O-H) O-H bending bands from clay minerals associated with biochar	(Zhao <i>et al.</i> , 2013) (Farmer, 1974)
796 and 780	quartz doublet	(Farmer, 1974)
462-464	Si-O-Si	(Qian & Chen, 2013)
452	Si-O rocking	Shahrokh Abadi <i>et al.</i> , 2015

The very broad band in the range 1200–970cm<sup>-1</sup> is indicative of several functional groups. Inorganic and organic silicon and phosphorus compounds, as well as carbohydrates and sulphates can contribute to this broad peak (Wen *et al.*, 2007). Sewage chars are known to contain high phosphorus levels suggesting that the peaks observed in 1200–950cm<sup>-1</sup> band arise from P containing functional groups such as asymmetric and symmetric stretching of PO<sub>2</sub> and P(OH)<sub>2</sub> in phosphate (Jiang *et al.*, 2004). Si-O asymmetric stretching could also be present between 1000–1100cm<sup>-1</sup> (Falaras, 1999) as well as symmetric C-O stretching of ethers.

A peak at 462–464 cm<sup>-1</sup> evident in both biochar and acid washed biochar is indicative of bending vibration of Si-O-Si (459–463 cm<sup>-1</sup>) (Qian & Chen, 2013). In the ashed biochar this peak seems to shift to a lower wavenumber 456cm<sup>-1</sup>. It is possible the signals at 462–464 cm<sup>-1</sup> relate to bending vibration of Si-O-Si (459–463 cm<sup>-1</sup>) and the signal at lower wavelength in the ashed biochar at 452cm<sup>-1</sup> relates to Si-O rocking (Shahrokh Abadi *et al.*, 2015). A weak intensity signal at 1984 cm<sup>-1</sup> is evident in the ashed biochar but not in the deashed samples. This signal could indicate metal – carbonyl bonds, typically terminal M-CO bonds occur at 2125 - 1850 cm<sup>-1</sup>. A

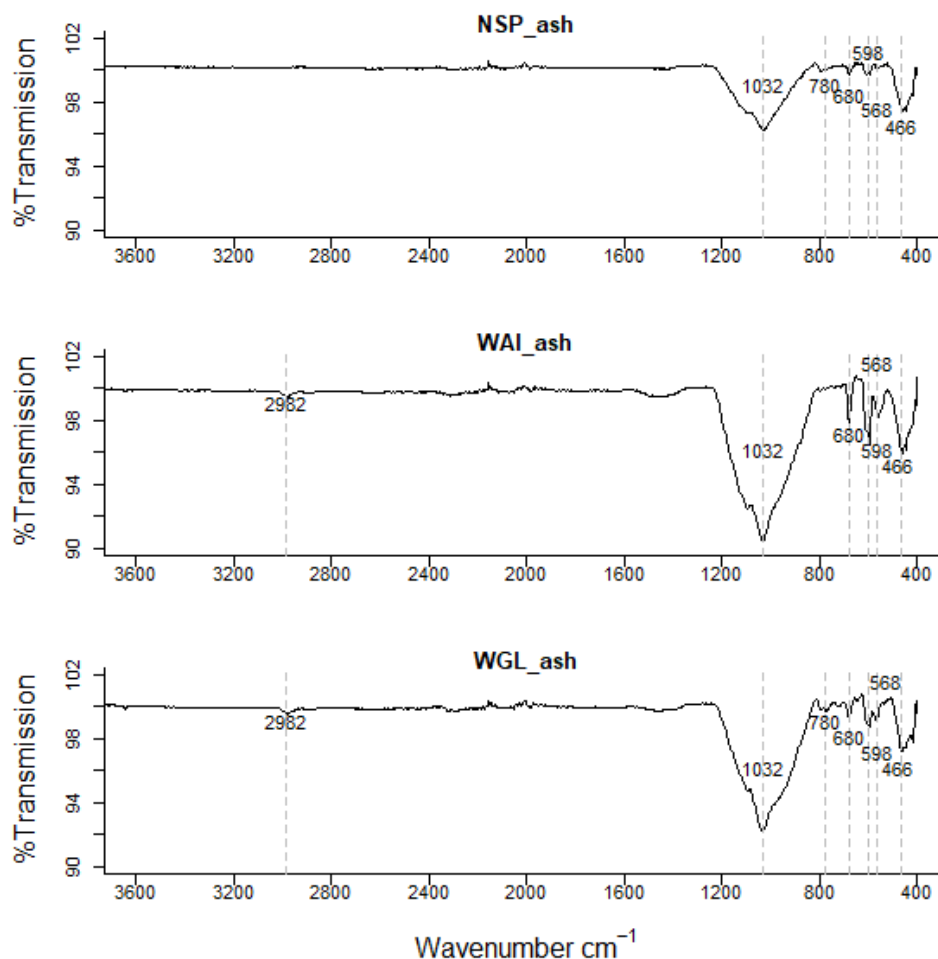
quartz doublet at 796cm<sup>-1</sup> and 780cm<sup>-1</sup> is evident in the ashed biochar sample (Farmer, 1974).

There are more signals recorded in the 900-400 cm<sup>-1</sup> region for the ashed biochar than the deashed biochar which relate to clay minerals associated with biochar. Bands below 600 cm<sup>-1</sup> can be caused by stretching inorganic compounds such as KCl and CaCl<sub>2</sub> (Hossain *et al.*, 2011).

The oxygen containing functional groups (OCFGs) present on biochars surface such as C=O groups determine its cation exchange capacity (CEC) (Banik *et al.*, 2018). It is this property that enables biochars to adsorb cationic nutrients such as NH<sub>4</sub><sup>+</sup>, Ca<sup>2+</sup>, K<sup>+</sup> within the soil and increases soils nutrient retention capability. The lack of C=O groups present in WGL\_BC could affect its ability to retain nutrients and therefore its suitability as a soil amendment.

#### Surface area

The shape of the isotherms indicate a Type II isotherm, however, Type II isotherms are generally typified by a lack of hysteresis and no saturation at P/P<sub>0</sub> near to 1; typical of nonporous and macroporous adsorbents (Thommes *et al.*, 2015).



**Figure 4.** FTIR spectra of ashed NSP biochar, ashed WAI biochar, and ashed WGL biochar.

A deviation from a true Type II isotherm can be described as a pseudo-type II isotherm. These isotherms are associated with delayed capillary condensation due to the small degree of pore curvature and non-rigidity of the aggregate structure of the adsorbent. (Sing & Williams, 2004).

Hysteresis is present in all isotherms and can be classified as either type H3 hysteresis loop or type H4 hysteresis loop according to International Union of Pure and Applied Chemistry (Thommes *et al.*, 2015). Hysteresis is caused by capillary condensation and is typical of mesoporous materials. H3 and H4 loops do not tend to close until equilibrium pressure is at or close to saturation pressure. H3 type is typical for loose aggregates of plate-like particles and in porous materials typical of pore networks containing macropores not entirely filled with condensate. H4 type loops suggest presence of slit-shaped pores including pores in the micropore region and plate-like particles with spaces between the parallel plates (Mokaya & Jones, 1995) and are common with activated carbons. H4 hysteresis loops are commonly observed with more complex materials consisting of both micropores and mesopores.

Adsorption and desorption  $N_2$  isotherms for all biochars (Figure 6.) showed low surface areas of between  $3.52 - 12.07 \text{ m}^2 \text{ g}^{-1}$  (Table 1) consistent with results reported in the literature for sewage sludge biochars which have low surface areas due to high ash content (Agrafioti *et al.*, 2013; Bagreev *et al.*, 2001; Schimmelpfennig & Glaser, 2012). It has been postulated that high ash contents reduce surface area by filling or blocking access to the biochar micropores (Song & Guo, 2012).

The low nitrogen uptake of all three biochars can be characteristic of materials with small ultra-micropores that are close to the kinetic diameters of nitrogen, since molecules cannot overcome the activation energy for passing through the pores at cryogenic temperatures (Kim *et al.*, 2011). To investigate this potential microporosity further  $CO_2$  adsorption isotherms at 273K were recorded for the three biochar samples (Figure 7).

The  $CO_2$ -based BET specific surface areas ( $S_{BET}$ ,  $\mu\text{SSA}$ ) and pore volume ( $\mu\text{PV}$ ) values were significantly larger than the  $N_2$ -derived BET specific surface area ( $S_{BET}$ ) and pore volume (TPV) values signifying that kinetic limitations with  $N_2$

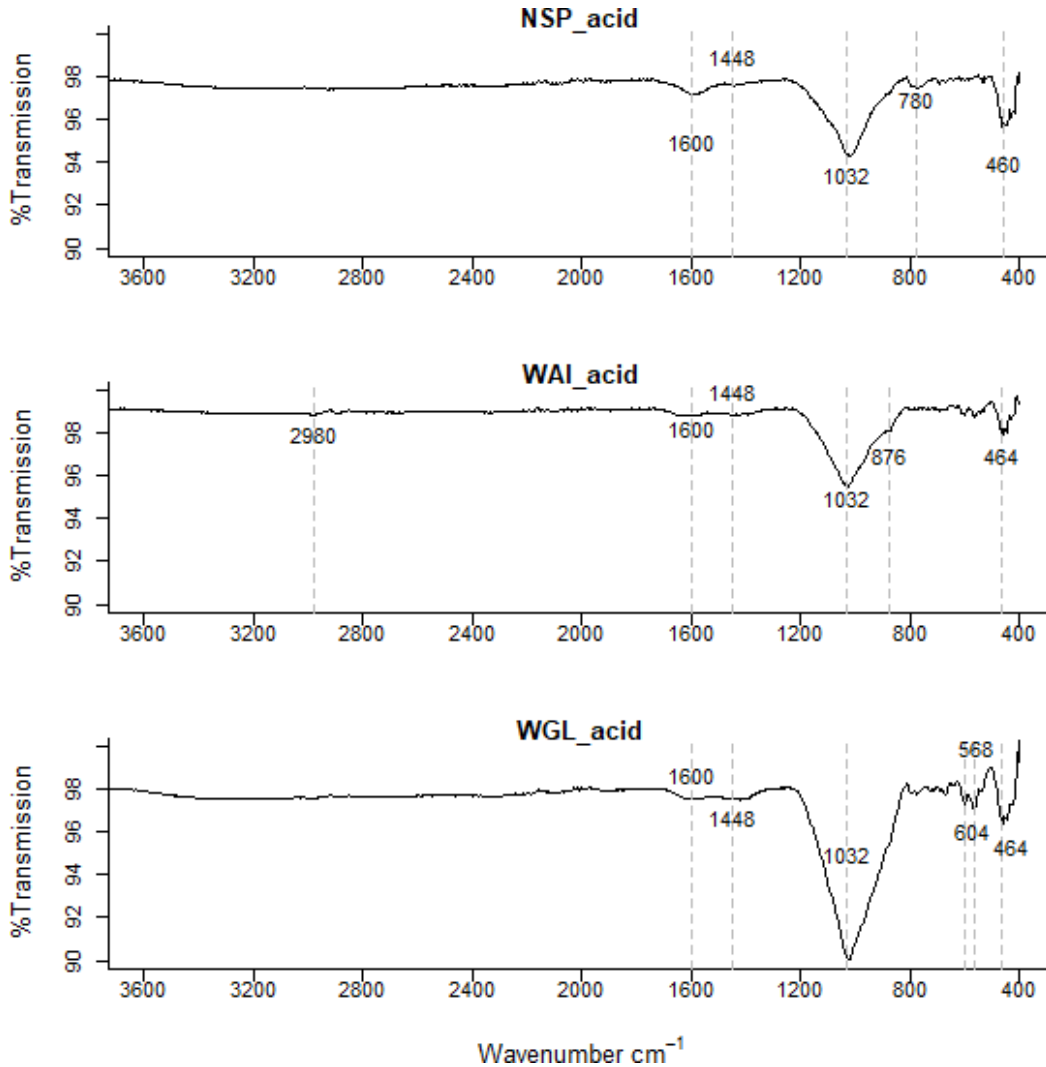


Figure 5. FTIR spectra of deashed (acid washed) NSP, WAI and WGL biochar.

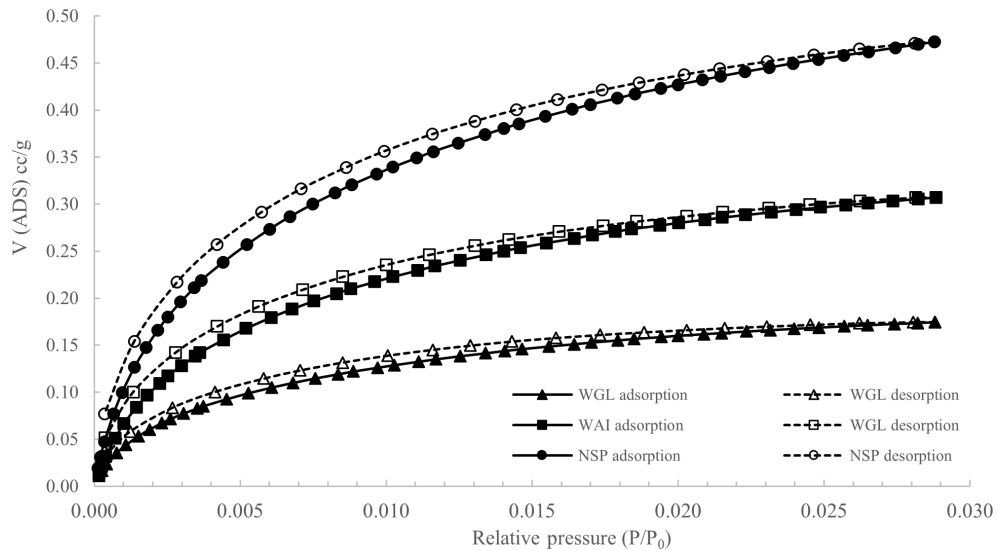
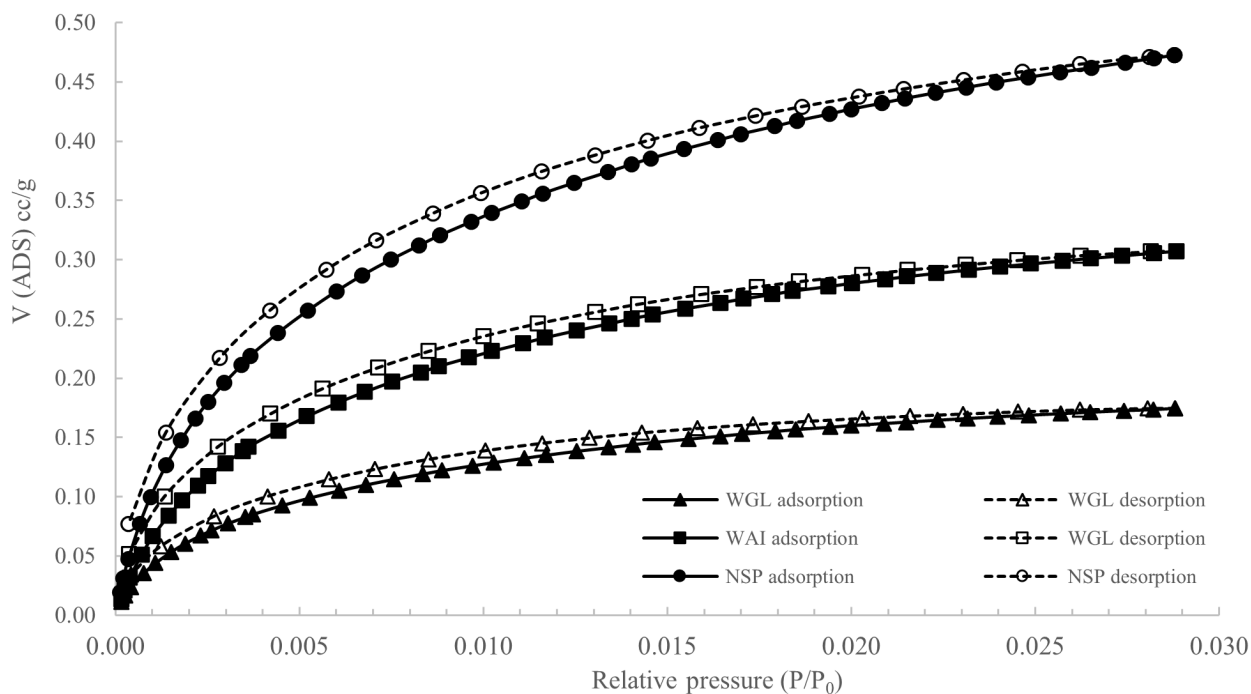


Figure 6. N<sub>2</sub> adsorption and desorption isotherms of WAI, WGL and NSP biochars ( $P/P_0$  = Relative pressure,  $V(ADS)$  cc/g = Volume of adsorption cc/g).



**Figure 7.** CO<sub>2</sub> adsorption and desorption isotherms for WAI, WGL and NSP biochars ( $P/P_0$  = Relative pressure,  $V$  (ADS) cc/g = Volume of adsorption cc/g).

physisorption were present for all biochars and there is some degree of microporosity present.

NSP\_BC showed the largest surface area measured with CO<sub>2</sub> and the lowest with N<sub>2</sub> indicating a more microporous structure whereas WGL\_BC had the highest N<sub>2</sub> SSA and lowest CO<sub>2</sub>  $\mu$ SSA signifying a slightly less microporous and more mesoporous structure. The greatest pore size distribution at pore diameters 4–15Å was recorded for NSP\_BC also indicating it had more of a microporous structure than WGL\_BC which recorded relatively sparse pore size distributions in this region (Figure 8b). In the mesoporous region, (16–150 Å), WGL\_BC pore size distributions were much greater than both NSP\_BC and WAI\_BC confirming WGL\_BC has a more mesoporous structure (Figure 8a).

The values obtained demonstrate the complex pore network within the biochar, even though the surface area values are generally low compared to other biochars there is still a degree of both microporosity and mesoporosity within the biochars. Low surface area biochars may be unsuitable for use as soil amendments as the water holding capacity is relatively low and the low porosities are not conducive to promoting soil microbial growth (Ishii & Kadoya, 1994; Thies & Rillig, 2009), which play an important role in nutrient cycling (Lambers *et al.*, 2008). The surface area could be increased by increasing the pyrolysis temperature (Song & Guo, 2012; Tomczyk *et al.*, 2020). The fast pyrolysis of municipal sludge biochar at temperatures 500 – 900 °C showed that increasing temperatures resulted in a greater microporous network within the biochar

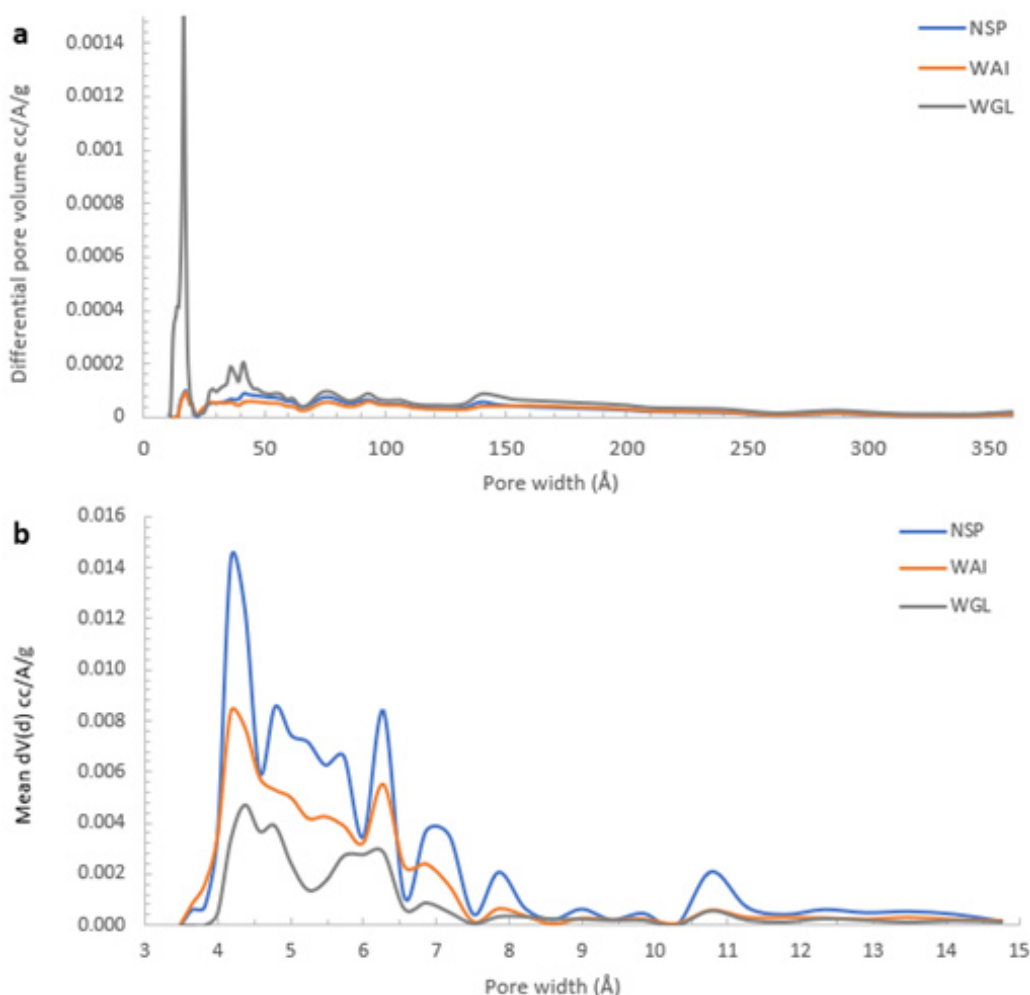
(Chen *et al.*, 2014). Previous work has shown that the greatest enhancement of sewage sludge biochar porosity occurred between 400 – 600°C (Bagreev *et al.*, 2001). However heavy metal concentration in biochars generally increase with pyrolytic temperature (Lu *et al.*, 2013). This is because heavy metals do not volatilize, so their concentration within the biochar increases with pyrolysis temperature (Chanaka Udayanga *et al.*, 2019; Hossain *et al.*, 2011; Wang *et al.*, 2021).

The larger surface areas (with CO<sub>2</sub>) and more microporous structure of NSP\_BC and WAI\_BC relates to their lower ash content of 67.0% and 62.3% respectively. The lowest surface area measured (with CO<sub>2</sub>) was that of WGL\_BC which recorded the highest ash content of 88.3%. These findings support the notion that lower surface areas relate to ash filling or blocking access to the biochar micropores (Song & Guo, 2012).

### Zeta potential

Zeta (electrokinetic) potential signifies the net charge between the surface plane and slip plane of a colloidal particle (Hiemenz & Rajagopalan, 1997). Zeta potential values yield information about the external surface charges of biochar particles in solution and indicates the sorption and nutrient holding characteristics of the biochar in soil. Negatively charged surfaces are unlikely to sorb negatively charged ions such as phosphate but are more likely to sorb positive cations such as heavy metal ions and ammonium ions.

The zeta potential values for all three biochar samples were negative in the pH range 5.0–9.5, revealing that negative charges



**Figure 8.** Pore volume weighted pore size-distribution derived from **a)**  $N_2$  (mesopore region 20–500Å) and **b)**  $CO_2$  (micropore region <20Å) for NSP, WAI and WGL biochars.

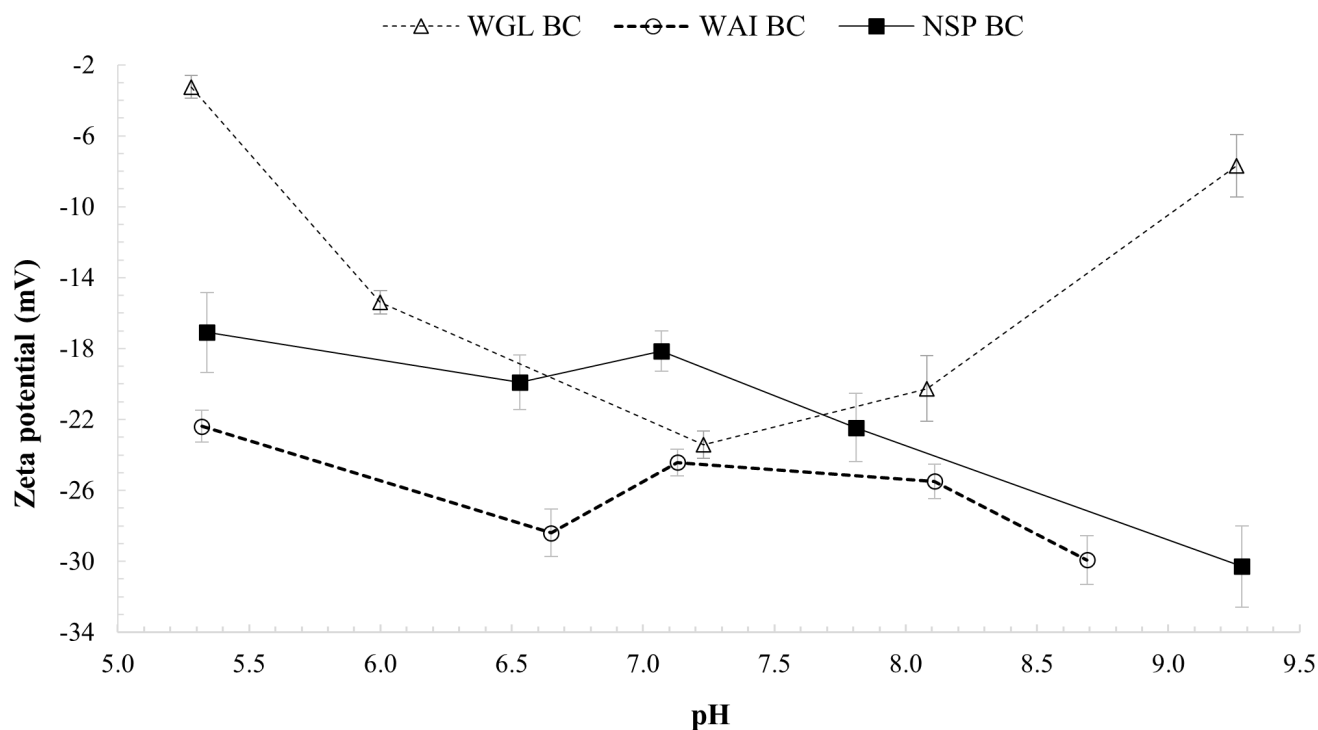
are carried on the surface of the biochar particles (Figure 9). FTIR spectra revealed the existence of oxygen containing functional groups ( $-COO^-$  and  $-OH$ ) on the biochars surface which can contribute considerably to surface charge of the biochars. The negative zeta potentials of all three biochars in the pH range 5.0 – 9.5 support this interpretation.

At acidic pH, the zeta potentials of the biochar samples became less negative, indicating that the association of  $-COO^-$  and  $-O^-$  with  $H^+$  reduced the negative charge of the biochars. With increasing pH, the zeta potential of WAI\_BC and NSP\_BC biochars become more negative due to increasing deprotonation of the biochar surface functional groups (Yuan *et al.*, 2011). However, at pH above 7 there was an increase in zeta potential for WGL\_BC from -23.4mV to -7.7mV indicating a decrease in negative surface charge. WGL\_BC contains the highest ash content of all the biochars (Table 1), and it is likely this that contributes to the increase in zeta potential values at  $pH > 7$ . The mechanism by which surface charge increases

at high pH values cannot be explained by deprotonation of the surface functional groups in the case of WGL\_BC. The higher ash content indicates some other mechanism occurring. Zeta potential of fine coal tailings containing several ash-forming minerals showed a similar trend which the authors attributed to the presence of alumina and silicate particles, which result in lower negative zeta potential values. They also noted that varying zeta potential values at high pH could be attributed to the binding of more cations such as  $Ca^{2+}$  (Kumar *et al.*, 2014). Positively charged calcium monohydroxide ions on the biochar surface would to some degree neutralize the negative surface charges resulting in less negative zeta potential values. (Liu *et al.*, 2002). It is possible that at pH values  $> 9$  WGL\_BC would have positive zeta potential values and thus more likely to sorb negatively charged ions such as nitrate or phosphate.

#### X-ray diffraction (XRD)

X-ray diffraction (XRD) analysis of the biochars revealed that mineral components in the crystal form were present in all



**Figure 9.** Zeta potentials of WGL\_BC, WAI\_BC and NSP\_BC at pH values from 5-9.5.

three biochars (Figure 10). Quartz was identified as the predominant crystalline phase with the highest peak at  $2\theta$  around  $26.6^\circ$  ( $d = 3.33 \text{ \AA}$ ) in NSP and WGL biochars. WAI biochar exhibited a more intense peak relating to  $\text{CaSO}_4$  (anhydrite). Quartz, sylvite, calcite, calcium sulphate, albite were the most common phases identified. These minerals are formed during pyrolysis due to a reaction between  $\text{CO}_2$  and alkaline-earth metals and alkaline oxyhydroxides.

Previous research has shown sewage sludge biochar to have a turbostratic structure where the carbon fraction is dominated by disordered graphitic crystallites (Srinivasan *et al.*, 2015; Uchimiya *et al.*, 2011). This is in discordance with the XRD results which show a lack of C (002) diffraction peaks ( $2\theta = 15\text{-}30^\circ$ ) and C (101) diffraction peaks ( $2\theta = 40\text{-}50^\circ$ ) due to amorphous carbon structures and graphite structures respectively. The biochars studied here do have a very high ash content and the lack of these peaks could be as a result of interference of high-intensity quartz peaks. Studies have shown that the high content of minerals, specifically quartz can affect the structural characterization of biochar carbon fraction (Feng *et al.*, 2015).

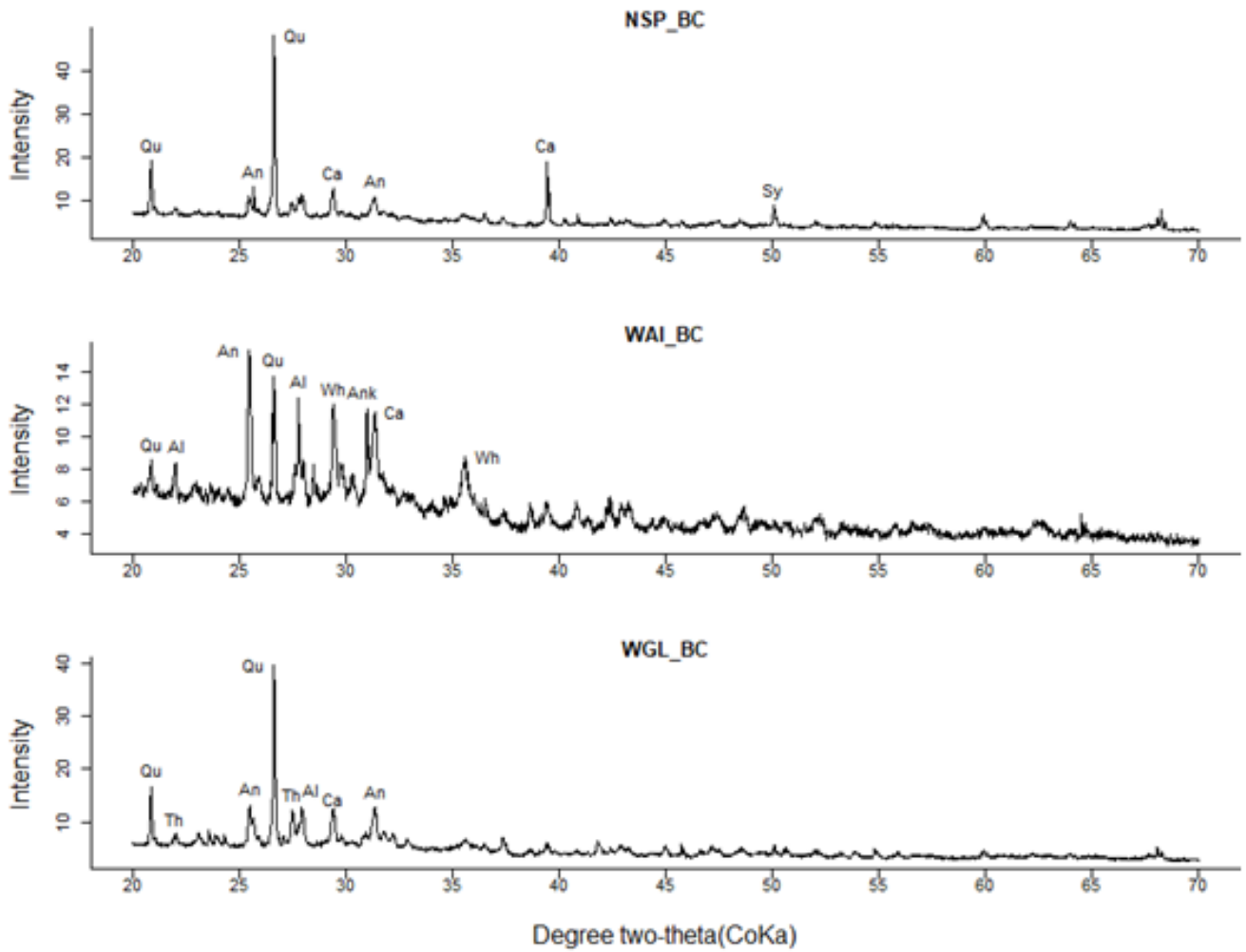
The difference in mineral composition between the three biochars could be due to possible contamination of FS by sand and grit caused by poorly lined containment structures (Niwaagaba *et al.*, 2014). The containment structures at each location would have to be investigated to reach a definitive

conclusion. Overall, the mineralogical composition of the biochars is in agreement with their high ash contents.

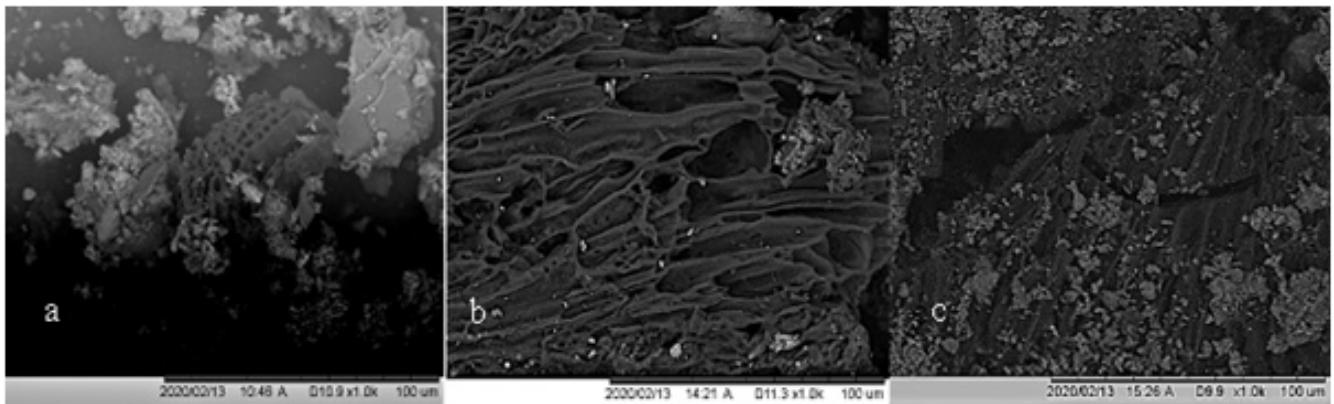
#### Scanning electron microscopy (SEM) with energy dispersive x-ray analysis (EDX)

SEM analysis reveals a complex porous structure evident in all biochars (Figure 11). The porous structure of biochars strongly resembles the cellular structure of the original feedstock (Fuertes *et al.*, 2010; Yao *et al.*, 2011). In the case of faecal sludge, cellular macroporous structures arise from undigested fibrous vegetable matter. The morphology of the biochar is honeycomb-like with cylindrical and slit like holes clearly observable. This porous structure can provide a specialized environment for the colonization of microbes (Thies & Rillig, 2012). This increase in mycorrhizal fungi contributes to increased mineralization of recalcitrant soil organic matter, ultimately improving soil and plant health (Anderson *et al.*, 2011; Zimmerman *et al.*, 2011). SEM images also show all three biochars have high ash content with EDX confirming the presence of mineral elements (Figure 12). The SEM images clearly showed a high presence of clay mineral particles/ash (white/grey) with a smaller amount of biochar particles present (black).

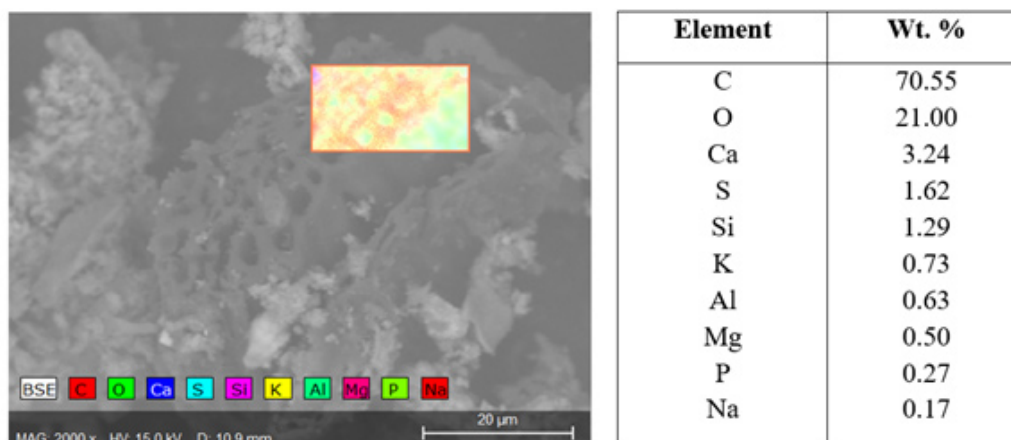
Visually WGL biochar had a higher ash content which is concurrent with the ash percentage from proximate analyses. EDX results on the biochar particles themselves revealed high volumes of carbon and oxygen (Figure 12). Also present were



**Figure 10.** XRD patterns of NSP, WAI and WGL biochar (Qu= Quartz, Al=Albite, Ca=Calcite, An = Anhydrite, Sy = Sylvite, Wh = Whewellite, Ank=Ankerite, Th= Thermanatrite).



**Figure 11.** SEM micrograph of (a) Original WGL biochar, (b) Original NSP biochar, (c) Original WAI biochar.



**Figure 12.** SEM-EDX map for all elements distribution across the area highlighted in image and associated energy dispersive X-ray (EDX) quantification of biochar.

silicon, calcium aluminium, potassium, magnesium, phosphorus, and sodium all of which are beneficial to plant health.

#### Cation exchange capacity

Cation exchange capacity (CEC) enables biochars to adsorb cationic nutrients such as  $\text{NH}_4^+$ ,  $\text{Ca}^{2+}$ , and  $\text{K}^+$ . It is thought this characteristic of biochar results predominantly from formation of carboxylic functional groups during oxidation (Cheng *et al.*, 2006).

There was a large variation in CEC values with WGL biochar (WGL BC) the highest CEC at  $129.3 \text{ cmolKg}^{-1}$  and NSP biochar the lowest CEC at  $41.9 \text{ cmolKg}^{-1}$  (Table 1). Fresh biochars from lignocellulosic biomass generally have lower CEC, with manure-based biochars exhibiting higher CEC values (Tag *et al.*, 2016). In the literature CEC values for biochar are highly variable, commonly ranging from  $6 \text{ cmol}_{(+) } \text{Kg}^{-1}$  (Munera-Echeverri *et al.*, 2018) to  $36.3 \text{ cmol}_{(+) } \text{Kg}^{-1}$  (Song & Guo, 2012) to as high as  $304 \text{ cmol}_{(+) } \text{Kg}^{-1}$ .

Yuan *et al.* (2011) proposed that high ash content biomass creates high CEC biochars and that K, Na, Ca, Mg, and P in the feedstock would promote formation of O-containing acidic functional groups such as carboxylic, and phenolic groups on biochar surface during pyrolysis and thus, result in higher CEC (Gaskin *et al.*, 2008). However, FTIR analysis showed a lack of acidic functional groups such as phenolic groups in these biochars. It is possible that the high ash content of these biochars could contribute to methodological problems in determining CEC (Graber *et al.*, 2017). There is a large range of CEC values reported in the literature and measurements are often poorly reproducible (Munera-Echeverri *et al.*, 2018). FTIR shows that there are carbonates and silicates present in these biochars which would result in the release of base cations and interference with the sum of measured exchangeable base cations (Munera-Echeverri *et al.*, 2018). WGL biochar records the highest CEC value ( $129.3 \pm 2.3 \text{ cmol.kg}^{-1}$ ) and the highest ash content of all three biochars implying that

it is the high ash content that is responsible for the high CEC value.

#### Conclusion

Overall, all three faecal sludge biochars had a high ash content, high pH, low carbon content, negative surface charge and low specific surface areas and pore volumes. The similarity of FTIR spectra between biochars signifies a uniformity of the organic component of all three biochars. Warangal biochar had a significantly higher ash content and pH compared to the Narsapur and Wai biochar. There were also differences in XRD spectra between biochars. These differences are likely related to the contamination of faecal sludge in the containment structure by sand or grit, or the sintered mineral deposits in the reactor itself. The variability of these faecal sludge biochar properties highlights the differences between small-scale laboratory and full-scale “real world” biochar production. Control over every single variable in large-scale faecal sludge biochar production is difficult and routine inspections of every containment structure at every location would be time-consuming. However, the pH and ash content of the biochars could be monitored periodically at the treatment plant. The low surface areas and porosity of these biochars could prove detrimental in its end use a soil amendment as these properties relate to water holding capacity and microbial activity. However, increasing the porosity of faecal sludge biochar is possible through techniques such as chemical and physical activation. Overall, the properties of these biochars, in particular the high alkalinity, shows their potential use as soil amendment particularly with acidic soil. The liming effect from these biochars and release of nutrients from the ash in the biochar itself could contribute to increased agricultural productivity especially in developing nations where the use of inorganic fertilizer on smallholder farmers is much lower. Future work should determine the biochars total and plant available macro-and micronutrient concentrations. Further investigation into the evaluation of these biochars as soil amendments with a focus on application to acidic soils is also recommended.



## Data availability

### Underlying data

Mendeley Data: FS biochar properties. <https://doi.org/10.17632/2xsdbdb38k.3> (Nicholas, 2022)

This project contains the following files:

- Figure 3 FTIR original\_final600.tiff- FTIR spectra unamended biochars
- Figure 4 FTIR ashed\_final600.tiff – FTIR spectra of ashed biochars
- Figure 5 FTIR acidwashed\_final600.tiff – FTIR spectra of acid washed biochars
- Figure 6 Nitrogen.tif – N<sub>2</sub> adsorption and desorption isotherms of WAI, WGL and NSP biochars
- Figure 7 CO2.tif – CO<sub>2</sub> adsorption and desorption isotherms of WAI, WGL and NSP biochars
- Figure 8 pore volumes600.tif - Pore volume weighted pore size-distribution graphs for a) N<sub>2</sub> (mesopore region 2-50nm) and b) CO<sub>2</sub> (micropore region<2nm) for NSP, WAI and WGL biochars.
- Figure 9 zeta potential600.tif – Zeta potentials of WGL\_BC, WAI\_BC and NSP\_BC at pH values from 5-9.5
- Figure 10 XRD500.tif - XRD spectra of NSP, WAI and WGL biochar
- Figure 11 SEM500.tif- SEM biochar images
- Figure 12 EDX500.tif – EDX biochar image
- CEC2.xlsx – All cation exchange capacity data
- CHNS\_results.xlsx – Elemental analysis (CHNS) data

- FTIR Data2.xlsx – all FTIR data
- XRD15.xlsx – Xray diffraction data
- N2 isotherm data.xlsx – Nitrogen adsorption and desorption isotherms data
- N2 Pore volume BET data.xlsx – Nitrogen pore volume data
- CO2 isotherm data.xlsx – Carbon dioxide adsorption and desorption isotherms data
- CO2 BET pore volume data.xlsx – Carbon dioxide pore volume data
- ph and electrical conductivity raw data.xlsx – pH and electrical conductivity raw data
- Raw zeta potential data.xlsx – raw zeta potential data

Data are available under the terms of the [Creative Commons Attribution 4.0 International license](https://creativecommons.org/licenses/by/4.0/) (CC-BY 4.0).

### CRediT authorship contribution statement

Larissa Nicholas: Conceptualization, Methodology, Investigation, Writing – original draft, Project Administration, Visualization. Ian Mabbett: Conceptualization, Methodology, Writing – review and editing, Funding Acquisition, Supervision. Henry Apsey: Investigation. Iain Robertson: Writing – review & editing.

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**Sadib Bin Kabir** 

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This study focuses on the biochar assessment derived from faecal sludge pyrolysis. Even though numerous research on small-scale faecal sludge-derived biochar characterization has been studied before, this study conducted the characterization of biochar in a full-scale operation. The work is clearly and accurately presented and it cites the current literature.

The study design is appropriate and the work is technically sound. This study's characterization methods include Fourier transform infrared (FTIR) spectroscopy, scanning electron microscopy (SEM), energy dispersive x-ray (EDX) spectroscopy, crystal x-ray diffraction (XRD), proximate analyses, and BET surface area porosimetry. The methods are explained in detail and are sufficient to allow replication by others.

Results showed that biochar samples had low specific surface area, high alkaline pH values, high ash content, and negative surface charge. In the Results section, the Fourier transform infrared section requires more improvement and critical discussion. The authors listed all the compounds present without proper explanation. X-ray diffraction analysis showed the mineral composition of each biochar differed slightly. Scanning electron microscopy analysis indicated a porous structure of each biochar with ash particles evident.

All the source data underlying the results ensure full reproducibility. The conclusions are drawn adequately supported by the results.

**Is the work clearly and accurately presented and does it cite the current literature?**

Yes

**Is the study design appropriate and is the work technically sound?**

Yes

**Are sufficient details of methods and analysis provided to allow replication by others?**

Yes

**If applicable, is the statistical analysis and its interpretation appropriate?**

Yes

**Are all the source data underlying the results available to ensure full reproducibility?**

Yes

**Are the conclusions drawn adequately supported by the results?**

Yes

**Competing Interests:** No competing interests were disclosed.

**Reviewer Expertise:** Faecal Sludge Treatment, Solid waste management, Wastewater treatment, Biofuel production, Solar-thermal energy storage system assessment.

**I confirm that I have read this submission and believe that I have an appropriate level of expertise to confirm that it is of an acceptable scientific standard.**

Reviewer Report 08 August 2023

<https://doi.org/10.21956/gatesopenres.15550.r34268>

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**Bhekumuzi P. Gumbi** 

School of Chemistry and Physics, University of KwaZulu-Natal, Durban, South Africa

### Overview

The Authors report on very important topic of nowadays circular economy of faecal sludge. Authors produced biochar from waste and characterized the biochar to find physico-chemical properties that can make it useful as soil enhancer. Various instrument were use to characterize the biochar including techniques that are not normal employed in the literature for characterization of biochars, which make this study significant.

### Observations

- The authors should list/tabulates the properties of soil enhancers and comments whether this biochar fit the criteria for application in soil conditioning.
- The authors should report data with standard deviation since they mentioned analysis was done in triplicate.
- Authors must be consistent when reporting units and heading font

## Comments

### *Method Section*

- Authors must provide more information ICP: calibrations curves, validations, linearity and procedures and preparation of standard.
- Subheading (measurement of potential) between HCL and Suspension; full stop is flying.
- Authors need to provide more details on procedures for determination of CECs.

### *Results and Discussion*

- Authors need to clarify whether Ash content and acidic functional groups are good or not for soil conditioning, and elaborate on how are correlated in this study.
- The XRD pattern predict as produced biochar has high crystallinity due to high ash content. Would authors explain whether crystallinity in general is an indication of high ash content.

### **Is the work clearly and accurately presented and does it cite the current literature?**

Yes

### **Is the study design appropriate and is the work technically sound?**

Yes

### **Are sufficient details of methods and analysis provided to allow replication by others?**

Partly

### **If applicable, is the statistical analysis and its interpretation appropriate?**

Partly

### **Are all the source data underlying the results available to ensure full reproducibility?**

Yes

### **Are the conclusions drawn adequately supported by the results?**

Yes

**Competing Interests:** No competing interests were disclosed.

**Reviewer Expertise:** Environmental Chemistry, Material Science, Biochar, Analytical chemistry, chromatography, electrochemistry

**I confirm that I have read this submission and believe that I have an appropriate level of expertise to confirm that it is of an acceptable scientific standard, however I have significant reservations, as outlined above.**

Reviewer Report 06 July 2023

<https://doi.org/10.21956/gatesopenres.15550.r33867>

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## Musa Manga

The University of North Carolina at Chapel Hill, Chapel Hill, North Carolina, USA

### Broad overview

The authors discuss and compare the properties of biochar obtained from the pyrolysis of faecal sludge at three treatment plants in India. They examined properties that influence its use as soil amendment/conditioner such as the ash content, pH, carbon content, organic surface groups, surface charge, mineral content, pore volume. This is a very insightful paper as the authors investigate an understudied area; they studied the characteristics of biochar from full scale treatment plants as opposed to former studies which have examined biochar from laboratory experiments. The manuscript is well written, and the authors provided broad discussion on their findings. However, there are still some shortfalls in the manuscript (as outlined below) that the authors need to address.

### General comments

- The authors did not state anywhere in the manuscript the reason for choosing the three different faecal sludge treatment plants (FSTPs). It will be good if the authors can clearly state why they chose these plants: are they the only three FSTPs in India? are they representative of FSTPs in India?
- Since the authors studied properties of biochar from large scale treatment plants, more value will be given to the paper if the authors can include in their discussion, comparisons between properties of biochar obtained from treatment plants and those obtained from laboratory experiments.
- In addition, since the authors decided to study the properties of biochar for use as a soil amendment, the authors should provide a separate section where they elaborately discuss the implications of these properties on biochar's use as soil amendment. In this section, they should also discuss what can be done to improve some of the properties that limit its use as soil amendment.

### Specific comments

#### Methods section:

##### *Under biochar preparation:*

Figure 2 was mentioned before Figure 1, this should be corrected.

##### *Under Characterization of biochar:*

Rather than saying "The biochar characterized were collected from the pyrolizer", it will be better to say "The following analysis were carried out on the collected biochar"

##### *Under chemical analysis:*

The full meaning of C, H, N, S should be provided at the point of first mention.



*Under proximate analysis:*

In this section, the authors state that the crucibles were first dried at a temperature of 750°C to remove moisture and other volatiles that are present. However, this temperature seems too high for just drying crucibles. The authors need to revisit this.

Results section:

*Under the FTIR Spectroscopy:*

The authors state that the constant values obtained in the study is an “an indication of the homogenous nature of faecal sludge” However, faecal sludge cannot be said to be homogenous in nature, it is rather heterogeneous.

**Review based on Gates Open Research Format:**

- Is the work clearly and accurately presented and does it cite current literature?

The work is clearly written and cites current literature.

- Is the study design appropriate and is the work technically sound?

No study design was provided in the work, samples were collected only once from the study areas. It would have been better if the samples were collected over a period of time and not just one time sample collection.

- Are sufficient details of methods and analysis provided to allow replication by others?

The authors provided elaborate descriptions of the methods they applied in their study which enables easy replication.

- If applicable, is the statistical analysis and its interpretation appropriate?

The authors did not provide good statistical analysis. They stated that the measure of uncertainty in the study is the standard deviation. However, they did not state how many samples they used for each analysis from each plant.

- Are all the source data underlying the results available to ensure full reproducibility?

YES, the authors provided all sources of data underlying the results from the study to ensure full reproducibility.

- Are the conclusions drawn adequately supported by the results?

The conclusions drawn from the study are well supported by the results. However, more conclusions and recommendations can still be made from the results.

**Is the work clearly and accurately presented and does it cite the current literature?**

Yes

**Is the study design appropriate and is the work technically sound?**

Partly

**Are sufficient details of methods and analysis provided to allow replication by others?**

Yes

**If applicable, is the statistical analysis and its interpretation appropriate?**

Partly

**Are all the source data underlying the results available to ensure full reproducibility?**

Yes

**Are the conclusions drawn adequately supported by the results?**

Yes

**Competing Interests:** No competing interests were disclosed.

**Reviewer Expertise:** Public health; epidemiology; water, sanitation and hygiene (WaSH);

**I confirm that I have read this submission and believe that I have an appropriate level of expertise to confirm that it is of an acceptable scientific standard, however I have significant reservations, as outlined above.**

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### Version 1

Reviewer Report 06 October 2022

<https://doi.org/10.21956/gatesopenres.15015.r32521>

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**Santiago Septien Stringel** 

WASH R&D Centre (formerly known as the Pollution Research Group), University of KwaZulu-Natal, Durban, South Africa

The reviewed paper provides the characterization of biochar issued from faecal sludge from three distinct faecal sludge treatment plants in India. The studied characteristics of the biochar samples allowed to assess the suitability of the reuse of this type of sanitation product in agriculture as soil improver. This type of advanced biochar characterization has been rarely done in literature. The results are quite interesting and contribute to increasing the body of knowledge on biochar derived from faecal sludge, which is relatively poor for the moment.

The paper is very well written in general, the results well discussed and analyzed, and the conclusion is concise and pertinent. Definitely, the paper deserves to be indexed after minor corrections.

**Abstract:**

- No comments.

**Keywords:**

- Some keywords are redundant: "faecal sludge" and "fecal". Remove one of the two.

**Introduction:**

- Paragraph 6: there are more advantages of the use of biochar as soil conditioner that you can mention (e.g., removal of pollutants, reduction of pollution of underground water, support the growth of microbial communities beneficial for plant growth, retention of nutrients).
- Paragraph 6: concerning the literature about faecal sludge derived biochar, you should provide more details of what has been already studied, the objectives of the studies and the identified gaps (areas of knowledge not yet covered).

#### Material and Methods:

- Biochar preparation:
  - Indicate the conventional characteristics of the faecal sludge samples (i.e., moisture content, total solids, suspended solids, volatile solids, ash, COD, pH, electrical conductivity);
  - With “solar drying”, do you mean “drying beds”? Be more specific.
  - Provide more information about the pyrolyzers (estimated heating rate, throughput, residence time, carrier gas, etc...). If possible, add photographs.
- Characterization of biochar:
  - For all tests, indicate the number of replicates and the statistical method to measure experimental uncertainty.
  - Mention explicitly that you characterized the biochar collected from the pyrolyzer.
  - “Proximate analysis” section: for moisture content determination, be more precise on the duration of the samples in the oven.
  - “FT-IR analysis section”, paragraph 1, line 4: “infrared” must be written without capital letter
  - “Surface area section”, paragraph 2: Was the degassing done at 130°C or 105°C? It's not clear.
  - “Cation exchange capacity” section: you wrote the name of the chemical elements in full letter and by the chemical nomenclature. Be consistent. Don't mix both styles.
- Results and Discussions:
  - “Proximate analyses, EC, pH and elemental analyses” section:
    - You should relate the higher ash content of the biochar to the initial ash content of the sludge.
    - As an explanation of the high ash content in faecal sludge, the disposal of liquid and solid waste in the toilets could also contribute to the high amount of inorganic material in the sludge.
    - It would have been interesting to measure the carbon content in the sludge to verify if this parameter increases or decreases after pyrolysis in your case.
  - “Fourier transform infrared (FTIR) spectroscopy” section:
    - You could conduct an element analysis of the ash from the three biochar samples and correlate the FTIR results to these results.
    - What could be the reasons that the sludge biochar have the same composition according to the FTIR spectra? What are the implications of this in the application?
    - There is not a clear difference between Figure 2 (biochar) and 3 (ashed-

- biochar). It seems to me that biochar and ashed-biochar are the same. Please clarify.
- Paragraph 2: in Figure 2, 3 and 4, we cannot see the spectrum in the 3800 cm<sup>-1</sup> to 3600 cm<sup>-1</sup> region (related to OH group variations within mineral matter).
  - Paragraph 4: it is not clear if the discussion corresponds to the ashed or deashed biochar.
  - Paragraph 6: it is a repetition of Paragraph 5, so remove it.
  - Paragraph 8: the third sentence repeats information previously said.
- ○ “Surface area” section:
    - Paragraph 2: it contradicts what has been stated in the previous paragraph. If the sorption isotherms are of Type II, it could be expected a nonporous and microporous adsorbent, but it was found that the biochar samples have a degree of microporosity and mesoporosity. Please clarify.
    - Paragraph 3: indicate to what corresponds “H3” and “H4”.
    - Paragraph 7: there is a lack of consistency in the notations of the biochar samples (e.g., instead of writing “WGL\_BC” you wrote “WGL Biochar”). Correct this in the manuscript.
    - Paragraph 7: Can the difference of porosity between samples be related to their ash content?
    - Paragraph 8: The adsorption of water in biochar could follow different mechanism than N<sub>2</sub>. It would have been interesting to determine the sorption isotherm with water (in a Dynamic Vapor Sorption or using the saturated salt solution), in order to have a more realistic assessment of the water holding capacity in the biochar. If you add this data, your paper will take an extra dimension, making it more appealing.
    - In Figure 7, for better clarity, be consistent with the units between the graph and the legend (use nm or A, but avoid mixing them).
    - In Figure 7a, the WGL\_BC peak is the highest in the micropore region, whereas it is the lowest in Figure 7b. Please clarify.
  - ○ “X-ray diffraction (XRD) section”:
    - Paragraph 1: “Anhydrite” must be written without capital letter.
    - Paragraph 2: Where we can see the carbon fraction peaks in the XRD graphs? Please include an indication in Figure 9.
    - Paragraph 2, sentence 5: what do you refer to with “these peaks”?
    - Paragraph 3: How were the high content of the biochar samples in Ca, Si and K, as well as its high alkalizing capacity, determined?
    - Paragraph 3: “Thermonatrite” must be written without capital letter.
  - Conclusion:
    - Indicate the weak points of using biochar derived from faecal sludge as soil condition, and suggest methods to overcome these weaknesses.
    - Precis in which context the high alkalinity of the biochar would be beneficial (i.e., acidic soils).

**Is the work clearly and accurately presented and does it cite the current literature?**

Yes

**Is the study design appropriate and is the work technically sound?**

Yes

**Are sufficient details of methods and analysis provided to allow replication by others?**

Yes

**If applicable, is the statistical analysis and its interpretation appropriate?**

Partly

**Are all the source data underlying the results available to ensure full reproducibility?**

Yes

**Are the conclusions drawn adequately supported by the results?**

Yes

**Competing Interests:** No competing interests were disclosed.

**Reviewer Expertise:** sludge and wastewater treatment, thermal process, biochar

**I confirm that I have read this submission and believe that I have an appropriate level of expertise to confirm that it is of an acceptable scientific standard.**

Author Response 30 Nov 2022

**Hannah Larissa Nicholas**

Keywords:

- **Comment 1:** Some keywords are redundant: "faecal sludge" and "fecal". Remove one of the two.

**Authors response:** For some reason I cannot amend keywords on the system.

Introduction:

- **Comment 2:** Paragraph 6: there are more advantages of the use of biochar as soil conditioner that you can mention (e.g., removal of pollutants, reduction of pollution of underground water, support the growth of microbial communities beneficial for plant growth, retention of nutrients).

**Authors response:** Agreed and added a section about this.

- **Comment 3:** Paragraph 6: concerning the literature about faecal sludge derived biochar, you should provide more details of what has been already studied, the objectives of the studies and the identified gaps (areas of knowledge not yet covered).

**Authors response:** Agreed. Another section with more details has been added to the introduction

**Material and Methods:**

Biochar preparation:

- **Comment 4:** Indicate the conventional characteristics of the faecal sludge samples (i.e., moisture content, total solids, suspended solids, volatile solids, ash, COD, pH, electrical conductivity);

**Authors response:** Unfortunately, I do not have this data.

- **Comment 5:** With “solar drying”, do you mean “drying beds”? Be more specific.

**Authors response:** Agreed and added more specific terms.

- **Comment 6:** Provide more information about the pyrolyzers (estimated heating rate, throughput, residence time, carrier gas, etc...).

**Authors response:** I have added the data that I have available to me and added a photograph.

**Characterization of biochar:**

- **Comment 7:** For all tests, indicate the number of replicates and the statistical method to measure experimental uncertainty.

**Authors response:** I have added more detail in the method section and confirmed that standard deviation values have been provided for experimental uncertainty

- **Comment 8:** Mention explicitly that you characterized the biochar collected from the pyrolyzer.

**Authors response:** Agreed and amended.

- **Comment 9:** “Proximate analysis” section: for moisture content determination, be more precise on the duration of the samples in the oven.

**Authors response:** This has been amended in the text.

- **Comment 10:** “FT-IR analysis section”, paragraph 1, line 4: “infrared” must be written without capital letter.

**Authors response:** This has been amended in the text.

- **Comment 11:** “Surface area section”, paragraph 2: Was the degassing done at 130°C or 105°C? It's not clear.

**Authors response:** Changed and made clearer.

- **Comment 12:** “Cation exchange capacity” section: you wrote the name of the chemical elements in full letter and by the chemical nomenclature. Be consistent. Don't mix both styles.

**Authors response:** This has been amended in the text.

**Results and Discussions:**

- “Proximate analyses, EC, pH and elemental analyses” section:

- **Comment 13:** You should relate the higher ash content of the biochar to the initial ash content of the sludge.-

**Authors response:** Agreed and amended.

- **Comment 14:** As an explanation of the high ash content in faecal sludge, the disposal of liquid and solid waste in the toilets could also contribute to the high amount of inorganic material in the sludge. It would have been interesting to measure the carbon content in the sludge to verify if this parameter increases or decreases after pyrolysis in your case.

**Authors response:** Agreed and amended.

“Fourier transform infrared (FTIR) spectroscopy” section:

- **Comment 15:** You could conduct an element analysis of the ash from the three biochar samples and correlate the FTIR results to these results.

**Authors response:** This would have been a really good idea. Unfortunately, as I have finished my PhD, I cannot do any further experimental work. Also, I didn't have access to an elemental analyser so samples had to be sent away for analysis which makes it a bit trickier.

- **Comment 16:** What could be the reasons that the sludge biochar have the same composition according to the FTIR spectra? What are the implications of this in the application?

**Authors response:** I think the homogeneous nature of faecal sludge leads to the same composition and the similar temperatures that the sludge was pyrolyzed at.

- **Comment 17:** There is not a clear difference between Figure 2 (biochar) and 3 (ashed-biochar). It seems to me that biochar and ashed-biochar are the same. Please clarify.-

**Authors response:** They are different, although not massively due to the very high ash content of the biochars. There is a low intensity peak in biochar between 1540 and 1650 whereas this peak is not evident in the ashed biochar (Figure 3.).

- **Comment 18:** Paragraph 2: in Figure 2, 3 and 4, we cannot see the spectrum in the 3800 cm<sup>-1</sup> to 3600 cm<sup>-1</sup> region (related to OH group variations within mineral matter). –

**Authors response:** I did cut that out to make the graph clearer but there were no peaks in that region. I would like to keep the graph as it is.

- **Comment 19:** Paragraph 4: it is not clear if the discussion corresponds to the ashed or deashed biochar.

**Authors response:** I have amended this to make it clearer.

- **Comment 19:** Paragraph 6: it is a repetition of Paragraph 5, so remove it.

**Authors response:** Amended- thanks for spotting this.

- **Comment 20:** Paragraph 8: the third sentence repeats information previously said. – Not sure I can find this section. So, no action has been taken.

“Surface area” section:

- **Comment 21:** Paragraph 2: it contradicts what has been stated in the previous paragraph. If the sorption isotherms are of Type II, it could be expected a nonporous and microporous adsorbent, but it was found that the biochar samples have a degree of microporosity and mesoporosity. Please clarify. –

**Authors response:** So later on we do clarify that the isotherms are pseudo type II. This is actually quite tricky. The shape of the adsorption isotherms is best suited to type II, but type II isotherms are characterized by a lack of hysteresis and there is hysteresis to some degree here, especially when looking at NSP and WAI biochars. Type IIs are characteristic for nonporous and macroporous adsorbents. As there are indications of hysteresis loops especially for WAI and NSP biochar, this would indicate a type IV isotherm according to the IUPAC classification but there is no plateau characteristic for this type. So really, I can't say its type II or type IV. According to (Sing and Williams, 2004) the isotherms can be classified as pseudo-type II, describing delayed capillary condensation due to the small rigidity of the aggregate structure of the adsorbent. So, I have essentially concluded it is a pseudo type II isotherm. I hope this makes sense!

- **Comment 22:** Paragraph 3: indicate to what corresponds “H3” and “H4”.

**Authors response:** I have made this much clearer.

- **Comment 23:** Paragraph 7: there is a lack of consistency in the notations of the biochar samples (e.g., instead of writing “WGL\_BC” you wrote “WGL Biochar”). Correct this in the manuscript.

**Authors response:** This has been corrected.

- **Comment 24:** Paragraph 7: Can the difference of porosity between samples be related to their ash content?

**Authors response:** Yes absolutely – I have added a paragraph at the end.

- **Comment 25:** Paragraph 8: The adsorption of water in biochar could follow different mechanism than N<sub>2</sub>. It would have been interesting to determine the sorption isotherm with water (in a Dynamic Vapor Sorption or using the saturated salt solution), in order to have a more realistic assessment of the water holding capacity in the biochar. If you add this data, your paper will take an extra dimension, making it more appealing.

**Authors response:** This is really interesting! Unfortunately, I have completed my PhD and am unable to perform any additional experiments, however, I hope to keep working in this field so this is something I shall remember for the future.

- **Comment 26:** In Figure 7, for better clarity, be consistent with the units between the graph and the legend (use nm or Å, but avoid mixing them).

**Authors response:** Agreed and amended.

- **Comment 27:** In Figure 7a, the WGL\_BC peak is the highest in the micropore region, whereas it is the lowest in Figure 7b. Please clarify.

**Authors response:** Sorry, this is confusing, it is the highest peak at but a smaller value than Figure 7b. So, the y axis is smaller by a factor of 10 approximately for figure 7a than 7b. Then for WGL-BC it is highest in the mesopore region - figure 7a. It is lowest in the micropore region in figure 7b. I think as it has higher ash content the ash blocks access to the micropores. So, for WGL\_BC the pore volume comes from the mesopore region. For NSP and WAI biochars their pore volume comes from the micropore region. So WGL\_BC has a higher peak in the mesopore region (Fig 7a) – its pore volume comes from bigger pore sizes and a lower peak in the micropore region due to ash blocking the micropores, so it has limited pore volume arising from the micropore region. Apologies if I have explained this terribly.

“X-ray diffraction (XRD) section”:

- **Comment 28:** Paragraph 1: “Anhydrite” must be written without capital letter.

**Authors response:** Amended.

- **Comment 29:** Paragraph 2: Where we can see the carbon fraction peaks in the XRD graphs? Please include an indication in Figure 9.

**Authors response:** This section has been taken out entirely - the links to the carbon fraction peak is tenuous, there may be a tail-end of a peak visible, further experiments with  $2\theta < 20$  would be needed to be able to positively identify any peaks and write confidently about them. The XRD results are still useful, and there is a lack of carbon fraction peaks in the spectra

- **Comment 30:** Paragraph 2, sentence 5:

**Authors response:** clarified as per previous comment.



- **Comment 31:** Paragraph 3: How were the high content of the biochar samples in Ca, Si and K, as well as its high alkalizing capacity, determined?

**Authors response:** Agreed that this wasn't determined clearly enough, so this section has been removed

- **Comment 32:** Paragraph 3: "Thermonatrite" must be written without capital letter.

**Authors response:** Amended.

Conclusion:

- **Comment 33:** Indicate the weak points of using biochar derived from faecal sludge as soil condition and suggest methods to overcome these weaknesses.

**Authors response:** Agreed this has been added to conclusion.

- **Comment 34:** Precis in which context the high alkalinity of the biochar would be beneficial (i.e., acidic soils).

**Authors response:** Agree and have added a paragraph to the conclusion

**Competing Interests:** No competing interests were disclosed.