

# FORMULATION AND OPTIMISATION OF SPENT BLEACHING EARTH-BASED BIO ORGANIC FERTILISER

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## ABSTRACT

Spent bleaching earth (SBE) generated from the palm oil refinery is convertible into value-added products instead of being discarded as waste to landfills. An alternative approach is to develop SBE-based bio organic fertiliser through co-utilisation with other sources of biomass of contrasting nutrients properties. The feasibility of blending SBE with various forms of biomass – oil palm trunk (OPT), oil palm frond (OPF), empty fruit bunch (EFB) and chicken litter (CL) – in different mixing ratios was studied and its optimum ratio determined. The mixtures of SBE and various biomass at different mixing ratios were analysed for their macronutrient and micronutrient content, pH, organic carbon (OC), total nitrogen, carbon:nitrogen ratio (C:N) and organic matter (OM) content. The optimised blend of SBE:OPT:CL at the ratio of 1:1:0.5 exhibited sufficient nutrient contents (N: P<sub>2</sub>O<sub>5</sub>: K<sub>2</sub>O = 0.65:1.59:1.63) and good physico-chemical properties (pH = 5.4, OM = 40% and C:N = 36:1) as a base material for bio organic fertiliser production. This optimum formulation was further enriched with urea (46% N), Christmas Island rock phosphate (CIRP, 25% P<sub>2</sub>O<sub>5</sub>) and muriate of potash (MOP, 66% K<sub>2</sub>O) to produce a bio organic fertiliser suitable for vegetable crops with desired nutrients N:P<sub>2</sub>O<sub>5</sub>:K<sub>2</sub>O ratio of 2:2:2.

**Keywords:** spent bleaching earth, oil palm biomass, plant nutrient, soil amendment, bio organic fertiliser.

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## INTRODUCTION

Spent bleaching earth (SBE) is an alumino-silicate mineral that mainly consists of montmorillonites with 2:1 unit layer structure, generated from the bleaching process in the palm oil refinery. In Malaysia, 5 – 10 kg of bleaching earth per tonne of crude palm oil (CPO) is used resulting in the generation of up to

170 000 t of SBE per annum (Boey *et al.*, 2011). This is indicative of a huge quantity of waste generated from the palm oil industry. The conventional method of disposing SBE at landfills is a waste of resources and causes environmental pollution (Loh *et al.*, 2013). Hence, the search for alternative approaches such as recycling or recovery of the nutrients from SBE as bio organic fertiliser (Loh *et al.*, 2013), soil amendment (Arias-Estévez *et al.*, 2007) or fertiliser supplement (Wang *et al.*, 2010) to improve yields of agricultural crops is highly desirable. SBE was found to increase the extractable phosphorus (P) of a degraded light-textured soil from its initial 7 mg kg<sup>-1</sup> P to 13-20 mg kg<sup>-1</sup> at an application rate of more than 20 t ha<sup>-1</sup> (Crocker *et al.*, 2004). In addition, SBE was able to rejuvenate degraded soil by enhancing

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the cation exchange capacity (CEC) of soil and increasing the water absorption capacity (Soda *et al.*, 2006).

However, SBE alone causes an imbalance in plant nutrient supply especially nitrogen (N) and potassium (K). Besides, it is acidic in nature. This has hindered its utilisation from becoming a stand-alone bio organic fertiliser for land application and agriculture (Soda *et al.*, 2006). Co-utilisation of SBE with oil palm biomass (Loh *et al.*, 2013), sawdust, wheat bran, rice husk, rice husk ash and chicken litter (Wang *et al.*, 2010; Soda *et al.*, 2006) has shown evidences in improving its nutrient and soil conditioning effect with improved quality in terms of available nutrients and reductions in acidity and hydrophobicity of SBE.

On the other hand, 80 million tonnes of oil palm biomass are generated from the oil palm industry yearly (Loh and Choo, 2013). Oil palm trunks (OPT) available during replanting, oil palm fronds (OPF) and empty fruit bunches (EFB) via normal estate and milling practices are normally returned back to oil palm plantations and left to rot in between the rows of oil palm to increase soil fertility. Hence, nutrients are released continually to support the growth of oil palms. The bioavailable N, P, K content of OPT is 1.691 kg, 0.163 kg and 4.892 kg per palm, respectively (Khalid *et al.*, 1999), whereas OPF contains 0.297 kg N, 0.035 kg P and 1.524 kg K per palm, respectively. It was reported that for a tonne of fresh fruit bunch (FFB) processed in a palm oil mill, 230 kg of EFB were generated along with the N, P, K nutrients of 8, 1 and 24 kg t<sup>-1</sup>, respectively (Stichnothe and Schuchardt, 2010). Nevertheless, direct dumping of biomass in plantation has some disadvantages like temporary immobilisation of nutrients, harbouring of rats and snakes, risk of fire, breeding site for rhinoceros beetles, high weight and volume in relation to nutrient temporary storage even though the various forms of oil palm biomass contain macronutrients for crop growth. In addition, nutrient leaching from oil palm biomass which causes a waste of resources cannot be avoided too.

Other locally available renewable farm waste such as chicken litter (CL) is produced abundantly and used as land application and soil supplement. According to Sellami *et al.* (2008), a chicken weighing 2.3 kg releases 0.15-0.20 kg of manure per day consisting of 1% - 6% of total N (dry matter). In Malaysia, an annual average of 26 million population of chicken (two-third of a total 208 million poultry population having growing cycle of 60 days) (Department of Veterinary Services, 2009) could produce approximately 1.4 million tonnes of chicken manure per annum. However, a continuous and heavy application of such manure to land may pose some environmental problems such as odour and pollution of ground and surface waters due to leaching of N-rich nutrients and accumulation

of heavy metals in soil (Lu *et al.*, 2010). From an agronomic point of view, the use of various abundant biomass resources in a more sustainable manner for nutrients recycling could provide alternatives and new opportunities in agriculture.

Therefore, the main aim of this work was to develop and formulate an effective bio organic fertiliser from SBE, oil palm biomass (OPT, OPF, EFB) and CL at a desired optimal N:P<sub>2</sub>O<sub>5</sub>:K<sub>2</sub>O ratio of 2:2:2 suitable for vegetable crops by incorporation and enhancement with mineral fertilisers (urea, Christmas Island rock phosphate - CIRP and muriate of potash - MOP). The effects of different raw biomass and mixing ratio on the physico-chemical properties of the fertilisers formulated were investigated.

## MATERIALS AND METHODS

### Material

The raw materials used in the bio organic fertiliser formulations consisted of SBE, OPT, OPF, EFB and CL. SBE was obtained from MPV Technologies Sdn Bhd in Pasir Gudang, Johor, Malaysia. Samples of EFB, OPT and OPF were provided by the oil palm industry whereas CL was commercially available. A total of nine blending treatments (*Table 1*) using different mixing ratios of SBE, OPT, OPF, EFB and CL were tested. The various raw materials used were dried and homogenised using an automated continuous-mode stainless steel grinder (Dickson DFY-300, speed 24000 rpm min<sup>-1</sup>, grinding capacity 300 g, voltage 240V/5Hz and power 1000 W). The optimised blend was then fortified with commercially available mineral fertilisers (urea, CIRP and MOP) at a ratio based on theoretical calculation to achieve the desired N: P<sub>2</sub>O<sub>5</sub>: K<sub>2</sub>O content.

### Chemical Analysis

The raw materials, their mixtures at different ratios and the fortified fertilisers produced were analysed for their physico-chemical and fertiliser properties. Moisture content was determined by a gravimetric method in an oven at 105°C; pH at a sample/deionised water ratio of 1:5 using PH 211 microprocessor pH meter (Soda *et al.*, 2006); total organic carbon (OC) contents by wet oxidation using the Walkley-Black dichromate digestion method. The factor used to convert OC to organic matter (OM) content was 1.724 (Chen *et al.*, 2004). The total N and S of samples were examined using an elemental analyser (CNS-LECO 2000). The C:N ratio was calculated from the measured values of total OC and N. The CEC was determined using 1M NH<sub>4</sub>-acetate buffer at pH 7.0 (Soda *et al.*, 2006). The total P (as P<sub>2</sub>O<sub>5</sub>) of samples was determined using

**TABLE 1. COMBINATION OF DIFFERENT BLENDING TREATMENTS FOR THE FORMULATION OF BIO ORGANIC FERTILISER**

Treatment code	Treatment description	Ratio (% w/w)
1	SBE + OPT + CL	1:1:0.1
2	SBE + OPT + OPF	1:1:0.1
3	SBE + OPT + EFB	1:1:0.1
4	SBE + OPT + CL	1:1:0.3
5	SBE + OPT + OPF	1:1:0.3
6	SBE + OPT + EFB	1:1:0.3
7	SBE + OPT + CL	1:1:0.5
8	SBE + OPT + OPF	1:1:0.5
9	SBE + OPT + EFB	1:1:0.5

Note: SBE - spent bleaching earth. OPT- oil palm trunk. OPF- oil palm frond. EFB - empty fruit bunch. CL- chicken litter.

the molybdenum blue method with molybdenum in  $H_2SO_4$  and colour formation was measured by spectrophotometer UV-120-01 at  $\lambda=880$  nm. The acid extractable cations (K, Ca, Mg) and total micronutrients (Cu, Zn, Mn, Fe) were extracted using aqua-regia solution and then determined using a flame atomic absorption spectrophotometer (F-AAS) Perkin Elmer Analyst 400.

## Statistical Analyses

Single factor analysis of variance (ANOVA) was used to estimate the significance of treatment. Besides, Tukey HSD test was used for comparison of treatment means when F values were significant at  $p<0.05$ .

## RESULTS AND DISCUSSION

### Characteristics of Raw Materials

All the biomass employed showed varying chemical compositions (Table 2), thus there is a need to formulate a biomass blend having balanced nutrient contents for them to be used as effective as possible as soil supplement. Among the biomass analysed, SBE had the lowest total N content ( $0.06 \pm 0.00\%$ ) and the highest C:N ratio (293:1) whereas the rest exhibited higher contents of total N than SBE, *i.e.*  $1.08 \pm 0.09\%$  (CL),  $0.19 \pm 0.01\%$  (OPT),  $0.55 \pm 0.01\%$  (OPF) and  $0.33 \pm 0.02\%$  (EFB). It has been shown that C:N ratio can be used to gauge how healthy the environment is for adequate

**TABLE 2. CHEMICAL CHARACTERISTICS OF RAW MATERIALS (mean  $\pm$  SD n=3)**

	SBE	OPT	CL	OPF	EFB
pH	5.33 $\pm$ 0.02	3.69 $\pm$ 0.02	8.75 $\pm$ 0.02	4.35 $\pm$ 0.11	4.65 $\pm$ 0.05
Organic carbon (%)	17.4 $\pm$ 0.4	29.6 $\pm$ 0.4	7.75 $\pm$ 1.5	26.4 $\pm$ 4.0	31.1 $\pm$ 1.1
Total N (%)	0.06 $\pm$ 0.00	0.19 $\pm$ 0.01	1.08 $\pm$ 0.09	0.55 $\pm$ 0.01	0.33 $\pm$ 0.02
C:N	293 $\pm$ 20	158 $\pm$ 8	7 $\pm$ 1	48 $\pm$ 7	94 $\pm$ 4
Total P (as $P_2O_5$ ) (%)	2.36 $\pm$ 0.34	0.02 $\pm$ 0.04	2.22 $\pm$ 0.27	0.03 $\pm$ 0.00	0.03 $\pm$ 0.01
CEC (cmol kg <sup>-1</sup> )	36.0 $\pm$ 0.2	9.2 $\pm$ 0.2	30.5 $\pm$ 0.5	13.5 $\pm$ 0.04	9.04 $\pm$ 0.02
Organic matter (%)	30.0 $\pm$ 0.8	51.0 $\pm$ 0.6	13.4 $\pm$ 2.7	45.5 $\pm$ 6.9	53.7 $\pm$ 1.9
Ext. K (as $K_2O$ ) (%)	0.27 $\pm$ 0.02	1.21 $\pm$ 0.06	2.25 $\pm$ 0.03	2.00 $\pm$ 0.06	1.59 $\pm$ 0.09
Ext. Ca (as CaO) (%)	3.58 $\pm$ 0.36	0.23 $\pm$ 0.03	14.6 $\pm$ 0.80	0.60 $\pm$ 0.06	0.14 $\pm$ 0.02
Ext. Mg (as MgO) (%)	1.55 $\pm$ 0.06	0.25 $\pm$ 0.01	3.26 $\pm$ 0.14	0.16 $\pm$ 0.03	0.16 $\pm$ 0.01
Total Cu (ppm)	41.4 $\pm$ 0.6	2.01 $\pm$ 0.6	60.7 $\pm$ 3.3	2.00 $\pm$ 0.5	7.15 $\pm$ 0.05
Total Zn (ppm)	30.1 $\pm$ 1.7	8.04 $\pm$ 0.6	313 $\pm$ 17	8.59 $\pm$ 2.9	18.0 $\pm$ 0.4
Total Mn (ppm)	359 $\pm$ 4	22 $\pm$ 4	361 $\pm$ 21	43 $\pm$ 4	21 $\pm$ 1
Total Fe (ppm)	10 026 $\pm$ 663	216 $\pm$ 167	5 205 $\pm$ 230	679 $\pm$ 234	307 $\pm$ 55

Note: Ext. - extractable. SBE - spent bleaching earth, OPT - oil palm trunk, CL - chicken litter, OPF - oil palm frond, EFB - empty fruit bunch, CEC - cation exchange capacity, SD - standard deviation.

microbial soil function (growth and survival) (Loh *et al.*, 2013). This implies that the C:N ratio of SBE was imbalanced in terms of its use as an organic fertiliser. Typically, a high C:N ratio is indicative of a lack of N in SBE, hence, an additional amount of N is required to achieve an optimal C:N ratio of 20:1 to 40:1 as recommended for organic fertiliser (Rao *et al.*, 2007). Thus blending SBE with N-rich organic materials might be adequate to improve the nutritional balance in its formulation. As CL had the highest total N content, its incorporation in the formulation could contribute to the reduction of the C:N ratio besides providing higher concentrations of essential nutrients which was comparatively lower in SBE such as K, Ca and Mg. The magnitude change in N content would be about 10 times greater in SBE than C content as reflected by the distinguishably greater differences between SBE and CL (Table 2). As per the formula C/N implies, a greater increase in N in the mixture compared to C will result in a lower C without having to establish their correlation, hence lower C:N ratio in the mixture. The addition of CL to the mixture will add more benefit to the already high total P content in SBE ( $2.36 \pm 0.34\%$ ). All other raw materials used exhibit low nutritional contents, however their addition to the mixture will improve the physical composition needed in physical conditioning of the treated soil.

Blending oil palm biomass (OPT, OPF and EFB) with SBE can add benefit to the mixtures as they are rich in K ( $1.21 \pm 0.06\%$ ,  $2.00 \pm 0.06\%$ ,  $1.59 \pm 0.09\%$ , respectively). SBE has higher amount of Ca and Mg compared to oil palm biomass, but is still lower compared to CL, thus the addition of CL will improve the nutritional composition in the blend. The higher content of Ca and Mg in SBE is due to higher amount of negatively charged exchange sites based in the structural layer of SBE which is saturated with ionic forms of Ca and Mg necessary for plant growth.

The pH of SBE at  $5.33 \pm 0.02$  was indicative of an acidic material due to the replacement of other metallic ions with  $H^+$  (Al-Zahrani *et al.*, 2000; Weng *et al.*, 2007). OPT, OPF and EFB had lower pH at  $3.69 \pm 0.02$ ,  $4.35 \pm 0.11$  and  $4.65 \pm 0.05$ , respectively, whereas CL had an alkaline pH of  $8.75 \pm 0.02$ . In theory, a reduction of soil pH (for alkali clay soil) can be anticipated with the addition of varying amounts of acid-activated SBE. On the other hand, soil that is too acidic can be amended using SBE from white wine refinery having initial higher pH (9.1), as shown by N6voa-Mu6noz *et al.* (2008) on Arenic regosol soil reaching a soil pH higher than pH 7. The mixture of SBE, OPT, OPF and EFB in this study can only be used to condition alkaline soil. However, associating them with varying amounts of CL will balance the pH desired for the organic fertiliser formulated.

SBE can be potentially used to improve charge properties of degraded soils due to its higher CEC

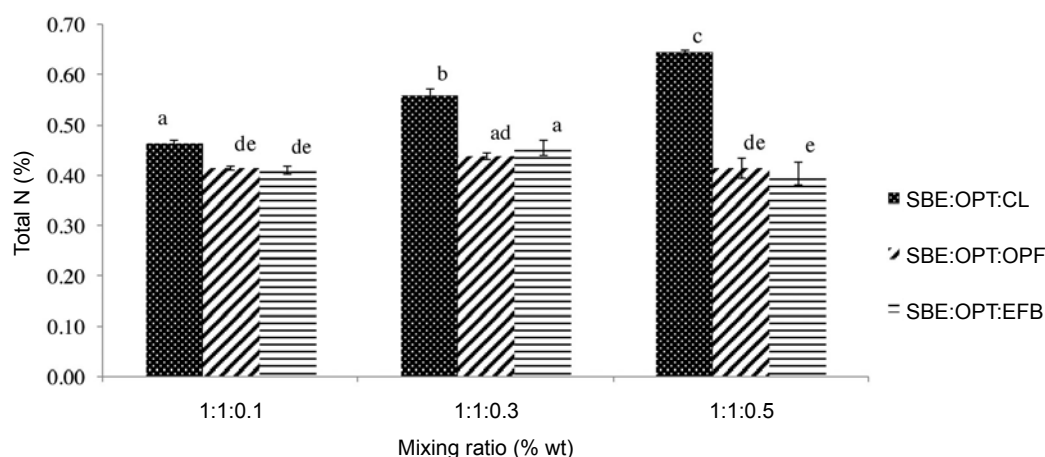
( $36.02 \pm 0.15 \text{ cmol kg}^{-1}$ ) than the oil palm biomass (OPT, OPF, EFB). Similar observation by Arias-Est6vez *et al.* (2007) found that the CEC of soil rose from  $1.6 \text{ cmol(+) kg}^{-1}$  to  $3.6 \text{ cmol(+) kg}^{-1}$  at  $30 \text{ g kg}^{-1}$  SBE applied. The blending of the lignocellulosic oil palm biomass in SBE as soil conditioner, besides supplying OC, can also improve aeration in soil due to active microbial rejuvenation. The concentrations of micronutrients in SBE (Cu, Zn, Mn and Fe) were much higher than those in the OPT, OPF and EFB (Table 2) and surprisingly have exceeded the typical concentrations sufficient for plant growth (Epstein, 1965).

### Total Nitrogen

An increased concentration of CL in the SBE:OPT:CL blend showed significance increase in the total N content ( $p < 0.05$ ) (Figure 1). The blend with the highest ratio of CL (1:1:0.5) had the highest N content ( $0.65 \pm 0.00\%$ ), followed by those mixed at ratio of 1:1:0.3 ( $0.56 \pm 0.01\%$ ) and 1:1:0.1 ( $0.46 \pm 0.01\%$ ). The total N content of the formulated blends correlated well with the proportion of the CL added ( $R^2 = 0.999$ ). The total N concentration in SBE:OPT:OPF and SBE:OPT:EFB blended in similar manner did not change significantly ( $p > 0.05$ ) regardless of the mixing ratio used. However, the incorporation of CL in the SBE:OPT:CL blend (1:1:0.5) increased the total N content to about 10-fold from the initial 0.06% to 0.65%. The reason is two-fold: (1) the initial higher content of total N in CL, and (2) unlike OPF and EFB, CL is known for its capability to furnish N in the form of uric acid (70%) and undigested proteins (30%) (Materechera and Mkhabela, 2002; Abouelenien *et al.*, 2010). SBE which has high CEC due to isomorphous substitution of  $Al^{3+}$  or  $Si^{4+}$  by lower charge cations like  $Fe^{2+}$ ,  $Fe^{3+}$  or  $Mg^{2+}$ , is able to exchange and retain ammonium cations,  $NH_4^+$  resulting from the anaerobic decomposition of uric acid in CL (Borah *et al.*, 2008; Redding, 2011), thus reducing the possibility for plant nutrient to leach unnecessarily. This result has therefore provided an indication of a formulated blend having relatively high exchangeable ability in holding and slow-releasing of  $NH_4^+$  to prevent excessive leaching of nutrient N and was supported by a previous study (Cheong *et al.*, 2014).

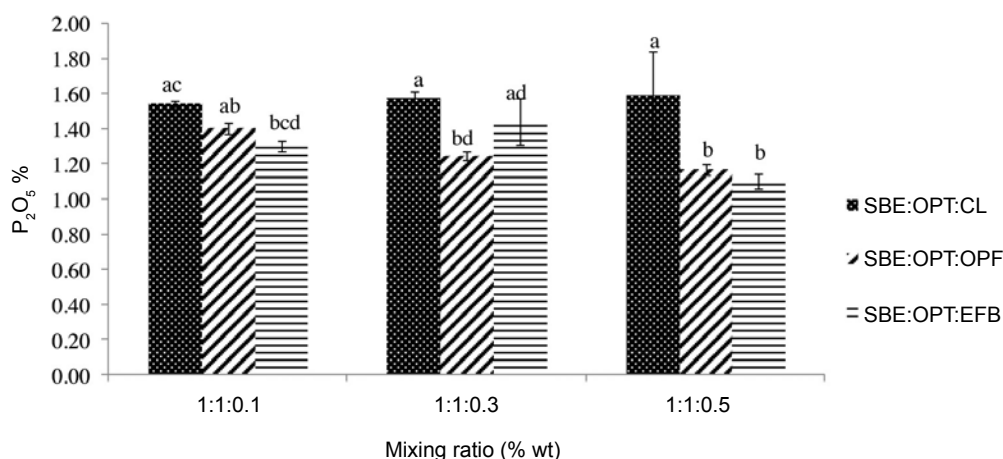
### Total Phosphorus (as $P_2O_5$ )

The biomass blends – SBE:OPT:CL, SBE:OPT:OPF and SBE:OPT:EFB – had comparatively higher contents of total P than the raw biomass forms (OPF, OPT, EFB) at various mixing ratios (Table 2 and Figure 2) due to the high initial P content ( $2.36 \pm 0.34\%$ ) in SBE. The high P in SBE is probably attainable from the bleaching process of CPO in the form of inorganic phosphate ( $HPO_4^{2-}$  or  $H_2PO_4^-$ ) (Gibon *et al.*, 2007). In



Note: SBE - spent bleaching earth, CL - chicken litter, OPT - oil palm trunk, OPF - oil palm frond, EFB - empty fruit bunch. Vertical error bars indicate 95% confidence interval with different letters mean significant differences between treatments ( $p < 0.05$ ).

Figure 1. Changes in total nitrogen (N) of the formulated blends at different mixing ratio.



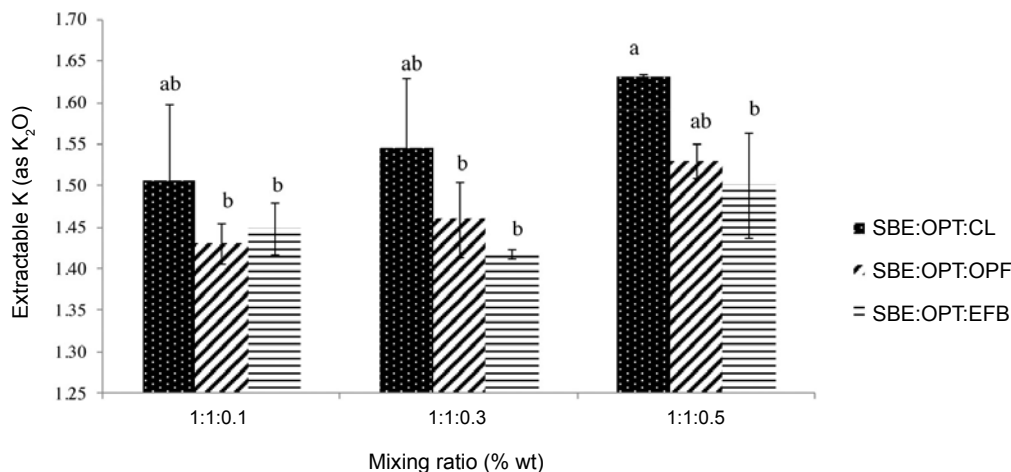
Note: SBE - spent bleaching earth, CL - chicken litter, OPT - oil palm trunk, OPF - oil palm frond, EFB - empty fruit bunch. Vertical error bars indicate 95% confidence interval with different letters mean significant differences between treatments ( $p < 0.05$ ).

Figure 2. Changes in total phosphorus ( $P_2O_5$ ) content of the formulated blends at different mixing ratio.

Figure 2, the formulated blends containing CL at three different mixing ratios showed significantly higher total P contents than those blends containing OPF and EFB. Among the nine formulated blends, SBE:OPT:CL constituted the expected highest total P content, *i.e.*  $1.55 \pm 0.01\%$ ,  $1.58 \pm 0.03\%$  and  $1.59 \pm 0.22\%$  at mixing ratio 1:1:0.1, 1:1:0.3 and 1:1:0.5, respectively, compared to the rest of the formulations regardless of different mixing ratios used due to the initial higher contents of total P in CL ( $2.22 \pm 0.27\%$ ) and SBE ( $2.36 \pm 0.34\%$ ). However, these blends did not show significance differences within treatments at varying amounts of CL. On the other hand, the blends consisting SBE:OPT:EFB did not decrease linearly like SBE:OPT:OPF arguably due to difficulties in ensuring SBE homogenisation during blending.

### Extractable Potassium (as $K_2O$ )

Of all the formulated blends, SBE:OPT:CL at the blending ratio of 1:1:0.5 was found to yield the highest proportion of extractable K, *i.e.*  $1.63 \pm 0.01\%$  (Figure 3) while SBE:OPT:EFB at 1:1:0.3 blending ratio contained the least ( $1.42 \pm 0.01\%$ ). The blends treated with CL showed significantly different K content ( $p < 0.05$ ) amongst the treatments except for SBE:OPT:OPF at 1:1:0.5 blending ratio, and *vice versa* for others containing OPF and EFB at varying mixing ratios. The increase in extractable K was directly proportional to the increasing amount of added CL ( $R^2=0.956$ ) having high K ( $2.25 \pm 0.03\%$ ), with the highest increase from  $0.27 \pm 0.02\%$  in raw SBE to  $1.63 \pm 0.01\%$  in SBE:OPT:CL (1:1:0.5). The ability of SBE to exchange and retain the K of CL



Note: SBE - spent bleaching earth, CL - chicken litter, OPT - oil palm trunk, OPF - oil palm frond, EFB - empty fruit bunch. Vertical error bars indicate 95% confidence interval with different letters mean significant differences between treatments ( $p < 0.05$ ).

Figure 3. Changes in extractable potassium (K) of the formulated blends at different mixing ratio.

in the formulation against leaching and be readily available for crop uptake is not evident in this study unless appropriate leaching test is carried out.

### pH

The pH varied at different mixing ratios and with raw materials used. However, the changes in all the formulated blends followed a similar trend. The pH of the formulated blends SBE:OPT:OPF and SBE:OPT:EFB at ratios of 1:1:0.1, 1:1:0.3 and 1:1:0.5 were significantly lower than those blended with CL (Figure 4). This could be due to the released OH<sup>-</sup> by ammonification of organic N whose concentration was the highest in CL compared to other oil palm biomass into ammonia and ammonium during its decomposition (Materechera and Mkhabela, 2002), causing pH in blended SBE:OPT:CL to increase more than other blends SBE:OPT:OPF and SBE:OPT:EFB (Materechera and Mkhabela, 2002). The dissociation of metal oxides of Cu, Zn, Mn and Fe in the highly moist CL may contribute to higher pH too. The pH values of all these blends were still within the optimal range for use as soil amendment. At these pH ranges, the affinity of soil to bind plant nutrients such as N, P, K, Cu, Zn, Mn and Fe forming metal chelates is enhanced resulting in better bioavailability to plants. This finding was in agreement with Piller *et al.* (2003) who stated that soils at pH > 6.0 were often associated with poor growth, reduced yields and chlorosis due to reduced uptake of Fe; even in the presence of adequate nutrients.

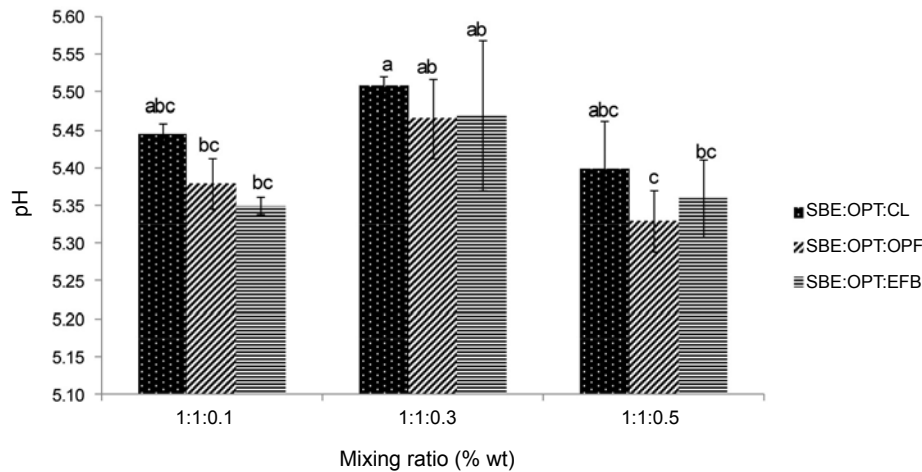
### Carbon to Nitrogen Ratio

The mixing of the appropriate raw materials in bio organic fertiliser could achieve an optimal C:N

ratio in the range of 20:1 to 40:1 (Rao *et al.*, 2007). Organic materials with high C:N ratios (>30:1) exhibit low decomposition rates due to immobilisation of applied N causing low bioavailability of N for soil microorganisms, and thus have a less pronounced effect on the growth and activity of microorganisms and soil aggregation (Ahmad *et al.*, 2006; N'Dayegamiye, 2009). In this study, the initial C:N ratios of the raw materials were > 30 except for CL (Table 2). Due to high OC content of the raw materials, the C:N ratios of the mixed treatment of SBE with OPF and EFB at varying mixing ratios remained high (61 - 66) and insignificant, but *vice versa* for SBE treated with CL (Figure 5). Additions of CL with a low C:N ratio in SBE can greatly increase N due to the mineralisation of organic N. The C:N ratio in SBE:OPT:CL (1:1:0.5) decreased significantly from 293:1 in the raw SBE to 35.7:1 in the respective blend showing a significant reduction of C:N ratio (87.8%) to an almost optimal ratio that can stimulate the rapid N mineralisation and the production of NH<sub>4</sub><sup>+</sup> needed for plant growth.

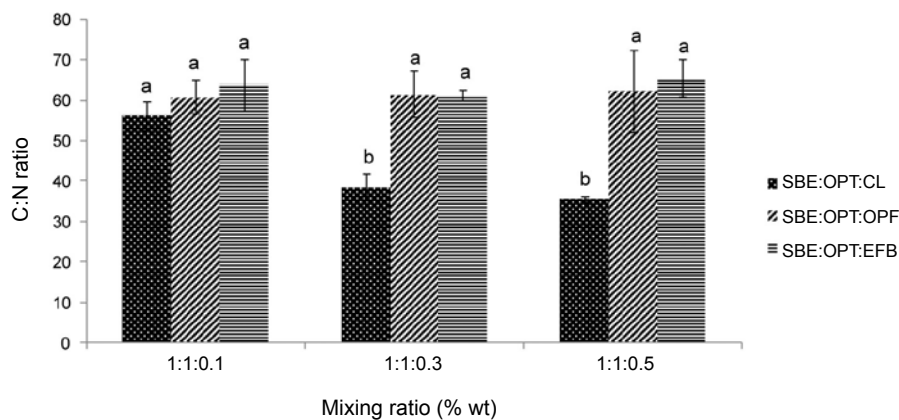
### Micronutrients Concentration

In Table 3, the changes in micronutrients concentration of all the formulated blends containing SBE were relative to the amount of raw materials used, *e.g.* the relatively higher concentrations of Cu, Zn, Mn and Fe in CL than other biomass such as OPT, OPF and EFB had resulted in higher micronutrients concentration in SBE:OPT:CL blends although their concentrations were lower compared to the raw SBE (Table 2). This trend was in agreement with the other two parameters investigated (total P and C:N ratio) in this study, largely due to the initial higher values in SBE, respectively. Thus, all the blends consisting



Note: SBE - spent bleaching earth, CL - chicken litter, OPT - oil palm trunk, OPF - oil palm frond, EFB - empty fruit bunch.

Figure 4. Changes of pH in the formulated blends at different mixing ratio.



Note: SBE - spent bleaching earth, CL - chicken litter, OPT - oil palm trunk, OPF - oil palm frond, EFB - empty fruit bunch.

Figure 5. The C:N ratio of the formulated blends at different mixing ratio.

of SBE:OPT:CL had much higher micronutrient concentrations than other blends using OPF and EFB, respectively (Table 3). These blends can possibly perform better in exchanging and maintaining the micronutrients in soil due to better adsorption/desorption of these metals by the more exposable aluminol and silanol groups present in the broken edges and outer layers of montmorillonite-based clay minerals like SBE (Abollino *et al.*, 2003). The Cu and Zn concentrations in the formulated SBE: OPT: CL blend at varying mixing ratios were within the recommended maximum allowable concentrations (MAC) or trigger action value (TAV) for the following applications: (1) land application of compost (Commission of European Communities, 1986), (2) trace metals in agricultural soils (Kabata-Pendias, 2011), and (3) fertilisers used for agricultural soils in Finland (Kabata-Pendias, 2011) (Table 3). Their

presence in soil is well within the tolerable intake level in agronomic crops, hence the formulated SBE:OPT:CL blends are safe to be used as soil amendment besides providing micronutrients for plant growth.

#### Optimised N:P:K Fertiliser from Spent Bleaching Earth and Oil Palm Biomass

The formulated SBE:OPT:CL blend (1:1:0.5) having the maximum N:P:K profile was further enhanced by mineral fertilisers (urea, CIRP, MOP) in order to achieve a targeted N:P<sub>2</sub>O<sub>5</sub>:K<sub>2</sub>O nutrient ratio of 2:2:2 for use in vegetable crops (Table 4). The resulting blend had acceptable fertiliser characteristics, *i.e.* pH 5.40 ± 0.06, C:N ratio 36:1 and allowable limits of heavy metal concentrations. Among the five amendments formulated, fertiliser

mix 1 exhibited the most promising enriched nutrient, *i.e.*  $1.57 \pm 0.06\%$  total N,  $1.93 \pm 0.03\%$  total P and  $1.94 \pm 0.09\%$  extractable K, which was very close to the desired target of N:P<sub>2</sub>O<sub>5</sub>:K<sub>2</sub>O of 2:2:2. A preliminary field trial carried out on egg-plant to investigate the effects of this fortified organic fertiliser on vegetative growth showed that it could promote plant growth, improve crop quality while increase crop productivity and yield (Cheong *et al.*, 2014). The increase in nutrients uptakes by

egg-plant during the trial was evidence of a fairly good cationic exchange property to resist nutrients leaching while still being available for uptake by plant roots (Crocker *et al.*, 2004).

### CONCLUSION

This study revealed that SBE blended with OPT and CL at the ratio of 1:1:0.5 had the most favourable

TABLE 3. TOTAL MICRONUTRIENTS CONCENTRATION OF THE FORMULATED BLENDS

Mixed treatment	Blend ratio	Cu (ppm)	Zn (ppm)	Mn (ppm)	Fe (ppm)
SBE+OPT+CL	1:1:0.1	29.3±0.8 <sup>a</sup>	116±7 <sup>a</sup>	215±2 <sup>ac</sup>	8 040±355 <sup>a</sup>
	1:1:0.3	32.5±0.8 <sup>b</sup>	127±25 <sup>a</sup>	220±2 <sup>a</sup>	7 401 ±1 330 <sup>ab</sup>
	1:1:0.5	34.4±1.0 <sup>c</sup>	88.3±3.2 <sup>b</sup>	258±4 <sup>b</sup>	7 469±479 <sup>a</sup>
SBE+OPT+OPF	1:1:0.1	26.1±0.6 <sup>d</sup>	38.6±1.4 <sup>c</sup>	206±7 <sup>cc</sup>	7 563±83 <sup>a</sup>
	1:1:0.3	22.8±0.5 <sup>e</sup>	34.8±0.2 <sup>c</sup>	187±3 <sup>df</sup>	7 088 ±135 <sup>ab</sup>
	1:1:0.5	19.8±0.7 <sup>f</sup>	16.4±1.0 <sup>d</sup>	175±7 <sup>d</sup>	5 855 ±332 <sup>bc</sup>
SBE+OPT+EFB	1:1:0.1	25.1±0.7 <sup>d</sup>	38.0±2.4 <sup>c</sup>	198 ±2 <sup>ef</sup>	7 351 ±51 <sup>ac</sup>
	1:1:0.3	25.2±0.4 <sup>d</sup>	37.8±1.2 <sup>c</sup>	196 ±4 <sup>ef</sup>	7 170±242 <sup>ac</sup>
	1:1:0.5	20.1±0.5 <sup>f</sup>	17.2±0.5 <sup>d</sup>	163 ±4 <sup>g</sup>	5 444±591 <sup>b</sup>
*MAC (1)		1 000-7 500	2 500-4 000	-	-
*MAC (2)		60-150	100-300	-	-
*MAC (3)		300	1 500	-	-
**TAV		60-500	200-1 500		

Note: SBE - spent bleaching earth, CL - chicken litter, OPT - oil palm trunk, OPF - oil palm frond, EFB - empty fruit bunch. \*MAC - maximum allowable concentration: (1) for land application of compost recommended by the Commission of European Communities, 1986, (2) for trace metals in agricultural soils (Kabata-Pendias, 2011) and (3) for fertilisers used in agricultural soils in Finland (Kabata-Pendias, 2011). TAV - trigger action value for trace metals in agricultural soils (Kabata-Pendias, 2011). Groups in a column detected as different are marked with different letters (a, b, c, *etc.*), while groups in a column detected as identical are marked with the same letter.

TABLE 4. NUTRIENT CONTENT (mean ± SD, N=3) OF THE ENRICHED FERTILISERS BEFORE AND AFTER AMENDMENTS WITH CHEMICALS

Source	Total N (%)	Total P (as P <sub>2</sub> O <sub>5</sub> ) (%)	Extractable K (as K <sub>2</sub> O) (%)	Total S (%)
Urea (A)	45.6±0.3	-	-	-
CIRP (B)	-	25.2±1.0	-	-
MOP (C)	-	-	66.1±3.2	-
SBE:OPT:CL (1:1:0.5) (D)	0.65±0.00	1.59±0.22	1.63±0.00	0.20±0.01
<i>*Amendments</i>				
Fertiliser mix 1 (D+A+B+C)	1.57±0.06(2)	1.93±0.03(2)	1.94±0.09(2)	0.15±0.00
Fertiliser mix 2 (D+A+B+C)	1.74±0.11	1.83±0.03	1.32±0.02	0.14±0.04
Fertiliser mix 3 (D+A)	1.91±0.07	1.42±0.22	1.01±0.06	0.18±0.01
Fertiliser mix 4 (D+B)	0.32±0.01	1.97±0.01	1.03±0.10	0.20±0.01
Fertiliser mix 5 (D+A+B)	1.90±0.06	1.95±0.04	1.01±0.03	0.19±0.01

Note: Figure in bold parentheses depicts targeted N:P:K values. CIRP - Christmas Island rock phosphate. MOP - muriate of potash. \* Amendments achieved via experimental fortification and actual analyses of the following: fertiliser mix 1= D 100 g (95.10 %) + A 2.96 g (2.82 %) + B 1.63 g (1.55 %) + C 0.56 g (0.53 %), fertiliser mix 2= D 94.85 g (94.85 %) + A 2.96 g (2.96 %) + B 1.63 g (1.63 %) + C 0.56 g (0.56 %), fertiliser mix 3= D 97.04 g (97.04 %) + A 2.96 g (2.96 %), fertiliser mix 4= D 98.37 g (98.37 %) + B 1.63 g (1.63 %), fertiliser mix 5= D 95.41 g (95.41 %) + A 2.96 g (2.96 %) + B 1.63 g (1.63 %).



characteristics for use as soil amendment. The total N, P and K in this blend increased largely while the C:N ratio greatly reduced from 293 to 36 compared to the raw SBE. Besides, the micronutrients are well below the MPC thus safely serving as an important plant nutrient source for crops growing in soils with micronutrient deficiencies. This blend can be further enriched with mineral fertiliser to achieve a desired targeted NPK of 2:2:2 which could commensurate with requirements for vegetable crops growth. In general, the conversion of SBE into value-added product through blending with other bio-based and chemical nutrients contributes to a sustainable agriculture and preserves the environment.

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### REFERENCES

- ABOLLINO, O; ACETO, M; MALANDRINO, M; SARZANINI, C and MENTASTI, E (2003). Adsorption of heavy metals on Na-montmorillonite. Effect of pH and organic substances. *Water Research*, 37: 1619-1627.
- ABOULENIEN, F; FUJIWARA, W; NAMBA, Y; KOSSEVA, M; NISHIO, N and NAKASHIMADA, Y (2010). Improved methane fermentation of chicken manure via ammonia removal by biogas recycle. *Bioresource Technology*, 101: 6368-6373.
- AHMAD, R; NASEER, A; ZAHIR, Z A; ARSHAD, M; SULTAN, T and ULLAH, M A (2006). Integrated use of recycled organic waste and chemical fertilizers for improving maize yield. *International J. Agriculture & Biology*, 8(6): 841-843.
- AI-ZAHRANI, A A; AI-SHAHRANI, S S and AI-TAWIL, Y A (2000). Study on the activation of Saudi natural bentonite. Part II: Characterization of the produced active clay and its test as an adsorbing agent. *J. King Saud University*, 13(2): 193-203.
- ARIAS-ESTÉVEZ, M; LÓPEZ-PERIAGO, E; NÓVOA-MUÑOZ, J C; TORRADO-AGRASAR, A and SIMAL-GÁNDARA, J (2007). Treatment of an acid soil with bentonite used for wine fining: effects on soil properties and the growth of *Lolium multiflorum*. *J. Agricultural and Food Chemistry*, 55: 7541-7546.
- BOEY, P L; SALEH, I M; SAPAWA, N; GANESAN, S; MANIAM, G P and ALI, D M H (2011). Pyrolysis of residual palm oil in spent bleaching clay by modified tubular furnace and analysis of the products by GC-MS. *J. Analytical and Applied Pyrolysis*, 91: 199-204.
- BORAH, M; SURESH, K and JATTY, S K (2008). Alternate method for determination of CEC of clay minerals. *Clay Research*, 27 (1 & 2): 25-33.
- CHEN, Q; ZHANG, X S; ZHANG, H Y; CHRISTIE, P; LI, X L; HORLACHER, D and LIEBIG, H P (2004). Evaluation of current fertilizer practice and soil fertility in vegetable production in the Beijing region. *Nutrient Cycling in Agroecosystems*, 69: 51-58.
- CHEONG, K Y; LOH, S K and SALIMON, J (2014). Effect of spent bleaching earth based bio organic fertilizer on growth, yield and quality of eggplants. *AIP Conf. Proc.* 1571:744 <http://dx.doi.org/10.1063/1.4858743>.
- COMMISSION OF THE EUROPEAN COMMUNITIES (EU) (1986). Council directive (86/278/EEC) on the protection of the environment, and in particular of the soil, when sewage sludge is used in agriculture. *Off. J. European Community L181 (Annex 1A)*. p. 6-12.
- CROCKER, J; POSS, R; HARTMANN, C and BHUTHORNDHARAJ, S (2004). Effects of recycled bentonite addition on soil properties, plant growth and nutrient uptake in a tropical sandy soil. *Plant and Soil*, 267: 155-163.
- DEPARTMENT OF VETERINARY SERVICES (DVS) (2009). Ministry of Agriculture and Agro-Based Industry Malaysia. <http://www.dvs.gov.my/statistik>.
- EPSTEIN, E (1965). Mineral metabolism. *Plant Biochemistry* (Bonner, J and Varner, J E eds.). Academic Press, London. p. 438-466.
- GIBON, V; GREYT, W D and KELLENS, M (2007). Palm oil refining. *European Journal Lipid Science Technology*, 109: 315-335.
- KABATA-PENDIAS, ALINA (2011). *Trace Elements in Soils and Plants*. 4<sup>th</sup> ed., CRC Press, USA. p. 20-286.
- KHALID, H; ZIN, Z Z and ANDERSON, J M (1999). Quantification of oil palm biomass and nutrient value in a mature plantation. I: Above-ground biomass. *J. Oil Palm Res. Vol. 2 (1)*: 23-32.

- LOH, S K and CHOO, Y M (2013). Prospect, challenges and opportunities on biofuels in Malaysia. *Advances in Biofuels* (Pogaku, R and Hj Sarbatly, R eds.). Springer. New York. p. 3-14.
- LOH, S K; JAMES, S; NGATIMAN, M; CHEONG, K Y; CHOO, Y M and LIM, W S (2013). Enhancement of palm oil refinery waste - spent bleaching earth (SBE) into bio organic fertilizer and their effects on crop biomass growth. *Industrial Crops and Products*, 49: 775-781.
- LU, L L; WANG, X D and XU, M H (2010). Effect of zinc and composting time on dynamics of different soluble copper in chicken manure. *Agricultural Sciences in China*, 9 (6): 861-870.
- MATERECHERA, S A and MKHABELA, T S (2002). The effectiveness of lime, chicken manure and leaf litter ash in ameliorating acidity in a soil previously under black wattle (*Acacia mearnsii*) plantation. *Bioresource Technology*, 85: 9-16.
- NÓVOA-MUÑOZ, J C; SIMAL-GÁNDARA, J; FERNÁNDEZ-CALVIÑO, D; LÓPEZ-PERIAGO, E and ARIAS-ESTÉVEZ, M (2008). Changes in soil properties and in the growth of *Lolium multiflorum* in an acid soil amended with a solid waste from wineries. *Bioresource Technology*, 99: 6771-6779.
- N'DAYEGAMIYE, A (2009). Soil properties and crop yields in response to mixed paper mill sludges, dairy cattle manure, and inorganic fertilizer application. *Agronomy Journal*, 101 (4): 826-835.
- PILLER, G; FUKUSHIMA, M and IWABORI, S (2003). Growth responses of three New Zealand Northern highbush blueberry cultivars (*Vaccinium corymbosum*) to nutrient availability. *J. Japan Society Horticulture Science*, 72 (1): 13-17.
- RAO, J R; WATABE, M; STEWART, T A; MILLAR, B C and MOORE, J E (2007). Pelleted organo-mineral fertilisers from composted pig slurry solids, animal wastes and spent mushroom compost for amenity grasslands. *Waste Management*, 27: 1117-1128.
- REDDING, M R (2011). Bentonites and layered double hydroxides can decrease nutrient losses from spent poultry litter. *Applied Clay Science*, 52: 20-26.
- SELLAMI, F; JARBOUI, R; HACHICHA, S; MEDHIOUB, K and AMMAR, E (2008). Co-composting of oil exhausted olive-cake, poultry manure and industrial residues of agro-food activity for soil amendment. *Bioresource Technology*, 99: 1177-1188.
- SODA, W; NOBLE, A D; SUZUKI, S; SIMMONS, R; SINDHUSEN, L and BHUTHORNDHARAJ, S (2006). Co-composting of acid waste bentonites and their effects on soil properties and crop biomass. *J. Environmental Quality*, 35: 2293-2301.
- STICHNOTHE, H and SCHUCHARDT, F (2010). Comparison of different treatment options for palm oil production waste on a life cycle basis. *International Journal Life Cycle Assessment*, 15: 907-915.
- WANG, X Q; ZHANG, J Q; LIU, B H and QIU, D L (2010). Spent bleaching clay (SBC) from oil refining as a substrate for the spawn production of shiitake mushroom (*Lentinula edodes*). *African Journal of Biotechnology*, 9(53): 9007-9011.
- WENG, C H; TSAI, C Z; CHU, S H and SHARMA, Y C (2007). Adsorption characteristics of copper (II) onto spent activated clay. *Separation and Purification Technology*, 54: 187-197.