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The influence of Bomet red rock powder on composite organic fertilizers prepared from *Tithonia diversifolia* leaves and *Musa acuminata* (banana) stalks

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ABSTRACT

Common organic composite fertilizers lack several bio-chemical and physical traits desirable in other fertilizers. This study aimed at investigating the role of Bomet red rock, BRR powder in improving green composite fertilizers from *Tithonia diversifolia* leaves and *Musa acuminata* stalks. The composite fertilizer without BRR powder was prepared by mixing the crude extracts of the two plants before addition of BRR powder (10% wt.) The composite fertilizer with and without the powder were analyzed for physical–chemicals, functional groups, surface, morphology, particle size, crystallinity and chemical composition. BRR powder was proven to boost the fertilizers pH, conductivity and solubility in water while reducing its loss on ignition value. Carboxylic and amide groups were sequestered while imprinting silica and ferric groups onto the fertilizer surface. BRR agglomerated the composite fertilizer particles, in the process smoothening the edges while reducing the particle sizes. The structure of the fertilizers changed from monoclinic to hexagonal with the dominant potassium alumino-silicate phase being replaced by silica upon addition of BRR. BRR powder was therefore found to positively affect the green composite fertilizer and should thus be used in preparation of quality and durable green compound fertilizers.

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KEYWORDS
Bomet-red-rock; green composite fertilizer; *M. acuminata*; *T. diversifolia*

Introduction

Over time, arable lands are gradually depleting in nutrients. This necessitates the need to supplement these nutrients in such pieces of land. Fertilizers are used to replenish used up soil nutrients. Most fertilizers are however tailored to specific nutrients only. As a result, a single piece of land supporting different types of crops can receive multiple fertilizers. This has a negative impact on the recipient soil due to mixture of chemicals which eventually react to yield other products. Soil warmth, sunlight and moisture accelerate these reactions (Onwuka 2016). It is therefore necessary to encompass multiple plant nutrients into a single compound, i.e., as a composite fertilizer.

Composite (or compound) fertilizers have several plant nutrients in a single fertilizer compound. These types of fertilizers include; calcium ammonium nitrate (CAN), mono- and diammonium phosphate (DAP), urea, and nitrogen–phosphorus–potassium fertilizer (NPK) amongst

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others. These fertilizers have the basic plant requirements (nitrogen, phosphorus and potassium). Some of the composite fertilizers have these basic nutrients as well as other minor nutrients. As a rule of thumb, a good fertilizer should have appreciable concentrations of nitrogen (Liu et al. 2014). Nitrogen is crucial at the vegetative growth stage and plant reproduction as well as in boosting fruit sizes and disease resistance (Carranca, Brunetto, and Tagliavini 2018). Nitrogen constitute a large portion of amino acids, the building blocks of plants (Guedes et al. 2011). As crops age, amino acids senescence from the older tissues to the younger ones (Chaka and Osano 2019). Phosphorus is useful in promoting root and shoot growth of crops (Abdolzadeh et al. 2010). Phosphorus also forms a bulk of several amino acids and enzymes with fundamental roles in plant growth. Potassium promotes flowering, fruiting and general plant hardiness (Prajapati 2012). Several enzyme functions are also controlled by potassium (Wang et al. 2013).

Whereas composite fertilizers enjoy many benefits over the single ones, they are also faced with demerits of mineral fertilizers. The two major drawbacks of mineral fertilizers are cost and pollution. Not only are these fertilizers expensive, but they are also unavailable in rural areas where most of the farms are found. This further increase the costs as they require transporting from the areas of production and sale to the areas of application. Continuous application of mineral fertilizer onto a piece of land lead to soil pollution. Chemicals present in the fertilizers at excessive concentrations kill or chase away important soil micro-organism (Di Benedetto et al. 2017). Application of mineral fertilizer by broadcasting method also contribute to air and water pollution. Volatile mineral fertilizers applied during the day or warm seasons also volatilize into the atmosphere to cause air pollution.

Composite green fertilizers partially solve the demerits of composite fertilizers (i.e., cost and pollution effects). However, these fertilizers do not contain enough concentrations of the prerequisite plant nutrients required. Composite green fertilizers have to be prepared from several sources in order to encompass all the necessary plant nutrients (Liu et al. 2014). This yields quite voluminous fertilizers and the process is tedious. However, the volume of green composite fertilizers can be minimized by mixing agricultural residues with rocks containing nutrients lacking in the vegetatious matter. This method of preparation of composite fertilizers involves agglomeration of the residues with the rock to form a compact solid mixture.

Tithonia diversifolia leaves have frequently been used to prepare organic fertilizers. Foliar fertilizers of *T. diversifolia* are common top-dress nitrogen boosters popular in most Kenyan rural areas. *Musa acuminata* (banana) stalks are quite rich in potassium (Jiwan and Tasleem 2018). *M. acuminata* stalks have also been severally used in preparation of organic fertilizers. The stalks are also excellent water retarders and can store water in the soil for long periods. Bomet red rock, BRR is a silicateous rock found in Bomet region, on the southern part of the Eastern Rift Valley. The rock find uses as a farm supplement in some few parts of Bomet region. The native farmers used BRR to help coagulate together the organic fertilizer matrix thus enhancing its physical appearance for handling, storage and durability. However, inclusion of BRR might interfere with other fertilizer properties; either negatively or positively (unaware to the farmer). For example, like most other rocks, BRR is likely to supplement essential inorganic minerals (such as phosphates) to the organic fertilizer. It is therefore crucial, to study the exact role of addition of BRR, from a scientific perspective in preparation of green composite fertilizers. Several chemical parameters and characterization analyses were conducted analyzing the green composite fertilizer with and without BRR powder.

Materials and methods

Design of experiment

Fresh *T. diversifolia* leaves and *M. acuminata* stalks were separately macerated to obtain their respective extracts. Water was added to aid in the extraction process. A mixture in the ratio of *T.*

diversifolia: *M. acuminata* (2:1) was obtained. A sample of the mixture was concentrated by shade-drying to obtain solid residues which were further dried and crushed to obtain powder samples. The rest of the mixture was agglomerated by adding BRR powder (ratio 3:1) such that the final constituent ratio was *T. diversifolia*: *M. acuminata*: BRR was 6:3:1. The final mixture was also dried and ground to obtain a final composite powder. Both powders (with and without BRR) were subjected to physical-chemical analysis, functional group analysis by Fourier transform infrared (FT-IR) spectroscopy, surface morphology by scanning electron microscopy (SEM), crystallinity by X-ray diffractometry (XRD) and particle size analysis by transition electron microscopy (TEM).

Preparation of the formulations and physical-chemical analysis was conducted in Maasai Mara University, Kenya (1.1041°S, 36.0893°E) while characterization was done at The University of Johannesburg, South Africa (26.2041°S, 28.0473°E).

Materials

The following equipment were used during the study;

Fourier Transform Infrared (PerkinElmer Spectrum Version 10.4.2), Scanning Electron Microscope (VEGA3 TEGSCAN Model) and X-Ray Diffractometer (Gonio XPERT-PRO; Spinner PW3064), Transmission Electron Microscope, JEM-2100 multipurpose, 200 kV analytical TEM.

Methods

Physical–chemical parameters of the green composite fertilizers

Several physical–chemical parameters including, pH, electrical conductivity, solubility, total solids content and loss on ignition tests were conducted.pH analysis was done using a pH meter (Hanna G-114) while electrical conductivity was done using a conductivity meter (Jenway 6510). Solubility of the green fertilizers was done by wet chemistry, checking the solubility points of the solutes (fertilizer) in water by gradual increase in temperature. The total solids and loss on ignition values were also done by standard wet chemistry methods as shown below;

For total solids, TS, 10.000 g of sample was weighed, M_1 using an Analytical balance and then placed in an oven conditioned at 105 °C for 6 hr before removing, cooling (in a desiccator) and reweighing to a constant mass, M_2. For loss on ignition, LOI, 10.000 *g* of sample, M_1 was put into a pre-weighed crucible and subjected to high temperature (>540 °C) in an oven for 1 hour, cooled in a desiccator then reweighed again to a constant mass, M_3.

$$\%\mathrm{TS} = \frac{M2}{M1} \times 100\% \tag{1}$$

$$\%\mathrm{LOI} = \frac{\mathrm{M3}}{\mathrm{M1}} \times 100\% \tag{2}$$

IR functional groups

The fertilizer samples were slowly concentrated to remove moisture until all the water dried. The samples were then cast into pellets using KBr pellet before analyzing for functional groups using IR Spectrometer.

Surface and morphological analysis

Scanning electron microscopy was used to impart a means to directly observe the morphological appearance of the samples. The images were captured using a field emission Scanning electron microscope. Prior to imaging, the samples were coated using the gold sputtering method.

Crystal properties, phases and chemical composition

The crystallinity index of the samples was analyzed using XRD. The samples in form of milled powder was placed on steel sample holders and leveled to obtain total and uniform X-ray exposure. The samples were then analyzed at room temperature (25 °C with a monochromatic CuKα radiation source ($\lambda = 0.1539$ nm) in the step-scan mode with a 2θ angle ranging from 10° to 60° with a step of 0.04 and scanning time of 5.0 min. To calculate the crystallinity of the different samples, the crystallinity index CrI was determined based on the reflected intensity data following the method, Eq. (3).

$$C_rI(\%) = \frac{I_{002} - I_{am}}{I_{002}} x100 \quad (3)$$

Here, I_{002} is the maximum intensity of the $_{002}$lattice diffraction peak and I_{am} is the intensity scattered by the amorphous part of the sample. The diffraction peak for plane $_{002}$ was located at a diffraction angle around $2\theta = 22°$ and the intensity scattered by the amorphous part was measured as the lowest intensity at a diffraction angle around $2\theta = 16°$.

Morphology and particle size analysis

Morphological properties and particle sizes of the samples was determined using Transmission Electron Microscope. The samples were dispersed in suitable aqueous suspension; (0.1% wt.) then deposited on the surface of a copper grid coated with a thin carbon film. Subsequently, the samples were dried before carrying out TEM analysis at an accelerating voltage of 100–120 kV. Particle size was calculated using ImageJ software package.

Data analysis

Data were recorded as mean ± standard deviation. Three degrees of freedom at a confidence level of 95% were constantly used during the study. Ms Excel and Originlab, version 6.5 statistical software were used.

Results and discussions

Physical–chemical parameters

The pH values of both composite fertilizer samples (with and without the rock powder) were slightly acidic. The composite fertilizer without BRR was more acidic (pH = 6.05 ± 0.05) owing to more volatile matters present. Most of the volatile matter have organic acids which contribute in lowering the pH values. The relatively higher pH values of the composite fertilizer with BRR can be attributed to increase in alkaline matter such as silica and alumina present in BRR. Nevertheless, the deviation in the pH values was insignificant. The values obtained are well in agreement with the pH range of most composite fertilizers (Zhang et al. 2017). Very acidic fertilizers can interfere with biosorption of crucial plant nutrients as well as intoxicate soil microorganisms (Bargaz et al. 2018). Prolonged use of such fertilizers leads to acidification of the soil prompting for soil liming. The solubility of the composite fertilizer with BRR was higher (23.50 ± 0.55/100 mL water at 25 °C) compared to that without the rock powder (16.00 ± 2.10/

Table 1. Physical–chemical properties of green composite fertilizer samples.

	Green composite fertilizer	
Parameter	Without BRR	With BRR
pH	6.05 ± 0.05	6.55 ± 1.00
Solubility (g/100 mL water at 25 °C)	16.00 ± 2.10	23.50 ± 0.55
Electrical conductivity (μS)	47.10 ± 6.90	89.00 ± 12.05
Total solids (%)	22.52 ± 0.30	33.30 ± 0.95
Loss on ignition, LOI (%)	13.14 ± 0.55	9.06 ± 0.01

Physical–chemical parameters of the green composite fertilizer samples prepared.

100 mL water at 25 °C). Table 1 summarizes the physical-chemical parameters of the synthesized green composite fertilizer samples.

The difference in solubility is as a result of more inorganic matter present in the fertilizer with BRR compared to that without. Most organic matter are non-polar thus insoluble in water. The vegetatious composite fertilizer (without BRR) had more organic matter present. High solubility of fertilizers enables them to be applied by spraying and top-dressing. These modes of fertilizer application are easy and reduce the time taken for application. The electrical conductivity value of the composite fertilizer sample with BRR was significantly higher (89.00 ± 12.05 μS) than that without BRR (47.10 ± 6.90 μS) at $p \geq .05$ ($n = 3$). The high conductivity of the sample with BRR powder can be attributed to presence of inorganic compounds in the BRR. Electrical conductivity, EC of a fertilizer sample dictates the formulation and solvent to be used for conveying, say during spraying. A high electrical conductivity in water implies that most of the fertilizer components are actually soluble in the water itself. Some of the ions responsible for high EC include alkali ions (Na and K) which are quite fundamental in plants growth. High electrical conductivity of fertilizers is a function of both total solids content and solubility. It is therefore not surprising that the total solids content in the composite fertilizer with BRR was higher than that without. The LOI value of the composite fertilizer sample without BRR was higher than that with BRR due to abundance of organic compounds in it. This results from the vegetatious *T. diversifolia* and *M. acuminata* extracts used during its preparation. High LOI values depict more volatilization of the fertilizer at warm conditions. It is thus feasible not to apply the green composite fertilizer without BRR during hot and sunny afternoons.

Functional groups analysis

There was a great disparity in the functional group spectra of the vegetatious composite fertilizer containing *T. diversifolia* and *M. acuminata* only and that with BRR powder. Addition of the powder into the vegetatious sample effectively sequestered most of the organic functional groups; in the process imprinting a C–O–H peak at 1006.81 cm^{-1} and several semi-ionic bonds before 1000 cm^{-1}. These changes in the spectra are illustrated in Figure 1.

Peaks due to bonded water in the vegetatious sample between 3800 and 3900cm^{-1} were quenched by addition of the rock powder. This can be attributed to exothermic reaction during the agglomeration of the vegetatious samples with the rock powder sample to form a compact dry composite fertilizer. Carboxylic OH group at 3321.96 cm^{-1} and C = O group at 1637.09 cm^{-1} peaks due to amino acids and carboxylic acids in the vegetatious matrix were confirmed (Bagchi et al. 2009). However, after agglomeration with the rock powder these peaks disappeared. Presence of these peaks combined with short peaks at 2400 cm^{-1} (due to nitride groups) is evidence of nitrogenous compounds in the composite fertilizer. Nitrogen is a key player in fertilizers and occur either as elemental nitrogen or ionic nitrogen combined with other elements such as carbon, oxygen or hydrogen (National Center for Biotechnology Information 2020). There was a shallow peak in the vegetatious fertilizer spectra between 2050 and 2150 cm^{-1}. Gupta, Jelle, and Gao (2015) have reported nitrile and phosphaalkyne groups to appear at this region. These peaks

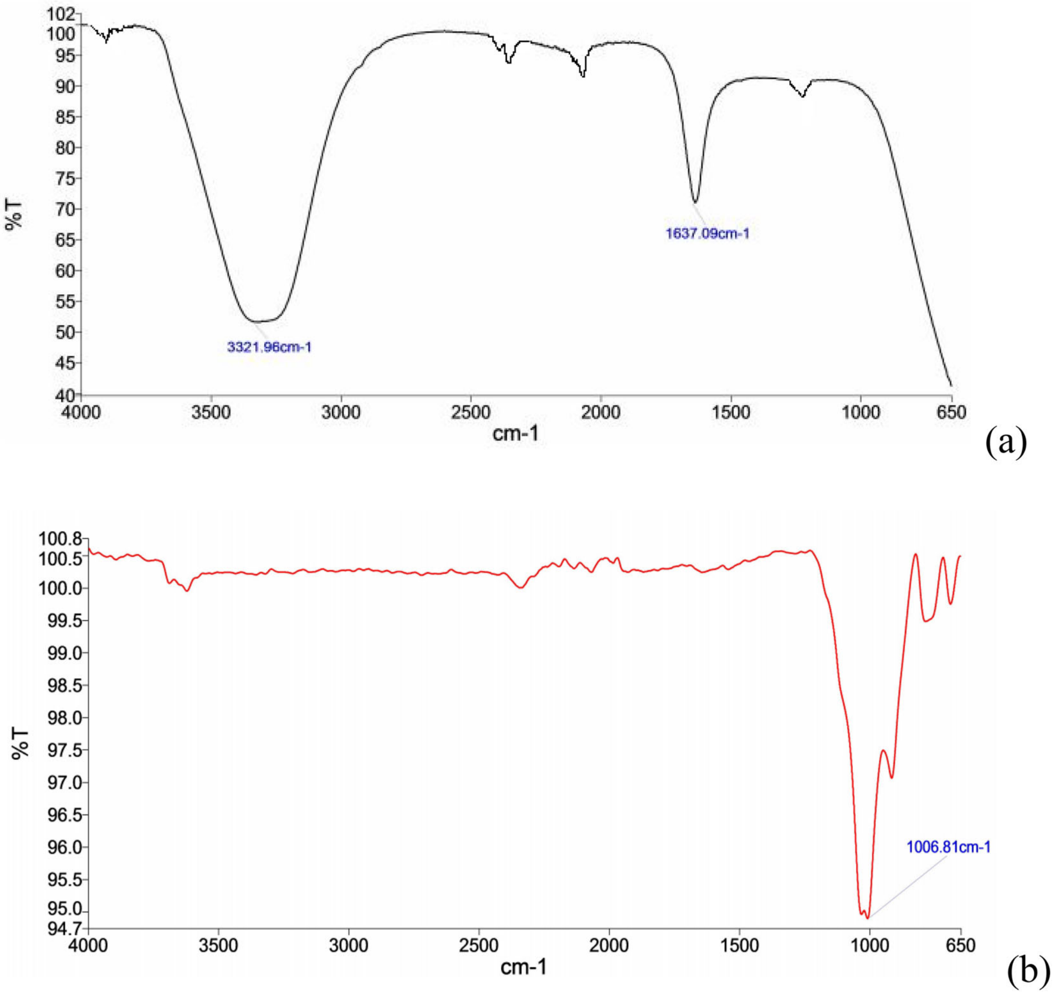

Figure 1. FT-IR spectra of green composite fertilizer without BRR (a) and containing BRR (b). From the spectra, addition of BRR powder sequestered several peaks while imprinting others.

were quenched upon agglomeration with BRR powder. This implies that the peaks are still present but not easily ionizable. Silica, ferric and potassium groups were observed between 650-950cm^{-1} after addition of the rock powder. This underlines the significance of BRR in provision of key minerals required in fertilizers.

Surface and morphological analysis

Addition of BRR powder to the vegetatious composite fertilizer not only changed its surface properties but also affected the morphology of the particles. The vegetatious particles were scattered without any specific uniformity in size before agglomeration (Figure 2a). There were wide gaps in between particles resulting from water and other liquid porous material in *T. diversifolia* and *M. acuminata* extracts. Most of the particles were less adhesive to each other due to less interactions between polar and non-polar compounds in the two extracts. This was not the case in the samples containing the red rock powder. Figure 2 illustrates the surface and morphological properties of the composite samples before and after agglomeration.

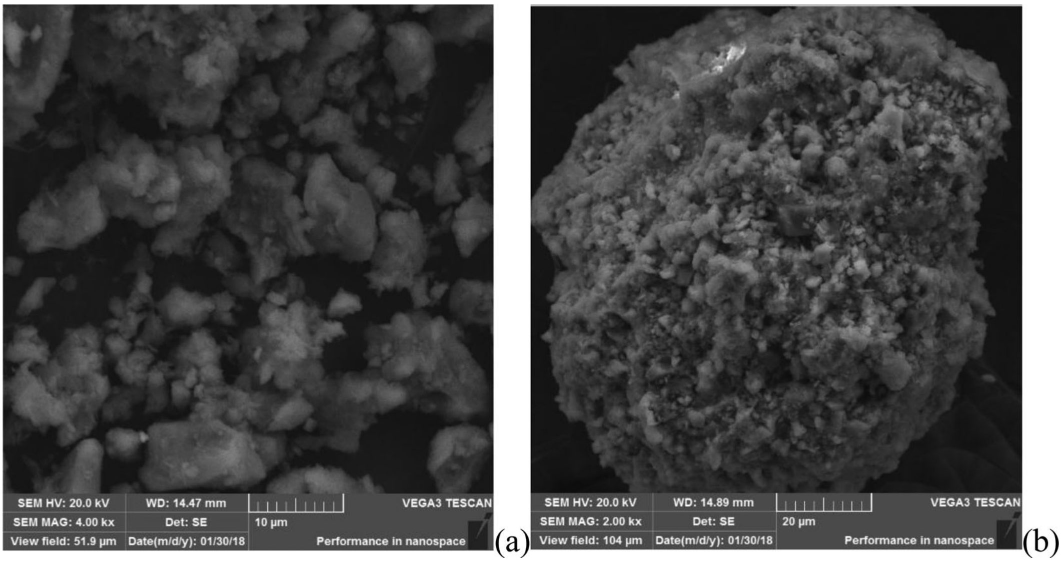

Figure 2. Surface and morphological properties of the composite fertilizer before agglomeration (a) and after agglomeration (b). The particles were quite diverse in their properties with the samples containing BRR powder being quite adhesive to each other unlike the vegetatious samples.

The BRR powder gelled the respective monomers of the extracts in the vegetatious fertilizer mixture into a compact body with more finite characters as observed in Figure 2b. Silicates and other alkalis common in ground rocks are quite insoluble in polar media such as the water in the vegetatious mixture. Reaction of BRR powder with the mixture thus led to precipitation; effectively filling up the pores in the vegetatious fertilizer. The edges of the compound were also quite smoother with more uniformity in the particles present. Cropnutrition (2020) described fertilizer particles with a rocky background (such as phosphate rock) to adopt smooth edges when combined with nitric acid during formation of compound fertilizers. The average particle size of the fertilizer samples before agglomerating with BRR powder were also larger than those after agglomeration process. Agglomeration reduced the porosity of the compounds compacting them into smaller more crystalline compounds. This has a benefit to fertilizer producers as the compact fertilizer compounds are easier to pack and transport compared to the porous ones. There was little variation in the shapes of the particles after addition of the red rock powder. The adhesive nature of the composite fertilizer sample (with BRR powder) indicates more degree of wettability. This fertilizer can thus be applied in several formulations either directly as granules or combined with water as foliar for spraying to crops.

Crystallinity, phases and composition of the composite fertilizer samples

X-ray analysis of the green composite fertilizer indicated a large difference in the crystallinity and thus the composition of the fertilizers with and without the BRR powder. The green vegetatious composite fertilizer diffractogram corresponded to that of orthoclase (a potassium alumino-silicate compound). This compound was monoclinic in crystal structure with a relative density of $2.60\,\mathrm{g\ cm^{-3}}$. This density is by far more than that of pure water implying abundance of ionic compounds present. The density was close to that of Ortas and Lal (2012) who obtained close fertilizer density for composite nitrogen and phosphorus fertilizers. The volume of the vegetatious compound fertilizer powder was $719.18 \times 10^6\ \mathrm{pm}^3$. Four formula units ($Z = 4$) could be attributed to this composite but no immediate reference material could be attributed (Reference Intensity Ratio, RIR of the diffractogram was blank). The diffractogram of the vegetatious composite

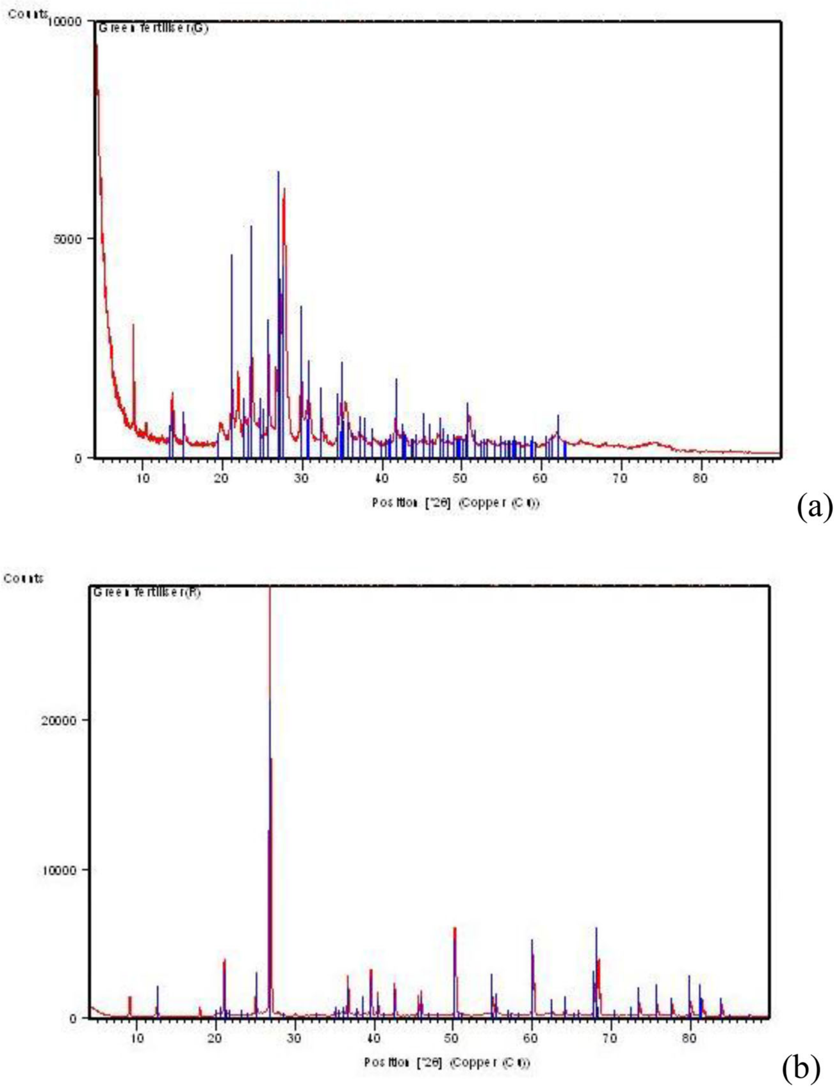

Figure 3. X-ray diffractograms of powder from the green composite fertilizer without (a) and with BRR powder (b).

fertilizer changed upon agglomeration with the red rock powder. The new mixture had a diffractogram corresponding to that of a silicatious rock, citing high silica content in the rock powder. The mixture also corresponded to kaolinite and anorthic rocks (Deer, Howie, and Zussman 1992). The mixture adopted a hexagonal crystal structure with $Z = 3$. Therefore, three formula units could be directly attributed to the composite mixture. The RIR value of the mixture was 1.61 indicating similarity with other composite fertilizers. The density of the composite fertilizer with the red rock powder increased slightly on addition of this powder (from 2.60 to 2.64 g cm^{-3}). This is due to increased dense ionic content as a result of adding the red rock powder. The volume of the composite fertilizer powder with BRR dramatically reduced from 719.18 (without BRR) to 113.18×10^6 pm^3. This is because the vegetatious mixture (without red rock) was more organic with a porous structure thus occupied a larger volume. Addition of BRR powder agglomerated the fertilizer particles together to form small definite units. The small volume, crystallinity and density of the vegetatious mixture allows for compact packing of these fertilizer for storage or transportation purposes.

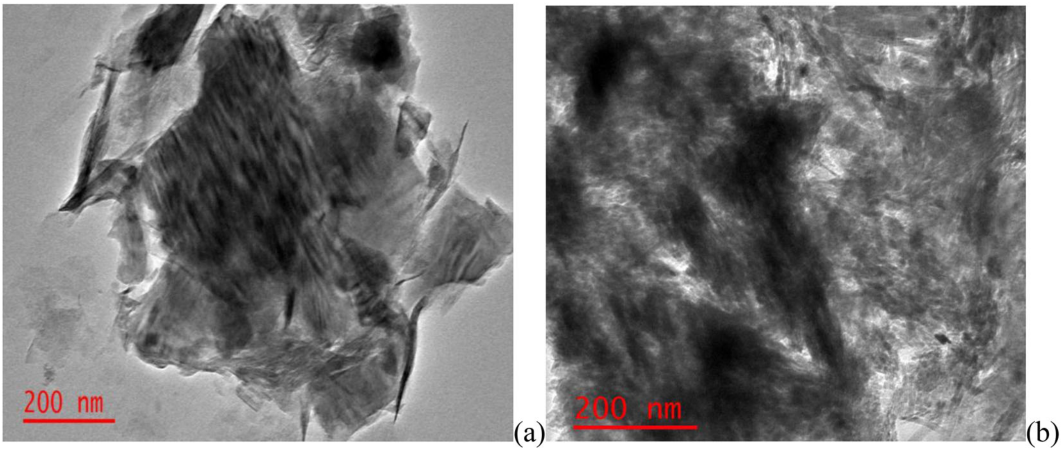

Figure 4. TEM images for the composite fertilizer without (a) and with (b) BRR powder.

The chemical composition of the green composite fertilizer without BRR was analyzed as: “’SiO_2” 46.54%, “TiO_2” 0.17%, “Al_2O_3” 36.37%, “Fe_2O_3” 0.72%, FeO 0.36%, CaO 0.22%, MgO 0.50%, “K_2O” 8.06%, “Na_2O” 0.46%, “Li_2O” trace, “H_2O” 6.83%, F 0.02%, and containing excess water over usual mica formula. Silica, alumina and potassium oxide were the most abundant minerals present. All the three minerals are quite crucial in plant growth and development. The d-spacings and relative intensities of the vegetatious (Figure 3a) and non-vegetatious (Figure 3b) green composite fertilizer samples are illustrated in Figure 3.

The chemical composition of the green composite mixture changed with addition of BRR. The mixture was analyzed as; LOI 4.43%, “SiO_2” 82.9%, “Al_2O_3” 11.0%, “Fe_2O_3” 0.71%, “CaO” 0.10%, “MgO” 0.11%, “TiO_2” 0.29%, “K_2O” 0.38%, “Na_2O” 0.09%, “SO_3” 0.03%, “P_2O_5” 0.02%. The LOI content (4.43%) was attributed to the organic matter from the vegetatious components of the mixture. The LOI value obtained at this point was quite different to that obtained at (“Physical–chemical parameters” above) due to the different mode of sample preparations for each test. Presence of silica in BRR dominated the composition of the composite fertilizer (82.9%). Only alumina content was more than 1% from the remaining compounds.

Particle size analysis of the green composite fertilizer samples

The TEM images of the green composite fertilizers indicated a reduction in particle size after addition of BRR powder. Figure 4 illustrates the TEM images before (a) and after addition of BRR powder (b) to the green composite fertilizer.

The TEM image of the fertilizer sample without BRR indicated the bulk of the minerals were concentrated at the center whereas vast parts lacked these minerals. This indicated a high degree of imbalance in nutrient position. The case was different in the fertilizer particles with BRR. The vegetatious fertilizer without the rock powder appeared to be quite dispersed and porous. From Figure 4a, the majority of the fertilizer particles were quite large. The average size was calculated to be 74.6 nm and skewed toward the right end (larger particles). This was by far much larger than the particles with the rock powder (29.9 nm). Inclusion of BRR powder into *T. diversifolia* and *M. acuminata* extracts coagulated the particles together. The resultant mixture was easily grindable leading to the small particle sizes. Liu and Lal (2015) obtained close fertilizer particle sizes in synthetic apatite nanoparticles as a phosphorus fertilizer for soybean (*Glycine max*). The smaller the fertilizer particles, the more effective it is. This is because a small volume of the fertilizer package contains a similar concentration of plant nutrients with a larger bag containing fertilizer with large particle sizes. This is a desired storage and transportation fertilizer quality.

Conclusions

Inclusion of BRR powder into the green composite fertilizer from *T. diversifolia* and *M. acuminata* extracts improved the quality of the composite fertilizer. The powder increased the pH, solubility and electrical conductivity of the composite fertilizer while lowering its LOI value dramatically. Carboxylic and amide groups in the green fertilizer were effectively sequestered on addition of BRR powder. The green composite fertilizer without BRR were found to be monoclinic and corresponded to potassium alumino-silicate compounds. Upon addition of BRR, the composite fertilizer formed had a hexagonal structure with abundant silica content. Addition of BRR slightly increased the density of the composite fertilizer while shrinking its size. The reduction in particle size of the green composite fertilizer was confirmed by both SEM and TEM analyses. Silica, potassium, aluminum and other important plant nutrients were found present in the composite fertilizer.

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Disclosure statement

The authors declare to have no conflicts of interest.

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Data availability statement

All data used is enclosed within this manuscript and any other supplementary sheet attached.

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