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# **Effect of coffee husk and cocoa pods biochar on phosphorus fixation and release processes in acid soils from West Cameroon**

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#### **Abstract**

AcidsoilinWestCameroon has limited phosphorus (P) availability whichlimits plant growth. This is mainly because of low pH, high levels of exchangeable alu- minium (Al) and iron (Fe) and fixation of P. In this study, acid soils, sampled in Bafang, were amended with biochar produced from coffee husks (CH) and cocoa pod husks (CP) at two different temperatures (350 and 550 °C) in other to evalu- ate the effect on the physicochemical properties of the acid soil and the effect on P sorption and desorption. The soil was amended with biochar at a rate of 0, 20, 40 and 80g/kg and incubated for 7 and 60days. Physicochemical properties of allsoil–biocharsamplesweredeterminedfollowedby sorption experiments and data fitted in the Langmuir and Freundlich isotherm models in other to evaluate soil P sorption capacity and its affinity to soil amended with biochar. Moreover, desorption studies were done to evaluate the availability of P in soil amended with biochar after sorption. The outcomes ofthis study reveal an increase insoil pH, electrical conductivity (EC), available P, soil organic carbon and a drastic de- crease in exchangeable Al and Fe. The point of zero charge of biochar-amended soil was higher than the control and increased with amendment rate. The ex- perimental data of the sorption of P on soils and soil–biochar samples fits into Langmuir and Freundlich models  $(R^2 > 0.9)$  suggesting that the P adsorption is controlled by both model mechanisms. Soil–biochar mixture results in a decrease in the sorption capacity as compared with the control and the decrease was pre- dominant with increasing amendment rate. At amendment rates of 20, 40 and 80 g/kg after 7 days of incubation,  $Q_{\text{max}}$  for SCH350 were 2267, 2048 and 1823 mg/ kg which increased to 2407, 2112 and 1990 mg/kg after 60days of incubation. This tendency was observed for all biochar inputs with respect to the increase in incubation days. Furthermore, desorption of P from soil–biochar mixtures was enhanced with biochar added at greater rate and produced at higher tempera- ture. The desorption percentage was increased by more than around 10% for all biochar types from 20mg/kgto80mg/kgamendment.Thus,biocharadditionto acid soils reduces P fixation to acid soil and improves P desorption to soil solu- tion, thereby providing more available P in the soil solution and better conditions for plant growth.

#### **KEYWORDS**

#### **1 INTRODUCTION**

Tropical soils in Africa are mostly acidic (Takow et al., [1991\)](#page-20-0) and acidic soils are widely spread in the western region of Cameroon (Tematio et al., 2004) which presents critical levels of P (Tchuenteu, [1997\)](#page-20-2). This is because of a high level of fixation via reaction with hydr(oxides) of Al and Fe found in clay minerals thus depriving P to plants resulting in poor growth (Mbene et al., [2017;](#page-19-0) Yadav et al., [2012\)](#page-20-3). The fixation of P in soils is influenced by soil organic matter, pH, exchangeable Al, Fe, amount and type of silicate clays, calcium carbonate content (Eriksson et al., [2015;](#page-18-0) Gérard, [2016;](#page-18-1) Guppy et al., [2005\)](#page-18-2) which affect soil biogeochemical processes (dissolution, adsorption and precipitation).

Generally, acid soils manifest a dominance in  $H^+$  and

Al<sup>3+</sup> ions in their soil solution (Ondo, [2011\)](#page-19-1), little or- ganic matter content because of mineralisation (Tiessen et al., [1994\)](#page-20-4) and lack of nutrients. One of the ways to re- mediate these limitations is amendments which include the application of lime (Pagani&Mallarino, [2012\)](#page-19-2), com- post (Chen et al., [2004\)](#page-18-3), wood ash (Nkana et al., [2002\)](#page-19-3), direct P fertilization (Ondo et al., [2017\)](#page-19-4), rock phosphate (Basak & Biswas, [2016\)](#page-17-0), etc. Excess use of P fertilizer and manure may constitute a risk of surface- and ground water impairment known as eutrophication (Parvage et al., [2013\)](#page-19-5). Excess liming will lead to soil compaction (Alavéz-Ramírez et al., [2012\)](#page-17-1). An alternative to these materials is the use of biochar.

Biochar has received great attention as an alternative option to overcome many aspects of soil limitations. This is a material derived from the pyrolysis of biomass in an atmosphere containing little or no oxy- gen(Joseph & Lehmann, [2009\)](#page-18-4). Biochar has multifunctional values that includes the use of it for the following purposes: soil amendment to improve soil health, nutrient addition, immobilizing agent for remediation of toxic metals and organic contaminants in soil and water and mitigation of greenhouse gas emissions (Abhishek et al., [2022\)](#page-17-2). It is also used in the domain of carbon sequestration, climate change mitigation and as a soil conditioner (Bolan et al.[,2022\)](#page-17-3).Feedstocksusedfor the production of biochars are mostly obtained from agricultural biomass, which is abundant and of little cost.

Coffee and cocoa are one of the main cash crops grown in Cameroon. Waste produced from these crops (coffee husk and cocoa pod husk) when poorly disposed of can generate serious environmental problems. In Bafang, coffee husks are disposed of in the river which can cause siltation of rivers (Acchar & Dultra, [2015\)](#page-17-4) and poor disposal of cocoa pod husks in cocoa farms can be source of black pot rot to plants (Yapo et al., [2013\)](#page-20-5). Thus there is a need to properly manage these materials to protect the environment.

Biochar application to acid soils alters soil physico- chemical properties (Chintala, Mollinedo, et al., [2014\)](#page-18-5) and improvesPavailability(Glaser&Lehr[,2019\)](#page-18-6).Biochar addition ameliorates soil acidity because of its alkaline nature by reducing the quantity of  $H^+, A l^{3+}, F e^{3+}$  and  $F e^{2+}$ 

and thus reducing P precipitation (Wang et al., [2012\)](#page-20-6). Earlier studies have reported that biochar produced from different feedstocks and pyrolysis temperatures has di- verse physical and chemical properties such as functional groups, mineral contents, pH, CaCO<sub>3</sub> content, cation ex- change capacity and surface area (Chintala, Schumacher, et al., [2014;](#page-18-7) Eduah et al., [2019;](#page-18-8) Liang et al., [2014\)](#page-18-9). Moreover,biocharaddedtoacidsoilshavecontrastingresults on the fixation of P through sorption studies. Some results have presented an increase in sorption of P over the control acid soils because of the binding of phosphate to the carbonates and oxides of Ca and Mg (Novak et al., [2009;](#page-19-6) Xu et al., [2014;](#page-20-7) Zhang et al., [2016\)](#page-20-8) while on the contrary, less sorption of P was also observed than the control acid soils

which was attributed to the precipitation of  $Al^{3+}$  and  $Fe^{3+}$  (DeLuca et al., [2015\)](#page-18-10) and the re- pulsion of the negative phosphate ions by the negative charge of biochar (Chintala, Schumacher, et al., [2014;](#page-18-7) Martínez et al., [2017\)](#page-18-11).

Few research works have been done to observe the changes in the physicochemical properties of biocharamended soil (Djousse Kanouo et al., [2019;](#page-18-12) Pouangam Ngalani et al., [2022\)](#page-19-7) in Cameroon and to our knowledge, none on how P fixation is affected by biochar amendment on acid soils from west Cameroon. The present study had the objectives to (i) find out the effect of CH and CP bio- char on the physicochemical properties of acid soil at different amendment rates and incubation times and (ii) the effect of biochar on P sorption and desorption.

#### **2 MATERIALS AND METHODS**

#### **2.1 Soil sampling and biochar preparation**

Soil samples were randomly collected at a depth of 15 cm on a 2-hectare cocoa and coffee farm in Bafang (5°08′55″N 10°09′32″E), West region of Cameroon. The soil samples were air-dried for 1 week, crushed, sieved through a 2-mm sieve and stored in plastic bags for incubation studies. The soil was a Loamy soil with 42.9% silt, 33.3% sand and 23.8% clay. It was acidic with a pH of 4.7, and electrical conductivity of 53.2  $\mu$ S/cm, exchangeable acidity of 3.1 cmol/kg and available P of 16.1 mg/kg.

Coffee husks and cocoa pod husks collected from coffee processing factories and cocoa farms in Bafang, were washed with tap water and rinsed with distilled water, dried at 105°C to remove moisture then ground and sieved with a 2-mm sieve. The biomass was then inserted in the muffle furnace set up at two different temperatures (350°C and 550°C) for 4 h and herein annotated as CH350, CH550, CP350 and CP550 respectively. Thereafter, the biochars were finely ground, dried (105°C) and stored in polythene bags for characterization and incubation experiments. This preparation process was proposed by (Pouangam Ngalani et al., [2022\)](#page-19-7).

### **2.2 Incubation study for soil–biochar**

An incubation pot- experiment study was done to investigate the effect of different biochar at varying pyrolysis temperatures, amendment rates and incubation periods on the physicochemical properties of the acid soil sample and P sorption and desorption capacity. The soil was mixed in transparent incubation plastic pots with CH350, CH550, CP350 and CP550 at the rate of 0 g/kg, 20 g/kg, 40 g/kg and  $80g/kg$  then incubated for 7 and 60 days. Each treatment was replicated four times at 70% field water capacity and left at room temperature. At the end of the incubation periods, the amended and the control soil samples were air-dried, ground and sieved to <2mm and then stored in plastic cups for characterization and P sorption and desorption studies. Soil– biochar mixtures were labelled as SCH350, SCH550, SCP350 and SCP550 for soil incubated with biochar pyrolysed at 350 and 550°C.

#### 2.2.1 Characterization of biochar, soil and soil– biochar samples

The ash content of the biochar was determined by dry combustion of the biochar in a muffle furnace at  $750^{\circ}$ C for 6h (Rehrahet al.[,2014\)](#page-19-8).The acid-neutralizing capacity of biocharwasdeterminedby mixingbiochar ina solution of HCl and back titrating with NaOH (Martinsen et al., 2015) and CaCO<sub>3</sub> equivalence was determine by Rayment and Lyons

[\(2011\)](#page-19-10) method. Fourier transform infrared spectroscopy (scan range from 400 to 4000 cm<sup>-1</sup>) was recorded on an Alpha spectrometer from Bruker Optics and XRD pat- terns of the biochar were registered with CuK $\alpha$  (20 from 5 to 80°) for 7 h using Bruker D4. The surface morphol- ogy and the elucidation of the chemical properties of the various biochar were investigated by Scanning Electron Microscope (SEM) and Energy Dispersive Spectroscopy (EDS). The samples were coated with 10 nm gold layer using the rotary pump sputter coater (Leica EM ACE600, Wetzaler, Germany). The coated samples were loaded on the ThermoScientific Prisma E SEM with an Oxford EDS system for SEM-EDS. These observations were operated at an accelerated potential of 20.00 KeV and magnifica- tions of SEM images at magnification 1000 $\times$  and 3500 $\times$  taken. Biochar pH and EC were determined by shaking a mixture of biochar to deionized water (1:5 wt/wt ratio) (Singh et al., [2010\)](#page-19-11) while the pH and EC of soil and soil– biochar mixtures were determined by shaking water and deionized water in the ratios 1:2.5 and 1:5 respectively (Pansu & Gautheyrou, [2007\)](#page-19-12). Available P from biochar, soil and soil–biochar were determined using the Olsen P solution  $(0.5 M NaHCO<sub>3</sub> at pH 8.5)$  and the aliquot taken for P Spectro colorimetry determination (Murphy & Riley, [1962;](#page-19-13) Olsen et al., [1954\)](#page-19-14). Quantification of or- ganic carbonfrombiochar, soilandsoil–biocharmixture was determined by Walkley and Black [\(1934\)](#page-20-9) method. Exchangeable acidity, exchangeable Al (Ex Al) and ex- changeable Fe (Ex Fe) were based on the principle of sample washing with a saline solution of 1 M KCl solu- tion (Pansu & Gautheyrou, [2007\)](#page-19-12). Exchangeable acidity, in the aliquot was determined by titrating against 0.01M NaOH with phenolphthalein as an indicator then for Ex Al, after adding 1 M KF to the former solution, the pink solution was titrated to colourless using 0.01 M HCl. Ex Fe was determined by analysing an aliquot of solution by complexation of  $Fe^{2+}$  with ortho-phenanthrolineandthe absorbance read with visible-spectroscopy.

#### **2.3 Point of zero charge analysis ofsoil and soil–biochar mixtures**

The salt addition method was used for the determination of the point of zero charge (PZC) (Bakatula et al., [2018\)](#page-17-5). 0.2g of soil and soil–biochar mixture was added to a series of 25 mL tubes containing 15 mL of 0.05 M NaNO<sub>3</sub> solution whose pH (pH<sub>i</sub>) was adjusted in the range 2, 3, 4, 5, 6, 7, 8, 9 and 10 with 0.1 M HNO<sub>3</sub> and 0.1 M NaOH. The tubes were agitated for 24h on a head-on agitator at 200 rpm, then left to settle. The pH (pH<sub>f</sub>) of the supernatant in each tube was then measured. The PZC were obtained after plotting a graph of pHf-pH<sup>i</sup> against pHi, that is, at the pH at which the curves cuts the  $p_{i}$ axis.

### 2.3.1 Sorption phosphorus study experiment soil–biochar samples

To determine sorption characteristics, related to soil– biochar properties and elucidate the mechanisms involved in sorption, the following experiments were performed. In this study, 0.2 g of each sample was mixed with 10 mL of  $KH<sub>2</sub>PO<sub>4</sub>$  solution containing 0, 5, 10, 20, 80, 160, 320 and 480mg/LP and 0.01M KClasbackground electrolyte in 25 mL centrifuge tubes. Then two drops of chloroform were added in each sample to act as microbial growth in- hibitor. The tubes were set on an end-end shaker for 16 h at 150 turns per minute. At the end of the shaking process, the samples were centrifuged at 5000 rpm for 10 minutes then filter through a Whatman No.5 filter paper to obtain a clear extract. The clear extracts were analysed for avail- able P following Murphy and Riley [\(1962\)](#page-19-13). Each adsorp- tion process was done in quadruplets. The quantity of P adsorbedat equilibriumby treatedsoil sampleswas calcu- lated using equation [\(1\)](#page-5-0) below

$$
Q_{\rm e} = \frac{(C_i - C_{\rm e}) \, x \, V}{m} \tag{1}
$$

Where *Q*<sup>e</sup> (mg/kg) is the quantity of P adsorbed by treated soil samples, *C*<sup>i</sup> (mg/L) the initial concentration of P, *C*<sup>e</sup> (mg/L)the quantityof P at equilibriumafteradsorption,*V* (L)thevolumeofPsolutionusedforadsorptionand*m*(kg) the mass of amended soil sample.

The experimental data of the quantity of P adsorbed at equilibrium were fitted into two important isotherm models namely Langmuir and Freundlich. Each isotherm is characterized by definite constants whose values express the surface properties and affinity of the amended soil samples. The nonlinear form of Langmuir isotherm model used is represented by equation [\(2\)](#page-5-1) which describes single-layer adsorption:

<span id="page-5-1"></span>
$$
Q_{\rm e} = \frac{Q_{\rm max} K_{\rm L} C_{\rm e}}{1 + K_{\rm L} C_{\rm e}} \tag{2}
$$

Where,  $Q_e$  (mg/kg) is the quantity of P adsorbed by the soil or soil/biochar mixture, Ce (mg/L) the quantity of P at equilibrium after adsorption, Omax the P adsorption maximum for Langmuir model (mg/kg),  $K<sub>L</sub>$  the equilibrium constant that determines the binding energy  $(L/kg)$ , the higherthe  $K_L$  the stronger the binding force.

The nonlinear form of Freundlich is given in equation [\(3\)](#page-5-2) below which describes multi-layer adsorption

<span id="page-5-2"></span>
$$
Q_e = K_F C_e^{\frac{1}{n}}
$$
 (3)

*n*

Where  $Q_e$  (mg/kg) is the quantity of P adsorbed by the soil or soil/biochar mixture,  $C_e$  (mg/L) the quantity of P at equilibrium after adsorption,  $K_F$  (mg/kg) the Freundlich constant indicating adsorption capacity not specifically the maximum adsorption capacity, and the adsorption intensity which indicates the heterogeneity of the material (Cucarella & Renman[,2009\)](#page-18-13).

### 2.3.2 Thedesorptionof phosphorus experiment

In order to evaluate the reversibility of phosphate sorbed onto soil and soil/biochar mixtures, their desorption characteristics were also determined.In the first phase, 10 mL of 80 mg/L of P was shaken with 0.2 g of soil or soil/biocharmixture for 16honan end-to-end shaker.Atthe end of the sorption experiment, the supernatant wasremoved; the amended soil samples were obtained as the residue and dried at room temperature. Then in the second phase, the desorption experiment of P from the amended soil samples from the dried residue was done in the following way, 0.1 g of the soil and soil/biochar was mixed with 10 mL of 0.01 KCl and agitated for 16 h, then centrifuged and the supernatant was analysed for the determination of P (Murphy & Riley, [1962\)](#page-19-13). The desorption percentage was calculated using equation [\(4\)](#page-5-3)below:

<span id="page-5-0"></span>Desorption percentage (
$$
\% = \frac{P_{desorbed}}{P_{sorted}} \times 100
$$
 (4)

<span id="page-5-3"></span>Where,  $P_{\text{softed}}(mg/kg)$ , is the quantity of P adsorbed at equilibrium and  $P_{\text{desorbed}}(mg/kg)$  is the quantity of P desorbed.

Nonlinear regression analysis was used to fit Langmuir and Freundlich isothermal equations to data. Curve fitting and Statistical analyses were done using Origin 8.5. The goodness of fit was evaluated based on coefficient of determination ( $R<sup>2</sup>$ ). The significant difference between the factors means for parameters under analysis were determined at a 5% level of significance ( $\alpha$  = .05).

#### **RESULTS AND DISCUSSION**

#### **3.1 Properties of biochar, soil andsoil– biochar mixture**

#### 3.1.1 Biochar

Properties of biochar are well discussed in our previous work (Pouangam Ngalani et al., [2022\)](#page-19-7). In summary, biochar characterizations reveal that its properties depend on the biomass type and pyrolysis temperature. Biochar produced from cocoa pod husk had a greater liming potential as compared with coffee husk andthe trend increases with pyrolysis temperature. All four biochars had pH above 9 andalarge ashcontentbecauseofthepresence of calcite,

kalinite and quartz. The acid-neutralizing capacity of CP biochar was significantly (*p* <.05) more than that of CH biochar irrespective of pyrolysis temperature. So we can hypothesize a greater  $H<sup>+</sup>$  consumption by CP biochar in acid soils as compared with CH biochar pyrolysed at the

same temperature. Available P is greater in biochar produced from CP than that produced from CH irrespective of pyrolysis temperature.

Scanning electron microscopy images of biochars produced at 350 and 550°C are shown in Figure [1](#page-8-0) at two different magnifications (2000 and 3500). The biochars displayed a heterogeneous surface morphology and a complex structure. The structure presented cracks, pores and crevices with irregular forms, randomly distributed and of different diameters. Biochar produced at 550 °C has rougher surfaces because of the presence of white particles resulting from the accumulation of minerals formed at higher temperatures. Pores on CP biochar were more regular in size as compared with CH because of the greater crystallinity of CP biochar. The presence of pores, cracks and crevices has possible effects on soil water retention, rooting patterns, soil aeration, nutrients sorption and habitat for soil biota (DeLuca et al., [2015;](#page-18-10) Lehmann et al., [2011;](#page-18-14) Sheng & Zhu, [2018\)](#page-19-15).

The SEM analysis was coupled with EDX analyses for chemicalmapping of elements in different biochar samples and results obtained are presented in Table [1.](#page-8-1) In Table [1,](#page-8-1) we observe an increase in C content and reduction in O with increasing pyrolysis temperature. The O/C ratio as determined with SEM–EDX decreases with rising pyrolysis production temperature of the biochar. This result is an accordance with previous results summarized in a review by Ahmed et al. [\(2016\)](#page-17-6). Other elements present were P, Mg and Ca whose presence in biochars makes them a potential for liming (Chintala,Schumacher,et al.[,2014\)](#page-18-7).Thepresenceofindium(inlargeamounts)maybebecauseitisatraceelement found in the soil, especially unpolluted soil, that can be ab- sorbedintoplantsandhencefoodchain(Adeniyietal., 2019).

#### 3.1.2 Physicochemical properties of amended soil samples

From Table 2, after 7 days of incubation, CH350, CH550, CP350 and CP550 at rate of 0, 20, 40 and 80g/kg raised the soil pH from 4.7 to 5.1, 5.7 and 6.5; 5.4, 6.3 and6.5; 5.2, 5.9 and 6.7; 5.7, 6.7 and 7.9 respectively. Also, after 60 days of incubation, CH350, CH550, CP350 and CP550 at rates of 20, 40 and 80g/kg raised the soil pH from 4.7 to 5.3, 5.6 and 7.3; 5.6, 6.2 and 7.5; 5.5, 6.0 and 7.1; 5.9, 6.7 and 8.2 respectively. It was observed that an incre- ment in soil pH rises from 7 days to 60 days.Moreover, the pH of soil–biochar mixtures increased with rising in pyrolysis temperature and with biochar application rate with CP having the greater impact compared with CH. The application of biochar on soil had a significant effect  $(p < .05)$  in increasing the soil pH. Soil pH increases because of addition of biochar, were attributed to increased,  $CaCO<sub>3(eq)</sub>%$  content and acid-neutralizing capacity of the biochar.Thepresence of silicate, carbonate, andbicarbonate minerals and negatively charged phenolic, carboxyl and hydroxyl groups on biochar surfaces also increased pH. At higher temperature, the silicates, carbonates and bicarbonates are mostly the cause of elevation in pH while at low-temperature produced biochar, the basic functional groupsonthebiochar suchas–COO- and–O- arerespon- sible (Yuan et al., [2011\)](#page-20-10).

An increase in EC was observed to be greater in soil– biochar mixed with CP than CH for the same amendment rate and pyrolysis temperature. Details from Table [2](#page-9-0) indicates that, after 7 days of incubation with 20, 40 and 80 g/kg amendmentssoil ECincreased to 116, 211 and 303µScm<sup>-1</sup> for CH350, 141, 258 and 345 µS/cm for CH550, 162, 317, 321  $\mu$ S/cmforCP350 and finally 200,343 then 403 $\mu$ S/cm forCP550 respectively as compared with control. This in- crease was inthe order CP550>CH550>CP350>CH350. Moreover, from 7 to 60 days of incubation, CH350, CH550, CP350 and CP550 at rates of 20,40 and 80g/kg increased the soil EC to 151, 259 and 596; 181, 317 and 630; 204, 364 and 642; 263, 496 and 872 respectively. The sharp increase in EC from the 7th to the 60th day of incubation is because of the dissolution of kalicinite and calcite by reaction with  $H^+$  in the acid soil solution to release calcium and potassium ions.

The application of biochar to acid soil samples re- sulted in an increase in available P as compared with the control. After 7 days of incubation, CH350, CH550, CP350 and CP550 at the amendment rates of 20, 40 and 80 g/kg raised the available P by 23.9%, 40.6% and 147.5%;24.0%,36.0%and32.0%;37.0%,49.7%and167.2% and 33.3%, 48.9% and 181.2% respectively.The increase was intheorderCP550>CP350>CH350>CH550.From 7 to 60 days of incubation, at an amendment rate of 20 and 40 g/kg, an increase in available P was observed; which can be attributed to; biochar as a source of solu- ble P, its reaction with complexing metals  $(Al^{3+}, Fe^{3+}$  and  $Fe^{2+}$ ), releasing P and promotion of microbial activity. Whereas, at 80  $g/kg$ , a decreases in available P was ob- served which could be due to the formation of Ca-P pre- cipitates attributed to the presence of  $CaCO<sub>3</sub>$  found in the large amounts of ash minerals in the biochar (Hollister et al., [2013;](#page-18-15) Wang et al., [2015\)](#page-20-11).

A marked decrease in exchangeable acidity, Ex Al and Fe was observed with incubation time (Table [2\)](#page-9-0). With respect to exchangeable acidity, after 60 days of incubation the amendment rate of 20, 40 and  $80 g/kg$  led to a significantdecreasewhichwasgreaterforCP550(93.4%,98.1% and98.8%),followedbyCH550(87.8%,96.2%and98.7%), then CP350 (85.6%, 92.8% and 96.2%) and finally CH350 (77.4%, 90.3% and 97.5%). After 7days of incubation, the drop in Ex acidity, Ex Al and Fe from the application of bio- char was in the order  $CP_{550}$  > CH<sub>550</sub> > CP<sub>350</sub> > CH<sub>350</sub>. The increase in amendment rate, had a significant drop in Ex Acidity, Ex Al and Ex Fe in the acid soil sample. The cause of reduction can be attributed to the precipitation of the metallic ions by alkaline oxides, carbonates and silicates in the biochar and complexation with organic functional groups (Qian et al., [2013\)](#page-19-16). Recent studies of the application of biochar to soils and its effect on soil acidity and Ex Al and Fe made the same observation (Chintala, Mollinedo, et al., [2014;](#page-18-5) Dume, Ayele, et al., [2017\)](#page-18-16).



<span id="page-8-0"></span>**F IGURE 1** Scanningelectronmicrographs andenergydispersive spectroscopycurves ofbiochars from CHandCPpyrolysed at 350 and 550°C.

Sample C		$\Omega$	$\mathbf{P}$	$Mg$ Cl	Si	${\bf Al}$	Fe Nb	In	$\mathbf S$	Sb	Au	O/C
CH350	$47.87$ 20.05 $/$ 0.18 0.09 $/$ 0.24						$\qquad \qquad$	$\frac{1}{26.77}$ 0.25 3.87 0.69 0.42				
CH550	43.87 17.39			(0.39)(0.510.60)				$32.42$ /		4.83		0.40
CP350	47.96 31.93 0.32			$0.85$ /		$0.38$ $0.50$ $0.12$ $0.75$		14.82  0.12  2.24				0.70
CP <sub>550</sub>		55.08 25.72 0.27 1.36				$0.23$ $0.25$ $0.09$ /		14.45	0.16 2.40			0.47

<span id="page-8-1"></span>**TABLE 1** Elemental compositions (%) obtain from SEM-EDS analysis and the O/C ratio.

An increase in SOC in soil amended with biochar was observed as compared with the control (Table [2\)](#page-9-0). The increase in SOC increased with incubation time and amend- ment rate. Elevation in the amount of SOC was more pronounced when amended with CP than CH biochar and for biochar produced at 350°C than when produced at 550°C.An increase insoil organic carbon isbecause of the presence of recalcitrant carbon (Nyambo et al., [2018\)](#page-19-17) and a decrease in the mineralisation of the soil carbon because of sorption of labile soil organic matter onto the biochar particles (Singh & Cowie, [2014\)](#page-19-18) and/or a very short-term inhibitory effect of microbial activity of biochar-associated volatile organic compounds (Spokas et al., [2011\)](#page-20-12).



<span id="page-9-0"></span>**TABLE 2** Physicochemical properties of soil and soil–biochar mixture after amending for 7 and 60 days.

#### **3.2 pH of point of zero charge**

The PZC is the pH value for which the net charge because of sorbed ions, other than  $H^+$  and OH $^-$  is equal to zero (Sposito, [2008\)](#page-20-13). Figure [2](#page-10-0) shows results of the determination of PZC and it is observed that biochar type amendments to soil increase the  $\text{pH}_{PZC}$  as compared with the control in the order SCP550 > SCH550  $>$  SCP<sub>350</sub>  $>$  SCH<sub>350</sub>. The increment in pH<sub>PZC</sub> to basic pH values increased with amendment rate for a constant incubation period. The increment in pH<sub>PZC</sub> after 7 days of incubation for amendment rates 20,40 and 80 g/kg was in the order 0.44, 0.57 and 1.68 for SCH350, 0.55, 1.02 and 2.01 for SCP350, 0.50, 1.0 and 1.98 for SCH550 and 0.68, 1.76 and 2.32 for SCP550. These same increments was observed for soil– biochar incubated for 60 days with increase in amendment rate in the order 0.78, 0.98 and 2.19 for SCH350, 0.97, 1.55 and 2.32 for SCP350, 0.87, 1.55 and 2.26 for SCH550 and finally 1.09, 1.81 and 2.76 for SCP550. Comparing  $pH_{PZC}$  for all types of biochar amendments and same incubation rate, there was a rise in  $pH_{PZC}$ 

from 7 to 60 days of incubation. Research has demonstrated that when pH is higher than the pH<sub>PZC</sub> of a variable charge soil, the soil possesses a net negative charge on the surface and the potential of the adsorption plane is negative (Xu et al., [2016\)](#page-20-14). In this light, we can estimate that adding biochar to soil consider- ably varied the surface charges of the soil resulting in the  $\rm pH_{PZC}$  greater than the  $\rm pH_{PZC}$  of the unamended soil. Thus on moving towards basic pH, one can expect soil–biochar mixture having a more and more negatively charged sur- face. This can be attributed to the presence of –COO- and –O- from the biochar and thus may repel negative ions.



<span id="page-10-0"></span>**F IGURE 2** Plots of∆pH vs.pHifor the determination ofpoint of zero charge for amended soil sample at 20,40 and80 g/kg incubated for 60days.

#### **3.3 Phosphorus sorption**

This phosphorus sorption study is aimed at determining the amount of P that the soil, soil–biochar adsorb in solution with different concentration of P at equilibrium. The adsorption data fitted in isotherms in other to elucidate the mechanism of adsorption and estimate the adsorption maximum, adsorptioncapacity andadsorptionenergies.

#### 3.3.1 Adsorptionofphosphorusonsoil

Figure [3](#page-11-0) represents the sorption curve for the adsorption of P onto soil incubated for 7 and 60days. A rapid increase in the adsorption capacities of both soil samples is observed (because of incomplete occupation of adsorption sites), followed by a gradual slowdown for greater concentrations, until the formation of a plateau indicating near saturationofthe soil samples is reached.This implies that the vacant adsorption sites for P declines as the concentration ofthe adsorbate rises (Enang et al.[,2019\)](#page-18-17) as the result of strong affinity of the soil to P at small concentrations, which will decrease as concentration rises (Sparks, [2003\)](#page-20-15). For a more systematic study, the experimental data were interpreted using Langmuir and Freundlich isotherms and the parameters obtained are presented in Table 3. The experimental data of the sorption of P on soil samples fits into the two models (*R* <sup>2</sup> > 0.9) suggesting that the P adsorption is controlled by both Langmuir and Freundlich

adsorption mechanisms. This result indicated that the P adsorption could be governed by multiple mechanisms. Adsorption of P on Cameroonian acid soil had let to different conclusion as to thebestfitisotherms.Previous workonthefixationof P on soil of the Eastern flank of Mount Cameroon displayed a best fit with a Freundlich model even though both isotherms had a coefficient of determination greater than 0.9 (Mbene et al., [2017\)](#page-19-0). Comparing the P sorption maximum obtained from a Langmuir isotherm of the unamended soil (i.e. amendment at 0 g/kg of biochar), incubated for 7 and 60 days, indicated a slight increase in  $Q_{\text{max}}$  and a decline  $\mathcal{K}_L$  of P with increase in incubation days after wetting the soil samples. We can suggest that, when dry soils are rewet, P mineralisation occurs causing P more available to the soil solution (Laboski & Lamb, [2003\)](#page-18-18), thus resulting in a rise in *Q*max. Moreover, the P sorption maximum values were more than those obtained from experimental data, indicating that adsorption sites were not all occupied by P with similar results obtain by Njoyim et al. [\(2016\)](#page-19-19). Also, a de- cline in binding energy is observed which may indicate a weakattachmentof P on the soil surface of S0 after 60 days of incubation. From the Freundlich isotherm parameters, the adsorption capacities and sorption intensity were quite similar.A slight increase in sorption intensity with the soil sample with increasing number of incubation days (0.2570 for S0, 7 days and 0.2671 for S0, 60 days) may tend to prove that S0 at 60 days of incubation have a greater affinity to P adsorption.



<span id="page-11-0"></span>**FIGURE 3** Langmuir and Freundlich phosphorus sorption isotherms of soil samples incubated for 7 and 60 days.

<b>Material</b>	Incubation		Langmuir		Freundlich			
	days	$K_{L}(L/kg)$	Qmax(mg/kg)	R <sup>2</sup>	1/n	$K_F$ (mg/kg)	R <sup>2</sup>	
S <sub>o</sub>	,	0.0418	2808.97	0.921	0.2570	610.15	0.986	
So	60	0.0359	3047.82	0.918	0.2671	613.33	0.9679	

**TABLE 3** Langmuir and Freundlich parameters for unamended soil (S0).

#### 3.3.2 Adsorptionofphosphoruson soil–biochar

#### *Langmuir isotherm model*

Figure [4](#page-13-0) represents the graph with experimental data fit- ted to Langmuir curves for the adsorption of P onto soil amended with biochar produced from CH and CP at 350°C and 550 °C incubated for 7 and 60 days at room temperature. Typical isotherms profile curves and plotted data were observed as in other works (Chintala, Schumacher, et al., [2014;](#page-18-7) Xu et al., [2014\)](#page-20-7). From Figure [4,](#page-13-0) we can ob- serve that the plateaus of the amended soil samples were below the control for all four biochar additions and ir- respective of the incubation periods. This indicates that the adsorption capacities and activity of the adsorption sites were reduced. A logical explanation can be that P released from biochar was sorbed to the soil surface and filled sorption sites, some of which are reversible (Sato & Comerford, [2006\)](#page-19-20). This situation leaves fewer avail- able P sorption sites, reducing the total capacity for the sorption of P from external sources. Thus this study illustrates that biochar can reduce the soil'sP-fixingpotential. From Figure [4,](#page-13-0) we observed that, the level of the plateau for all the four biochar declines with increase amendment rate from 20 to 80g/kg, thus indicating a reduction in adsorption capacities with increase amendment rate. From the Langmuir isotherm parameters, P sorption maximum declines with increasing amendment rate for all four biochar types. For example, *Q*max are 2267, 2048 and 1823 mg/kg for amendments with CH350 and 2122, 20,322 and 1904 mg/kg for amendments with CP350 at amendment rates of 20, 40 and 80 g/kg for 7 days respectively. Studies have shown that adding biochar reduces the sorption maximum of P (Eduah et al., [2019\)](#page-18-8), but at greater biochar amendment rates the sorption maximum is more than the unamended soil (Xu et al., [2014\)](#page-20-7). From these observations, it appears that the amendment rate of the biochar in this study was not sufficient to let the soil– biochar mixture to adsorb P more than the control. It was observed that, when the biochar rate increases, the pH of the soil–biochar mixture rises (Table [2\)](#page-9-0), which affects the adsorption of P (Haynes, [1982\)](#page-18-19) because of the formation of more negative charges on the surface of the soil–biochar samples as confirmed by the PZC analysis, thus increasing repulsion of anionic P forms (Chintala, Schumacher, et al., [2014;](#page-18-7) Murphy & Stevens, [2010\)](#page-19-21). It is observed that, soil amended with biochar produced at higher temperature had a reduced P sorption maximum for all four biochars. The EC of amended soil was found to increase with biochar produced at higher temperature (Table [2\)](#page-9-0) which will increase the ionic strength of solution and may reduce the positive electric potential of the soil surface through a screening effect and ultimately reduce the P sorption (Chintala, Schumacher, et al., [2014\)](#page-18-7). At amendment rates of 20, 40 and 80 g/kg after 7 days of incubation,  $Q_{max}$  for SCH350 were 2267, 2048 and 1823 mg/kg which increased to 2407, 2112 and 1990 mg/kg after 60days of incubation. This tendency was observed for all four biochar additions with respect to increased incubation days. In general, it was observed that soil–biochar with CP biochar had a lesser P sorption maximum as com- pared with those incubated with CH biochar for 7 days of incubation, whereas, at 60 days of incubation the adsorption maximum were more or less the same. Other studies, however, showed reduction in phosphate sorption in acidic soils as a result of biochar application because of precipitation of  $Al^{3+}$  and Fe<sup>3+</sup> (sites for phosphate complexation) (DeLuca et al., [2015\)](#page-18-10) and increase in repulsion by the negative charges newly created in the soil, by -O<sup>−</sup> and –COO<sup>−</sup> from biochar (Martínez et al., [2017\)](#page-18-11). This can be confirmed with the rise in soil organic carbon content (Table [2\)](#page-9-0). In this study, we observe that biochar added to soils serves as a source ofPrather thanasasink, resulting ingreateravailabilityofPtosoilswithincreasingbiochar rate.

<span id="page-12-0"></span>Binding energy data (Table [4\)](#page-12-0) obtained from Langmuir isotherms showed a decline with increasing amendment rate irrespective of biochar type and number of incubation days. For example, soil amended with CH550 for 60 days of incubation had a drop in binding energy from 0.0497, 0.0410 and 0.0303 L/kg and for SCP550 after 7 days of

incubation; the drop was from 0.0318 to 0.0225 then 0.0134 L/kg for amendment rates of 20, 40 and 80 g/kg respectively. Also, the binding energy increase in the soil– biocharmixture amended with biocharproduced at lower temperature, that is, in the order SCH<sub>350</sub> > SCH<sub>550</sub> and for SCP<sub>350</sub> > SCP<sub>550</sub>. Also, binding energy increased with increasing number of incubation days for each biochar type and corresponding amendment rate. For example, the increase in binding energy from 7 to 60 days for the soil–biochar mixture amended with 20 g/kg of CH550, was 0.0369 to 0.0497L/kg, 0.0278 to 0.0410 for 40L/kg and then from 0.0256 to 0.0303L/kg for 80g/kg and the case of SCP<sub>550</sub> the binding energy was 0.0318 to 0.0352 L/ kg, 0.0225 to 0.0353 for 40g/kg and then from 0.0134 to 0.0217 L/kg for 80 g/kg. The order of decreasing binding energy of soil–biochar samples suggests that P can be easily desorbed. These results suggest that, compared with active sites in the original acidic soil, newly developed hydr(oxide) in the biochar-amended soil might retain phosphate ions more loosely (Baninajarian & Shirvani, [2020;](#page-17-8) Xu et al., [2014\)](#page-20-7). Increased amendment rates in soil– biochar mixtures result in an increase in pH, reduced concentrations of free Fe, Al and Mn oxides which may be transformed to Fe,Al and Mn hydroxides and reduced the availability of high energy sorption sites (Stevenson & Vance, [1989\)](#page-20-16). Generally, phosphates are strongly fixed to acidic soils not only through chemisorption on high- energy surfaces of Fe and Al oxides and hydroxides but also via precipitation of Al and Fe phosphate. However free Al and Feconcentrations in biochar-amended soil are lessened (Table [2\)](#page-9-0) thereby reducing the availability of high energy sorption sites for phosphate ions (Dume, Tessema, et al., [2017\)](#page-18-20).

In general, it was observed that soil–biochar mixtures with CP had a smaller P sorption maximum as compared with those incubated with CH after 7 days of incubation, whereas, at 60 days of incubation the adsorption maximum were more or less the same. For all biochar, the P sorption maximum obtain from a Langmuir isotherm model were greater than from experimental data indicating that all the biochar sites were not occupied by P.



<span id="page-13-0"></span>**F IGURE 4** Langmuir phosphorus sorption isotherms for SCH350,SCH550,SCP350 and SCP550 at amendment rate of 0, 0, 40 and 80 g/kg incubated for 7 and 60days.



### **TABLE 4** Langmuir and Freundlich parameters for soil-biochar samples incubated for 7 and 60 days.

#### *Freundlich isotherm model*

from CH and CP at 350°C and 550°C incubated for 7 and 60 days at room temperature. This simulated curve profile, is Figure [5](#page-15-0) represents simulated Freundlich curves for the adsorption of P onto soil amended with biochar produced similar to the Freundlich curve for So. From Table [4,](#page-12-0) it is observed that the sorption capacities  $(K_F)$  of soil–biochar mixtures are less than the control (S0), which can be related to the lesser P sorption maximum for soil–biochar mixtures Also, a decline in sorption capacity with increasing incubation rate for all the biochar types, irrespective of incubation period, was . observed. As an example, at 60 days of incubation, soil amended with CH350 showed a decrease in sorption capacity from 535, 460 to 286mg/kg and with CP350 amendment, the decrease was from 483, 418 to 274mg/kg with increases in amendment rate of 20, 40 and 80g/kg respectively. Soil amended with biochar pyrolysed at higher temperature, had a significant decrease in P sorption capacity. Moreover it was noted that increase in incubation days from 7 to 60 days increases P sorption capacity of the soil–biochar samples for all biochar types. For example, comparing the rise in sorption capacity between 7 days and 60days of incubation for SCH350 was 459 to 535 for 20 g/kg,382 to459for40g/kgand302to288for80g/kg. The sorption intensity is used to describe the heterogeneity ofthe adsorption surface. The affinity of P adsorption on soil–biochar increases as  $\approx$  1.



<span id="page-15-0"></span>**F IGURE 5** Freundlichphosphorus sorptionisotherms forSCH350,SCH550,SCP350andSCP550atamendment rateof0,20,40and 80g/kg incubated for 7 and 60days.

[\(2014\)](#page-20-7) incubated acidic soil samples with wheat straw biochar. The decrease in sorption capacity of the soil– biochar From Table [5,](#page-16-0) the sorption intensity of SCH350, SCH550, SCP350 and SCP550 is slightly greater than for S0 after 7 days of incubation, while for 60days ofincubation,the sorption intensity of SCH350, SCH550, SCP350 and SCP550 at rate of 20 and 40g/kg presents a relatively smaller or the same sorption intensity with S0, but with amendment rate of  $80g/kg$ , the sorption intensity was greater, while for CP350 and CP550 soil amendments, the sorption intensity were greater than for S0 for all amendment rates. Increasing the number of incubation days; decreases the sorption intensity of P onto the soil–biochar mixture. Thus for greater values, the adsorption of phosphate to soil–biochar mixture was favourable and happened on heterogeneous surfaces. Previous studies were consistent with this observation as reported by Yao et al. [\(2011\)](#page-20-17), who indicated a low adsorption capacity with biochar derived from sugar beet tailing same as Xu et al. mixture which is closely related with sorption maximum from Langmuir data can be due to variation in

physicochemical properties of the samples. An increase in pH <sup>4</sup> and PZC of the samples, will lead to increase in negative charge surfaces, thereby increasing repulsion, thereby reducing P sorption capacity (Murphy & Stevens, [2010\)](#page-19-21). Higher pH depresses the formation of HPO <sup>2−</sup>, which is preferentially adsorbed by soil colloids (Xu et al., [2014\)](#page-20-7).

			7 days		60 days		
Material	Amendment rate	$\boldsymbol{P}_{\text{sorted}}$	$P_{\rm desorbed}$	$DP$ (%)	$\boldsymbol{P}_{\text{sorted}}$	$P_{\rm desorbed}$	DP(%)
So	$\mathbf{0}$	1571.19	429.00	27.30	1516.16	412.45	27.20
	20	1223.21	384.62	31.44	1299.33	376.69	29.00
SCH <sub>350</sub>	40	1164.72	392.05	33.66	1113.33	368.32	32.63
	80	1017.58	412.37	40.52	894.49	346.67	38.76
	20	1213.08	452.01	37.26	1216.33	375.85	30.90
SCH <sub>550</sub>	40	1146.62	389.28	33.95	1076.17	360.69	33.52
	80	846.79	394.52	46.59	944.18	372.41	39.44
	20	1205.46	392.95	32.60	1186.92	373.25	31.45
SCP <sub>350</sub>	40	1137.60	375.57	33.01	1058.28	366.95	34.67
	80	882.53	386.37	43.78	890.86	361.73	40.60
	20	1161.99	381.29	32.81	1205.12	365.76	30.35
$\rm{SCP}_550$	40	1052.36	360.35	34.24	1013.20	385.49	38.05
	80	858.76	364.95	42.50	820.84	332.06	40.45

<span id="page-16-0"></span>**TABLE 5** Percentage desorption of P from soil and soil–biochar mixture.

## **3.4 Phosphorus desorption from soil and soil–biochar mixture**

The extent of desorption of P from these samples can indicate the degree of P availability in the soil solution and to plants. In this work, the P desorption percentage was used to evaluate the quantity of P desorbed from soil and soil– biochar mixtures as presented in Table [5.](#page-16-0) Results showed that, the amount and percentage of P desorbed were influenced by biochar type, biochar production, the rate of amendment and the number of incubation days. P desorption from biochar amended soils were greater than unamended soil. This is because of the fact that during amendment of soil with biochar, P is released from bio- char and sorbed onto strong adsorption sites which is not reversible (Sato & Comerford[,2006\)](#page-19-20). This process leaves fewer adsorption sites forP,thereforenewly sorbedP will be on low energy adsorption sites, which may ease desorption. Also, the presence of acid-functional groups such as carboxylic and phenolic groups on the biochar causes P removal from clay minerals through ligand exchange andor enhancedliganddissociationofoxidesandhydroxides of Fe and Al (Kirk et al., [1999\)](#page-18-21). Cocoa pod biochar amended soil had a greater P desorption percentage than CH biochar amended soil samples. Soil–biochar mixtures with high temperature pyrolysis biochar desorbed P more than the low pyrolysis temperature biochar at amendment rates of 40  $g/kg$  and 80  $g/kg$ . Whereas at 20  $g/kg$ , there was no significant change in P desorption. It was observed that higher pyrolysis temperature biochar adsorbed less than low pyrolysis temperature biochar for the same amendment rate but desorbs more. Increase in amendment rate, elevate the desorption percentage of P from the soil– biochar mixtures. The desorption percentage increased by more about 10% for all biochar types from 20 mg/kg to 80 mg/kg amendment rate.An increase in incubation days for soil–biocharmixtures resultedinadecline inde- sorption percentage of P. In all cases, an increasing P de- sorption percentage, was related to a decline in binding energy for soil–biochar samples as can be seen in Table [5.](#page-16-0) Previous research corroborated the assertion that a decrease in

binding energy increased desorption of P which was attributed to increase in soil pH with biochar application (Eduah et al., [2019\)](#page-18-8).

#### **4 CONCLUSION**

This work demonstrates that acid soil amended with biochar produced at different pyrolysis temperatures influenced the soil chemical properties dissimilarly and decreased soil P sorption capacity as compared with the control. The incubation study of acid soil revealed an increase in soil pH and electrical conductivity with application of biochar at different incubation rates. A marked reduction in exchangeable acidity, Al and Fe occurred after incubation. Thus biochar shows a potential for mitigating soil acidity. Furthermore, phosphate sorption by these amended soils was satisfactorily described by Langmuir and Freundlich models. A reduction in P sorption capacity was observed as compared with the control. The desorption process of P from soil–biochar mixtures was enhanced at greater biochar rates and pyrolysis temperature. From this study we can suggest that P retention and availability can be modulated when biochars are applied at a convenient rate and pyrolysed at an appropriate temperature of about 550 °C. Thus, biochar can be appropriate for P management practices for acid soils in the Western region of Cameroon. Field study with locally produced biochar should be directly applied to farmlands in other to evaluate the fate of ap- plied phosphate fertilizers.

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