# Growth Performance of Cowpea in Spent Oil-Contaminated Soils Ameliorated with Cocoa Shell Powder and Biochar

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Received: 27.07.2018; Accepted: 19.11.2018; Published Online: 30.12.2018

#### ABSTRACT

The study assessed the ameliorative potentials of cocoa shell powder and biochar on spent engine oil (SEO) soils using the growth performance of cowpea. Twenty-four polyethylene bags were set up consisting of seven treatments ( $T_1$  to  $T_7$ ) contaminated with 2% v/w SEO and control ( $T_0$ ) each replicated three times. Cocoa shell biochar (CSB) was applied to  $T_2$ ,  $T_3$  and  $T_4$  at rates 0.25%, 0.5% and 1.0% while uncharred cocoa shell powder (CSP) was incorporated into  $T_5$ ,  $T_6$  and  $T_7$  at rates 0.25%, 0.5% and 1.0%. Chemical properties of CSB, CSP and soil treatments were determined by standard methods. Cowpea seeds were sown and germination and growth parameters were determined at 3 and 6 weeks after sowing. The result showed CSB was alkaline and rich in exchangeable cations. SEO-contamination negatively impacted soil nutrient composition, weakened germination by 27% and negatively affected growth of cowpea. Plants in  $T_0$  had significantly highest growth and biomass. CPB (especially 1%) amendment significantly improved leaf initiation and area compared with plants in  $T_1$ . Growth declined with increasing CSP amendment. In conclusion, conversion of cocoa shells to biochar is necessary eliminate the acidic effects of the raw cocoa shell and effectively condition the soil.

Keywords: Contaminants, Cowpea, Engine oil, Growth, Soil amendment, Soil nutrient

#### **ABBREVIATIONS**

CSB – cocoa shell biochar CSP –cocoa shell powder (uncharred) LA – Leaf area PAH – polycyclic aromatic hydrocarbon SEO – spent engine oil TPH – total petroleum hydrocarbon TOC – total organic carbon WAS – weeks after sowing

### **INTRODUCTION**

Soil contamination or pollution, the alteration of the soil's natural environment by man-made chemicals (Panagos *et al.* 2013), is increasing affecting cultivation and yield of crops in many parts of the world. Soil pollution has been attributed to a myriad of sources including accidental oil spills, oil and fuel dumping, application of fertilizers and pesticides, illegal dumping of refuse, disposal of ammunitions and agents of war, disposal of electronic and nuclear wastes (Panagos *et al.* 2013, SCU-UWE 2013). Pollution from spent engine oil, otherwise known as waste engine oil or spent lubricant oil, is one of the widespread environmental problems in Nigeria and surpasses crude oil pollution (Atuanya, 1987, Odjegba and Sadiq 2002). This problem has been linked to indiscriminate dumping of engine oil (a petroleum product used to reduce friction in engines) into gutters, drainages and open plots by mechanics after servicing and subsequent draining from automobile and generator engines (Odjegba and Sadiq 2002). Oil-contaminated soils are of serious environmental concern as they are unsuitable for agricultural purposes due to heavy metals toxicity, poor wettability and low nutrient mobilization (Panagos *et al.* 2013). The effects of spent oil – contaminated soils on the performance of crops including Amaranthus (Odjegba and Sadiq 2002), cowpea (Kayode *et al.* 2009, Lale *et al.* 2014), and maize (Okonokhua *et al.* 2007, Kayode *et al.* 2009) have been well documented.

Soil amendment as a strategy for remediation is a long standing procedure aimed at reducing the risk of pollutant transfer to receptor organisms or proximal waters (Beesley *et al.* 2011). Organic materials are a popular

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choice for amendment due to their biodegradable nature that often require little pre-treatment prior to direct application to soils. Soil amendment also serve as a suitable means for disposing surplus organic residues. Carbon rich amendments, such as activated carbons and biochars, have been deployed for soil and sediment restoration purposes due to the high sorption ability (Brändli et al. 2008, Cho et al. 2009, Beesley et al. 2011) and environmental safety. Unlike activated carbons, biochar is a specialized form of charcoal derived from the pyrolysis of biological residues such as wood, plant matter, manures and suitable for use in soils (Beesley et al. 2011, Pires 2015). Biochar serves as a catalyst that enhances plant uptake of nutrients and water (among others) while enhancing raw organic materials supply of nutrients to plants and soil microorganisms (Hunt et al. 2010). The nutrient enhancement of biochar make it suitable for remediation processes. Zheng et al. (2010) have demonstrated that the use of biochar, in sustainable agriculture, as a means of improving soil fertility while reducing dependence on chemical fertilizer. Biological nitrogen fixation and beneficial mycorrhizal relationships in common beans (Phaseolus vulgaris) upon biochar applications have been documented (Rondon et al. 2007, Warnock et al. 2007). Despite the numerous advantages of biochar as soil amendment, decreased plant growth due to temporary levels of pH, volatile/mobile matter, and/or nutrient imbalances associated with fresh biochar have been reported (McClellan et al. 2007). The use of cocoa shell for amendment of contaminated soils and production of biochar is scarce.

Considering the large amount of organic residues generated from cocoa shell in farms across cocoa producing states in Nigeria, and increasing pollution due to proliferating automobile and generator mechanic activities, there is the need to assess the ameliorative potentials of cocoa shell biochar and uncharred organic powder on spent oil-contaminated soils using the growth performance of cowpea. Cowpea is a multipurpose crop grown in semi-arid regions of Africa and Asia for its fresh or dried seeds, fresh pods and leaves used as vegetables and the green or dried leftover parts of the leaves and stems (haulms) used as fodder for livestock (Pottorff *et al.* 2011). The outcome of this study will go a long way to utilizing cocoa shell – raw or pyrolysed as biochar, in rejuvenating contaminated soils thereby increasing spaces for agricultural production.

### MATERIALS AND METHODS

The study was carried out in the Botanical Garden of the University of Ilorin, Ilorin, Nigeria. Cocoa shells (husks) were dried and separated into two portions. A portion was subjected to pyrolysis in a kiln at 450-500 °C until charred organic materials were obtained. The charred organic materials were crushed with mortar and pestle and passed through 0.5 mm pore sized sieve to obtain fine cocoa shell biochar (CSB). The second portion of cocoa shell was crushed using mortar and pestle, milled with a warring blender and sieved through 0.5 mm pores to obtain the uncharred cocoa shell powder (CSP). The biochar and powder were analyzed for pH, total petroleum hydrocarbon (TPH), total organic carbon (TOC), nitrogen, phosphorus, potassium, calcium and magnesium.

Top soil collected from a fallow land within the Botanical Garden was sieved through 2 mm pore and homogenized. 5 kg of soil was packed into each of twenty-four polyethylene bags consisting of 7 treatments and the control each replicated three times. The treatments comprised of 2% w/v spent engine oil (SEO) contamination and different levels of cocoa shell biochar (CSB) or uncharred shell powder (CSP) were set up and tagged as  $T_1$  to  $T_7$ .  $T_1$  was without amendment while CSB was applied to  $T_2$ ,  $T_3$  and  $T_4$  at concentrations of 0.25%, 0.50% and 1.0% respectively. CSP was applied to  $T_5$ ,  $T_6$  and  $T_7$  at concentrations of 0.25%, 0.50% and 1.0% respectively. The control,  $T_0$ , was without SEO or amendment.

Soil samples were collected for chemical determination of pH (in H<sub>2</sub>O and 0.01M CaCl<sub>2</sub> solution), total petroleum hydrocarbon (TPH), total organic carbon (TOC), nitrogen, phosphorus, exchangeable potassium, calcium and magnesium. Soil chemical analyses was also carried out after harvest of the test crop. Soil pH was determined in distilled water and 0.01M CaCl<sub>2</sub> solution using 1:2.5 soil-water ratio. Soil samples were ovendried at 105 °C to constant weight prior to TPH determination according to method by Villalobos *et al.* (2008). TOC was determined using wet digestion method as outlined by Walkley and Black (1934). Nitrogen was determined by macro Kjeldahl method of digestion, distillation and back titration (Bremmer, 1996). Available P was extracted using Bray P1 solution and determined using colorimetric method as outlined by Olsen and

Sommers (1982). Exchangeable potassium, calcium and magnesium were extracted using neutral 1M  $NH_4OAc$  solution. The concentrations of K and Ca were determined using flame photometer (Jenway Model PFP7) while Mg was determined using spectrophotometer (Jenway Model 6305).

The soils in the polyethylene bags were watered for two weeks prior to planting of the test crop (cowpea). Five seeds of cowpea (*Vigna unguiculata* L. (Walp.)) were planted in each polyethylene bag. The number of seeds that emerged from the soil were counted at one week after sowing (1 WAS) was used to determine the percentage germination. Thereafter, the plants were reduced to two stands per bag. Data on growth parameters (stem length and girth, number of leaves, and leaf area) were collected at 3 and 6 WAS. Stem length was obtained using measuring tape while girth was measured using electronic Vernier caliper. Leaves were counted by observation. Leaf area was measured using electronic Leaf Area meter. One plant was uprooted per bag, rinsed and oven dried at 80 °C to constant weight to determine the dry weight per plant at 3 and 6 WAS. Data obtained were subjected to one way proc ANOVA in SAS 9.1.3 Software. Significantly different means were separated using Fisher's LSD at 0.05  $\alpha$  level.

### RESULTS

The chemical composition of the soil amendments (CSB and CSP) is presented in Table 1a. CSB had significantly higher pH, organic carbon, nitrogen, phosphorus, potassium, calcium and magnesium compared with CSP. The chemical properties of the control and spent engine oil – contaminated soils amended with varying levels of CSB and CSP is presented in Table 1b. There were significant differences in the chemical properties assessed. T<sub>3</sub> and T<sub>4</sub> had the highest pH in CaCl<sub>2</sub> (6.01) and water (6.91) respectively. Soil pH increased in the CSB treatments. T<sub>1</sub> had the highest concentration of TPH (1.10%) and decrease significantly with increasing CSB and CSP in the amended soils. T<sub>7</sub> had the highest organic carbon concentration (17.56%) while T<sub>5</sub> had the least (14.91%). T<sub>4</sub> had the highest total nitrogen concentration (0.67%) while T<sub>5</sub> and T<sub>6</sub> had the least (0.41%). T<sub>2</sub> had the highest phosphorus concentration (17.56%) while T<sub>1</sub> had the least (12.09%). T<sub>4</sub> had the highest concentration (17.56%) while T<sub>1</sub> had the least (0.41%). T<sub>2</sub> had the highest phosphorus concentration (17.56%) while T<sub>1</sub> had the least (16.20 cmol<sub>e</sub> Kg<sup>-1</sup>), calcium (2.78 cmol<sub>e</sub> Kg<sup>-1</sup>) and magnesium (1.63 cmol<sub>e</sub> Kg<sup>-1</sup>). T<sub>1</sub> had low concentrations of exchangeable K, Ca and Mg compared with other SEO treatments that were amended (T<sub>2</sub>, T<sub>3</sub>, T<sub>4</sub>, T<sub>5</sub>, T<sub>6</sub> and T<sub>7</sub>).

Treatment	pН	pН	TOC	Ν	Р	K	Ca	Mg
	(H <sub>2</sub> O)	(CaCl <sub>2</sub> )	(%)	(%)	(%)	(mg g <sup>-1</sup> )	(mg g <sup>-1</sup> )	(mg g <sup>-1</sup> )
CSB	10.20 <sup>a</sup>	10.61 <sup>a</sup>	17.40 <sup>a</sup>	3.55 <sup>a</sup>	130.38 <sup>a</sup>	192.33ª	2.48 <sup>a</sup>	5.09 <sup>a</sup>
CSP	4.81 <sup>b</sup>	4.83 <sup>b</sup>	14.70 <sup>b</sup>	1.23 <sup>b</sup>	17.83 <sup>b</sup>	76.92 <sup>b</sup>	1.11 <sup>b</sup>	$0.60^{b}$
LSD <sub>0.05</sub>	0.184	0.175	0.717	0.207	5.820	1.228	0.327	1.612

Table 1a. Chemical composition of cocoa shell biochar (CSB) and uncharred cocoa shell powder (CSP).

Means with the different letters in a column are significant at 0.05  $\alpha$ -level.

**Table 1b.** Chemical properties of spent engine oil-contaminated soils amended with varying levels of cocoa shell biochar (CSB) and uncharred cocoa shell powder (CSP).

Treatment	pН	pН	TPH	TOC	Ν	Р	K	Ca	Mg
	$(H_2O)$	(CaCl <sub>2</sub> )							
			(%)			(%)	(cmole Kg <sup>-1</sup> )		
T <sub>0</sub>	6.03 <sup>d</sup>	5.19 <sup>c</sup>	0.00 <sup>d</sup>	15.69 <sup>c</sup>	0.43 <sup>b</sup>	16.08 <sup>b</sup>	0.03 <sup>e</sup>	1.81 <sup>b</sup>	1.07 <sup>b</sup>
$T_1$	6.20 <sup>c</sup>	5.40 <sup>b</sup>	1.10 <sup>a</sup>	16.06 <sup>bc</sup>	0.28 <sup>c</sup>	12.09 <sup>d</sup>	0.03 <sup>e</sup>	0.53 <sup>e</sup>	0.23 <sup>d</sup>
$T_2$	6.43 <sup>b</sup>	5.52 <sup>b</sup>	0.50 <sup>bc</sup>	16.50 <sup>b</sup>	0.43 <sup>b</sup>	17.56 <sup>a</sup>	5.15 <sup>b</sup>	$2.48^{a}$	0.53 <sup>c</sup>
T3	6.20 <sup>c</sup>	6.01 <sup>a</sup>	0.43 <sup>bc</sup>	16.55 <sup>b</sup>	0.61 <sup>a</sup>	16.25 <sup>b</sup>	6.07 <sup>ab</sup>	2.56 <sup>a</sup>	0.72 <sup>c</sup>
$T_4$	6.91 <sup>a</sup>	5.52 <sup>b</sup>	0.33°	16.65 <sup>b</sup>	0.67 <sup>a</sup>	14.11 <sup>c</sup>	6.20 <sup>a</sup>	$2.78^{a}$	1.63 <sup>a</sup>
T5	6.02 <sup>d</sup>	5.15 <sup>b</sup>	0.57 <sup>b</sup>	14.91 <sup>d</sup>	0.41 <sup>b</sup>	12.17 <sup>d</sup>	3.79°	1.30 <sup>c</sup>	0.68 <sup>c</sup>
$T_6$	6.00 <sup>d</sup>	5.15 <sup>c</sup>	0.50 <sup>bc</sup>	16.62 <sup>b</sup>	0.41 <sup>b</sup>	12.33 <sup>d</sup>	2.59 <sup>d</sup>	1.10 <sup>cd</sup>	0.63 <sup>c</sup>
<b>T</b> <sub>7</sub>	5.60 <sup>e</sup>	5.17 <sup>c</sup>	0.50 <sup>bc</sup>	17.36 <sup>a</sup>	0.45 <sup>b</sup>	12.77 <sup>d</sup>	2.59 <sup>d</sup>	0.81 <sup>de</sup>	0.52 <sup>c</sup>
LSD <sub>0.05</sub>	0.139	0.137	0.210	0.595	0.075	1.202	1.016	0.397	0.264

Means with the same letter(s) down a column are not significant at 0.05  $\alpha$ -level.

There was significant variation (P < 0.05) in the percentage germination of cowpea seeds in the treatments.  $T_0$  and  $T_3$  had the higher germination (100%) but was not different from the other treatments except  $T_1$  (73%) (Figure 1). The growth parameters of cowpea plants in the treatments and control are presented in Figures 2a-e. There were significant differences in the stem length of cowpea in the treatments and control at 3 and 6 weeks after planting (Fig. 2a). The plants in  $T_0$  (Control) had longest stem at 3 WAS (14.0 cm) and 6 WAS (18.7 cm). The stem length of plants in the contaminated soils (unamended and amended) were not significantly different for the growth period considered. Generally, cowpea stem length in the SEO-contaminated soils increased with increasing CSB but decreased with increasing CSP.



Figure 1. Percentage of germinated cowpeas in spent oil contaminated soils amended with varying levels of CSB and CSP.

Cowpea stem girth in the treatments and control varied significantly (P < 0.05) at 3 and 6 WAS (Fig. 2b). The Control (T<sub>0</sub>) had the widest girth at 3 WAS (4.50 mm) and 6 WAS (5.00 mm) but was not significantly different from T<sub>4</sub> during the periods considered. Cowpea plants grown in unamended SEO-contaminated soils (T<sub>1</sub>) had the least girth at 3 WAS (2.63 mm) and 6 WAS (3.17 mm) but was not significantly different from T<sub>2</sub>, T<sub>3</sub>, T<sub>5</sub>, T<sub>6</sub> and T<sub>7</sub>. Stem girth of cowpea also increased with increasing CSB but decreased with increasing CSP.



**Figure 2**. (a) Stem length (b) stem girth (c) number of leaf (d) leaf area and (e) dry weight of cowpeas grown in spent oil – contaminated soils amended with varying levels of CSB and CSP.

Cowpea grown in the unpolluted control soil ( $T_0$ ) had the highest number of leaves at 3 and 6 WAS (11 and 18 leaves respectively). Plants grown in  $T_1$  had the lowest number of leaves at 3 and 6 WAS (5 and 8 leaves) but was not different from  $T_2$ ,  $T_3$ ,  $T_6$  and  $T_7$  at 3 WAS as well as  $T_2$ ,  $T_5$ ,  $T_6$  and  $T_7$  at 6 WAS. Number of leaves also increased with increasing CSB but decreased with increasing CSP (Fig. 2c). The leaf area (LA) varied significantly (P < 0.05) among the treatments. Cowpea plants in the control had the highest LA while plants in  $T_1$  had the least LA. Leaf area increased with increasing CSB and decreased with higher concentration CSP in the treatments at 3 and 6 WAS (Fig. 2d).

The control  $(T_0)$  had the significantly highest dry weight per plant at 3 WAS (0.54 g) and 6 WAS (4.70 g). There was increase in dry weight with increasing CSB and decreasing CSP concentrations, however, no significant difference was observed in the treatments (Fig. 2e).

## DISCUSSION

The higher pH of cocoa shell biochar (CSB) in this study confirms earlier report by McClellan *et al.* (2007) that biochar have high (alkaline) pH which is beneficial in reducing acidity in native soils. Most biochars are alkaline owing to their ash content, causing release of base cations, and alkaline properties of organic functional groups (Yuan and Xu 2012). The higher organic carbon (TOC), total nitrogen, exchangeable potassium, calcium and magnesium in CSB than CSP is consistent with the observation of Obia *et al.* (2015).

The significantly lower values of pH, TOC, total N, available P and exchangeable Ca and Mg in contaminated soil suggests spent engine oil contamination negatively influenced these parameters. This result of low nutrient in the SEO-contaminated soil ( $T_1$ ) is connected to anaerobic condition resulting from reduced permeability, surface sealing, increased compaction and decrease pore spaces (Atlas 1977, Nwite 2013). This condition has been reported to increase the population of soil anaerobic organisms (Baldwin 1922, Gbadebo and Adenuga 2012). Adu *et al.* (2015) also reported that contamination with spent oil significantly influence soil pH, OC and nitrogen. Nwite (2013) likewise observed decreases in CEC and ECEC in oil-contaminated soils but the parameters were significantly improved upon addition of biochar and followed by uncharred organic waste. The significant reduction in TPH and improvement in nutrient status of SEO-contaminated soils amended with CSB and CSP showed these organic materials had a way of modifying the soil properties. Decline TPH concentration in the amended soils confirm the report of Nwite (2013), however, CSB had higher sorption than CSP.

Increased pH in the biochar-amended soils is associated to the alkaline nature of the CSB while low pH of CSP (uncharred cocoa shell powder) also contributed to low pH in CSP-amended soils. The observed increase in pH in the biochar-amended soil is consistent with increases in pH reported elsewhere (Shackley *et al.* 2012, Carter *et al.* 2013, Obia *et al.* 2015). Beesley *et al.* (2011) confirmed the potential of biochar to increase soil pH, OC and exchangeable cations. Uchimiya *et al.* (2011) reported that cation exchange capacity (CEC) was the primary mechanism by which biochar enhance nutrient retention in soils. According to them, adding biochar increased CEC and the rate at which the soil solution attain equilibrium. This result confirms earlier studies that have shown that the characteristics of biochar to plant growth can improve over time after its incorporation into soil (Cheng *et al.* 2006, 2008, Hunt *et al.* 2010, Major *et al.* 2010).

The significant reduction in the percentage of germinated cowpea seedlings in SEO–contaminated soil was prompted by anaerobic condition caused by oil pollution (Udo and Fayemi 1975). Vwioko *et al.* (2005) posited that reduction in germination in plants exposed to contamination with petroleum products could result from oil coating on seed surface that affect physiological functions in the seed. According to Henner *et al.* (1999) some volatile 3-ringed polycyclic aromatic hydrocarbons (PAHs) fractions in spent engine oil have severe inhibitory impact on germination of several plant species. Likewise, PAHs have been documented to have indirect secondary effects on germination including disruption on plant–water–air relationships (Renault *et al.* 2000) and affect population of microorganisms (Nicolotti and Egli 1998). The comparable result in seed germination in the amended soils with the uncontaminated (control) affirm the remediation potentials of CSB and CSP. The addition of soil amendments (CSB and CSP) ensures the availability of water, proper porosity and nutrients to the plant under stress conditions (Shao *et al.* 2005) such as those imposed by the SEO contamination.

The decline in growth of cowpea plants in the SEO-contaminated soil is attributed to low pH and nutrient earlier reported. This observation confirms earlier report on decline in cowpea growth resulting from spent oil contamination (Kayode *et al.* 2009, Lale *et al.* 2014). Oil polluted soils is rendered unsuitable for growth of plants for a long time until the oil degrade to a tolerable level (Udo and Opara 1984). The significant increases in the leaf area and number of leaf of plants in CSB treatments, especially the 1% addition, relative to the unamended SEO-contaminated soil affirm the oil-remediating potential of biochar. Sorption of contaminants such as PAHs have been found to increase by factors of up to 700 after the addition of biochars (Zhang *et al.* 2010). This increased sorption translates into reductions in the bioavailable PAH fraction (Beesley *et al.* 2010) and reductions in PAH accumulation in soil organisms upon biochar amendment (GomezEyles *et al.* 2011).

Reduced biomass production by plants grown in SEO-contaminated soils is related to decline in the leaf area and number of leaf influenced by modification of soil properties by oil. Okon and Mbong (2013) attributed such reduction in crop yield in SEO-contaminated soil to reduction in the leaf area exposed to photosynthesis

affected by reduction in soil nutrients including nitrogen (Agbogidi *et al.* 2007) and phosphorus (Dimitrow and Markow 2000). Leaf area growth determines light interception and is an important parameter in determining plant productivity (Koester *et al.* 2014).

Convincingly, SEO contamination altered nutrient composition of the soil and retarded growth of cowpea. However, application of cocoa shell biochar (up to 1%) improved soil condition and growth of the crop whereas increased addition of uncharred cocoa shell powder decreased soil pH (increased acidity) and reduced cowpea growth. It is recommended that cocoa shell waste be converted to biochar prior to use as soil amendment will help to condition the soil against contaminants while eliminating the toxicity imposed by raw cocoa shells. There is the need to educate automobile and generator mechanics to avert the menace posed by indiscriminate disposal of spent engine oils.

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