Characteristics and agrarian properties of biochar derived from pyrolysis and co-pyrolysis of rubberwood sawdust and sewage sludge for further application to soil improvement

Liaqat Ali

Prince of Songkla University

Arkorn Palamanit

Prince of Songkla University

Kuaanan Techato

Prince of Songkla University

Md. Shahariar Chowdhury

Prince of Songkla University

Khamphe Phoungthong (✉ khamphe.p@psu.ac.th)

Prince of Songkla University <https://orcid.org/0000-0003-4218-1439>

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Abstract

This study investigated the characteristics of biochars derived from pyrolysis of rubberwood sawdust and sewage sludge, and co-pyrolysis of these feedstocks at the ratios of 50:50 and 75:25. All feedstocks were pyrolyzed at 550°C in slow pyrolysis with a moving bed pyrolysis reactor. Then, the investigated characteristics of biochar samples were determined and are reported. The rubberwood sawdust biochar (RWSB) had a higher in carbon content (86.70 wt%) and was lower in oxygen content (7.89 wt%), while sewage sludge biochar (SSB) had a higher ash content (65.61 w%) and a low carbon content (24.27 wt%). The weight losses of biochars were observed in TGA while the DTG graphs show degradation rate of biochar produced in pyrolysis specific conditions. RWSB had a lower content of Si, Fe, K, Na and P than SSB as observed by XRF. The pH of RWSB, SSB and the blends (50:50, 75:25) of biochars was in the range 8.41–10.02. High carbon content of the biochar confirms potential for its use in carbon sequestration. The large pore volumes and specific surface areas of biochars were found by SEM and BET. The available functional groups in biochars were C–O, C = C, and C–H as confirmed by FTIR. Water holding capacity (WHC) and water releasing ability (WRA) of RWSB, SSB, and the blends (50:50 and 75:25) of biochars were 1.01–3.08 (mL/g) and 1.19–52.42 (wt%), respectively. In this study, our results show that blending woody and non-woody based biochars can help address nonpoint source contaminants in environment. So, these all findings should develop as tune to parameters of thermal degradation of biomass and bio-biowaste with sustain and eco-friendly biochar production.

Introduction

The global population growth is increasing the demand for food production, while extensive use of fertilizers is essential to high crop yields. However, especially the phosphate fertilizers impact ecosystems with eutrophication of water bodies and algal blooms (Novais et al. 2018; Choi et al. 2019). Further, the exploitation of fields can eventually deplete them from some essential nutrients reducing the crop yields in the future. Sustainable approaches that avoid chemical fertilizer use may be ecologically more advantageous (Xiang et al. 2020). Cost-effective sustainable approaches have a high current priority in agricultural research (Lee et al. 2019). It is necessary to explore alternative techniques to maintain the sustainability agriculture while maintaining its productivity.

Various contaminants (phosphate, heavy metals, antibiotics, pathogens, chemical precipitation, dyes, greenhouse gases, volatile organic compounds and endocrine-disrupting chemicals) in the environment need to be managed, either preventing their emissions or mitigating them by cleanup measures. These pollutants can be caused by acid rain, ozone layer depletion, and global warming. Effective technologies to control or improve the environment are desired (Xu et al. 2019; Gopinath et al. 2021). Methods used in the past studies for efficient removal of contaminants such as heavy metals include adsorption using industrial solid waste, activated carbon, metal oxides, clay minerals, coagulation and filtration (Loganathan et al. 2014; Bashir et al. 2019; Vieira et al. 2020). Comparatively, among these techniques adsorption was the most effective and efficient alternative. In the adsorption method, biochar is a good option for treating heavy metal contaminations. Biochar rich in carbon is considered a green material for

removing hazardous elements, which can be metals/metalloids or non-metals in groundwater or land surface. This type of absorbent has zero or minimal impact on any ecosystem, is easy to use, has high efficiency at a low cost and is widely available (Jung et al. 2017; Chen et al. 2019; Yudha et al. 2019; Said et al. 2020).

Biochar can be produced from various abundant sources like forestry residues, agricultural wastes, industrial waste, municipal solid waste (MSW), or sewage sludge (SS) from wastewater treatment plants. According to a report by Miyaoka et al. (2017) the production of sewage sludge waste in Bangkok Thailand is 30,000 to 350,000 m³/day from wastewater treatment plants. These types of bio-wastes in a circular bioeconomy should be recycled in a value-added form (Phoungthong et al. 2018; Phoungthong and Suwunwong, 2020). The conversion to valuable biochar for sustainable crops (Abbas et al. 2021) fit that concept. Consequently, these types of biowastes might be reuse/recycled after pyrolysis at an elevated temperature to generate bioenergy, particularly from sewage sludge that is a widely available waste, although its energy efficiency is low because of the high moisture content, but this can be improved by mixing with other types of energy feedstocks. For example coal or rubberwood have been mixed with sewage sludge, and with paper waste for thermochemical conversion (by pyrolysis, gasification, and combustion) (Cabuk et al. 2020; Ali et al. 2021b). The conversion of lignocellulosic biomass or biowaste into biochar by pyrolysis and co-pyrolysis is strongly dependent on the type of reactor, residence time, types of feedstocks, and operating temperature (Li et al. 2019; Suwunwong et al. 2020a; Ali et al. 2021a). Pyrolysis is a technique converting waste residues to valorize them, and agricultural biomass can be converted into biochar, pyrolysis gas, and biofuel in the absence of oxygen by thermal processing. On the other hand, the concept of co-pyrolysis is gaining increased interest because of its efficiency with energy-efficient operation, and complements pyrolysis. Mostly the toxic elements can be removed or decrease in biochar after high temperature pyrolysis or co-pyrolysis (Nagy and Dobó 2020). Remarkably, after co-pyrolysis, the biochar surface area has increased and its pore diameter also can be improved depending on control of temperature and feedstock blend ratio.

Biochar is carbon dense material from solid waste with a high surface area (Lu et al. 2013; Suwunwong et al. 2020b). It can achieve an excellent adsorption ability for use in conditioning soil or water (Gunarathne et al. 2019; Suwunwong et al. 2021). On the other hand, biochar is environmentally friendly, with its distinctive characteristics including high specific surface, porosity, high adsorption capacity, and low toxic metal content (Mohanty et al. 2013; Hussain et al. 2021). Particularly the physicochemical characteristics of biochar depend on the type of feedstock and type of pyrolysis. The physicochemical characteristics of biochar, like polarity, surface area, atomic ratio, elemental composition, and porosity can be affected by those choices (Oliveira et al. 2017; Phoungthong et al. 2018).

The application of biochar is not limited to remediation of polluted soil, it can also increase the crop yield and improve properties of healthy soil. According to Sustainable Development Goals (SDGs-6), biochar is the best option for cleaning water and to adsorb soil pollutants (Smith et al. 2019). On the other hand, biochar can be applied in cosmetics, in removing dyes from sensitive surfaces, remove leachates from soil, etc. (Gul et al. 2015). For the soil fertility, biochar can play a key role as soil conditioner because of

its high carbon content, and it can improve the physicochemical and biological properties of soil. Organic carbon in soil increases water retention capacity (Glaser et al. 2002). Moreover, the water holding capacity (WHC) of soil depends on the specific surface of biochar and its hydrophobicity (Verheijen et al. 2010). Markedly, the biochar produced in this current study was derived from rubberwood sawdust (RWS) and sewage sludge (SS) biomass feedstocks and with different blended ratios (50:50, 75:25) and decomposed by pyrolysis and co-pyrolysis, and its pH was suitable for soil amendment and wastewater treatment, but after hydrothermal treatment carbonaceous process (HTCP) of biochar it could also filter pollutants from water. Both biomasses (rubberwood sawdust and sewage sludge) were decomposed at 550°C. A few studies have reported that the hydrothermal treatment carbonaceous process (HTCP) of waste sewage before pyrolyzing it can improve its heating values as well as reduce energy use and help solve the sludge pollution problem (Catallo and Comeaux 2008; vom Eyser et al. 2015; Das et al. 2016).

Sewage sludge derived biochar can contain elements like Pb and Cd that are not environmentally friendly. These can be treated with exchange reactions to other elements. For example, for the Pb^{2+} sorption by sewage sludge derived biochar it can be exchanged with other metals like Ca^{2+} , Mg^{2+} and other cations embedded in the biochar (Lu et al. 2012). There are some mineral components in biochar, including carbonates and phosphates that can play unique roles in stabilizing heavy metals in soils to salt precipitates, reducing their bioavailability. Nevertheless, the heavy metals and contaminants in sewage sludge biochar (SSB) are not of great concern and can be treated by cation exchange reaction processes (Cao et al. 2009). Thus, rubberwood sawdust biochar (RWSB) with large pore diameter and sewage sludge biochar (SSB) with large specific surface can be useful for the adsorption of hazardous elements from soil.

The aim of the present study was to assess the feasibility of biochar from lignocellulosic biomass feedstocks with non-lignocellulosic bio-waste for improvement of soil fertility, and to determine how pyrolysis temperature influences the physico-chemical characteristics of biochar. The determinations done to characterize the products were proximate analysis, ultimate analysis, X-ray fluorescence (XRF), TGA and H/C and O/C, pH, Carbon sequestration (CS), Scanning Electron Microscopy (SEM), Brunauer, Emmett and Teller (BET), FTIR, water holding capacity (WHC) analysis, and water releasing ability (WRA) analysis, after pyrolysis at 550°C.

Materials And Methods

Biochar preparation

Pyrolysis and co-pyrolysis of rubberwood sawdust (RWS) and sewage sludge (SS) biomass feedstocks were carried out by slow pyrolysis with a moving bed pyrolysis reactor of cylindrical shape. The reactor was preheated to the set pyrolysis temperature at 550 °C, and most of the organic volatiles from RWS and SS feedstock were released at this condition. The feedstocks were converted into biochar at a higher temperature. After completion of the processing, the biochar was taken from the reactor and kept in

containers for cool-down. Then the cool biochar was kept in desiccators to prevent absorption of moisture. Finally, the prepared biochars were used or tested further.

Determination of biochar characteristics

Basic components and atomic ratios

The characterization of biochar samples was done in terms of proximate analysis, ultimate analysis, XRF, TGA, pH, Carbon sequestration (CS), SEM, BET, FTIR, water holding capacity (WHC), water releasing ability (WRA) and H/C and O/C ratios. The ultimate analysis of CHNOS on a dry basis used Thermo Scientific FLASH 2000 Organic Elemental Analyzer (Thermo Scientific, Italy) with an in-house method, while the oxygen (O) of biochar was calculated as difference from other ultimate analysis values (Kongto et al. 2021). The proximate analysis was done on as received basis by using a Thermogravimetric analyzer (TGA), the Macro (TGA 701, LECO, USA) following the procedure in ASTM D7582. The H/C and O/C atomic ratios (on dry basis) in biochar were determined via Van Krevelen method, and were compared to various previous studies (Odeha et al. 2017).

Major and minor elements

The major and minor elements reported for the biochars include Si, Ca, Fe, K, Mg, Na, P, Cu, Zn, Mn, Cd and Pb. The biochar was subjected to X-ray fluorescence spectrometry (X-ray fluorescence spectrometer, Zetium, PANalytical, Netherlands).

Thermogravimetric analysis

Thermal decomposition of the biochars RWS, SS, RWS50:SS50 and RWS75:SS25 used TGA and thermal differential analysis DTA in a thermogravimetric analyzer (Perkin Emer, USA), according to ASTM E1131 procedure. The equipment was heated with N₂ atmosphere from room temperature to 50-1000 °C at 10 °C /min heating rate. Approximately 3 g sample was used in each run.

pH of biochar

The pH of biochar samples was measured (UB-10 Denver Instrument) at room temperature. The 1 g biochar sample was mixed with 20 ml of deionized water in a 50 ml glass bottle for 30 minutes on a shaker. The pH meter before use was calibrated with pH 4, pH 7 and pH 10 buffers. All cases were measured in triplicates (Krutof et al. 2020).

Carbon sequestration (CS)

The carbon sequestration index R_{50} was applied to assess thermal recalcitrance of the freshly produced biochars. The thermal recalcitrance of biochar can be acquired from TG analysis biochar, and was recently introduced by Harvey et al. (2012).

$$R_{50,\text{biochar}} = \frac{T_{50,\text{biochar}}}{T_{50,\text{graphite}}} \quad (1)$$

Here, the $T_{50,\text{biochar}}$ and $T_{50,\text{graphite}}$ are two different temperatures at which 50% weight loss was caused by oxidation and volatilization of biochar and graphite, respectively. The water and ash contents were subtracted from the TG thermograms and the temperature was obtained directly. Thus, the last remaining/retained carbon in the solid is known as carbon sequestration potential. It is calculated by subtracting the carbon loss during pyrolysis from the initial C in raw biomass and multiplying by the recalcitrance (R_{50}) of C in the biochar as follows (Choudhary et al. 2019; Shrivastava et al. 2021).

$$\text{Carbon sequestration (\%)} = \frac{M_{(g)} \times \text{Yield (\%)} \times C\%_{\text{Biochar}} \times R_{50}}{M_{(g)} \times C\%_{\text{Feedstock}}} \quad (2)$$

Here,

$M(g)$ = Weight of the total feedstock

Yield (%) = Pyrolyzed biochar amount

$C\%_{\text{Biochar}}$ = Carbon amount of biochar

$C\%_{\text{Feedstock}}$ = Carbon amount of feedstock

SEM and BET analysis

Scanning electron microscopy (SEM) of biochars used a high vacuum condition with accelerating voltage of 20 kV (JSM-5800 LV, JEOL, Japan). Prior to observation each sample was sputter coated with gold. The BET isotherms of biochar samples were also measured (ASAP2460, Micrometric, USA).

Functional groups

The active organic functional groups of biochars were identified via Fourier Transform Infrared Spectroscopy (FTIR). The biochar from pyrolysis or co-pyrolysis at 550 °C was subjected to FTIR (Vertex70, Bruker, Germany). Each spectrum was an average of 32 scans from 400-4000 cm^{-1} at 4 cm^{-1} spectral resolution. The FTIR results were elucidated based on a literary survey.

Hydraulic properties of biochar

In hydraulic properties, the two main ones are 1) water holding capacity (WHC), and 2) water releasing ability (WRA). These were determined for the biochars. The water holding capacity (WHC) was

determined using an in-house developed method. Before the experiment the glass beakers and filter papers were kept oven dried. The biochar samples were also kept in an oven for 10 hours at 105 °C (or until reaching a constant mass). The dry biochar samples were soaked in deionized water for complete saturation in a cylindrical glass beaker, for 24 hours (Song and Guo 2012). After that, the excess water on surfaces of biochar was drained over 30 minutes. During draining, dry filter papers were used to hold the biochar, and then the moist biochar samples were weighed using a digital balance, while the wet filter papers with particles were placed in an oven for drying. When dry the filter papers again weighed and the remaining particle weight from total biochar sample on filter paper was determined. Thus, the water free biochar samples, and the retained water in sample were determined by weight, for a comparison of before and after. The water holding capacity was calculated according the Eq. 3 (Ulusal et al. 2020).

$$WHC\ (mL/g) = \frac{\text{water retained biochar-dry biochar}}{\text{dry biochar}} \tag{3}$$

The water releasing ability (WRA) of biochar sample was determined by using an in-house method at ambient room temperature of 25 °C. After removing surface water on biochar, the moist biochar was kept at room temperature for air drying naturally. The samples were kept for 12 hours after which every hour they were weighed. After sufficient drying time, the biochar samples were weighed. For released and remaining water of biochar, mass of water released (WRAS), and mass of water remaining (WRAM) were estimated using Eqs. 4 and 5:

$$\text{Water releasing ability (WRAS \%)} = \frac{(X_i - X_{it})}{X_{it}} \times 100 \tag{4}$$

$$\text{Water remaining ability (WRAM \%)} = 100 - \text{WRAS} \tag{5}$$

Here,

At t = 0, $X_i = X_{it}$ WRAS₀= 100%

X_i = Mean mass of water before releasing at each period

X_{it} = Mean mass of water after releasing at the final period

t = time

Results And Discussion

Characterization of biochar

Proximate analysis and bulk density

The proximate analysis and bulk density of biochars from RWS, SS, RWS50:SS50, and RWS75:SS25, are shown in Table 1. The reported results include moisture content (MC), volatile matter (VM), fixed carbon (FC), and ash content (AC) in the respective ranges 4.44-4.95, 11.51-14.40, 15.04-78.26 and 5.79-65.61 (wt%). The results directly correlated with feedstock types, whereas pyrolysis or co-pyrolysis temperatures also could have been manipulated. Based on results from this study, it can be seen that the moisture content still present in the biochar was not zero, even when pyrolyzed at a high temperature, which agrees with previous studies by Kabir et al. (2017) and Palamanit et al. (2019). Both of these prior studies reported moisture contents in the range 2.40-4.42 (wt%). However, the moisture content had a certain impact on biochar yield, while a higher moisture in the biomass was favorable for the biochar yield from processing at a higher pressure (Nanda et al. 2016). The volatile matter in biochar was reduced as temperature increased, as this is the comparatively light molecular weight components in liquid or gas form (Palamanit et al. 2019). It can be seen that the volatile matter in biochar still remained in the range 4.32-14.40 (wt%), indicating that the pyrolysis was incomplete while full decomposition would need more time or higher temperatures. When the volatile matter escaped from pores of biochar during the production process, it enhanced soil water movement and soil water retention characteristics (Diatla et al. 2020). Moreover, the high fixed carbon content in biochar indicated that loss of volatile matter from the biochar. According to Palamanit et al. (2019) and Bhattacharjee and Biswas (2018) a high amount of fixed carbon in biochar is favorable for use as a solid fuel, because of high energy content. Biochar produced at a higher temperature has a large amount of fixed carbon (aromatic C-C bonds), and has good stability for use in soil amendments (Al-Wabel et al. 2018). The fixed carbon is helpful in improving stability of biochar during storage. The ash content of sewage sludge biochar (SSB) was higher than that in rubberwood sawdust biochar (RWSB) as well in blends. The ash content is inversely related to fixed carbon: ash content increasing means fixed carbon is decreased (Yang et al. 2020). Nevertheless, the ash contents in both raw biomass and biochar are common and constitute non-volatile and non-combustible components (Sakulkit et al. 2020).

The bulk densities of RWS, SS, RWS50:SS50 and RWS75:SS25 biochars were in the range 181.74-567.32 (kg/m³), as shown in Table 1. It can be seen that the rubberwood sawdust biochar (RWSB) had a lower bulk density than the sewage sludge biochar (SSB) or the blends of RWSB with SSB. This is because SSB had a much higher ash content of metallic elements. Moreover, the bulk density of biochar samples is dependent on biomass feedstock type. Bulk density is important to the potential applications as low density biochars need a large area or volume for storage and transportation (Odetoye et al. 2014; Palamanit et al. 2019). On the other hand, a low bulk density is due to high porosity which affects the potential for soil aeration and enhance interactions with water (Githinji 2014).

Table 1. Proximate analysis, ultimate analysis, bulk density and atomic ratios for the biochar samples

Property	Biochar					
	RWS	SS	RWS50:SS50	RWS75:SS25	PPS500 ^a	OPT ^b
Proximate analysis (wt%, as received basis)						
Moisture content (MC)	4.44 ± 0.03	4.95 ± 0.07	4.71 ± 0.01	4.55 ± 0.03	6.09	3.27
Volatile matter (VM)	11.51 ± 0.01	14.40 ± 0.01	12.95 ± 0.00	12.54 ± 0.08	4.32	14.15
Fixed carbon (FC)	78.26 ± 0.01	15.04 ± 0.02	46.63 ± 0.02	62.42 ± 0.02	85.36	74.26
Ash content (AC)	5.79 ± 0.02	65.61 ± 0.02	35.71 ± 0.01	20.49 ± 0.00	54.77	8.32
Ultimate analysis (wt%, as dry basis)						
Carbon (C)	86.70 ± 0.10	24.27 ± 0.03	55.14 ± 0.00	71.15 ± 0.13	80.79	77.45
Hydrogen (H)	3.32 ± 0.40	0.87 ± 0.01	2.10 ± 0.00	2.73 ± 0.05	3.29	2.38
Nitrogen (N)	0.49 ± 0.00	2.97 ± 0.03	1.73 ± 0.01	1.10 ± 0.00	0.27	0.51
Sulfur (S)	0.04 ± 0.00	0.44 ± 0.01	0.25 ± 0.01	0.10 ± 0.01	N/A	0.06
Oxygen (O)	7.89 ± 0.60	5.13 ± 0.07	6.11 ± 0.05	6.65 ± 0.09	11.13	11
Bulk density (kg/m³)	181.74 ± 0.45	567.32 ± 0.58	317.87 ± 0.41	273.86 ± 0.49	225	N/A
H/C	0.46	0.43	0.45	0.46	0.04	0.43
O/C	0.07	0.16	0.08	0.07	0.14	0.43

*In this study, the oxygen content was determined by difference; PPS500, and OPT mean pigeon pea stalk and oil palm trunk, respectively. ^{a,b}Results from Sahoo et al. (2021), and Sakulkit et al. (2020), respectively. N/A means not available

Ultimate analysis and atomic ratios of biochar

Table 1 presents the elemental compositions and atomic ratios H/C and O/C of the biochars from RWS, SS, RWS50: SS50, and RWS75: SS25. The carbon (C), hydrogen (H), nitrogen (N), sulfur (S) and oxygen (O) contents in the biochars were in the ranges 24.27-86.70, 0.87-3.32, 0.49-2.97, 0.04-0.44 and 5.13-7.89 (wt%), respectively. The ultimate analyses were consistent with the proximate analyses, as indicated by the relation between volatile matter and fixed carbon content, and hydrogen and nitrogen

contents. It can be observed that the carbon contents in sewage sludge biochar (SSB) and its mixes with rubberwood sawdust biochar (RWSB) were below that of RWSB. The thermal decomposition temperatures were affected by biochar structure and composition. The results clearly show that the biochars from pyrolysis have increased carbon and reduced oxygen contents, as found in some recent studies (Sakulkit et al. 2020; Shrivastava et al. 2021; Sahoo et al. 2021b). Several studies have reported that carbon-rich biochar has good potential for soil amendment and stabilizes or immobilizes heavy metals in the soil, and decreases concentrations of hazardous metals in plants, but such performance depends on the types of feedstock and the experimental conditions (Abdelhafez et al. 2014; Rizwan et al. 2019; Wang et al. 2020). Furthermore, the carbon (C) in biochar is the key element in all the functional groups. On the other hand, oxygen (O) and hydrogen (H) contents of biomass feedstock strongly influence biochar properties, including association/disassociation and polarity of hydrogen ions, which can strongly impact the biochar interactions with organic and inorganic solutes (Hassan et al. 2020).

The H/C and O/C atomic ratios in RWS, SS, RWS50:SS50 and RWS75:SS25 biochars are shown in Table 1. The H/C and O/C ratios in biochar samples were in the ranges 0.43-0.46 and 0.07-0.16, respectively. On increasing the temperature of carbonization, the H/C and O/C of atomic ratios tend to decrease, and Table 1 indicates they were stable in this study. These results are similar to the recent studies by Wani et al. (2020) and Sakulkit et al. (2020). According to Usevičiūtė and Baltrėnaitė-Gedienė (2020), on increasing the temperature from 300 to 500 °C in pyrolysis of biomass (grass and wood) both H/C and O/C atomic ratios decreased. Normally, demethylation (CH_3) is evaluated from the H/C ratio, while decarboxylation (CO_2) is assessed from the O/C ratio, and the eventual solid biochar is enriched in carbon content (Wiedner et al. 2013). The main purpose of evaluating the H/C and O/C atomic ratios is for use as proxy indicators of biochar stability, and these correlate negatively with aromatic carbon (C). However, biochar produced at a comparatively low temperature with high O/C ratio can have low carbonization and lacks stability against degradation in the soil surface, so it displays comparatively high reactivity (Usevičiūtė and Baltrėnaitė-Gedienė 2020; Ulusal et al. 2020). According to EBC suggestions, biochar H/C ratio must be below 0.6 while O/C must be below 0.4 in carbon black substances. So, in the present study the H/C and O/C atomic ratios satisfy these criteria and the biochars are suitable for soil application.

X-ray fluorescence (XRF)

Table 2 presents the major and minor inorganic elements in biochars from RWS, SS, RWS50:SS50 and RWS75:SS25 as identified by X-ray fluorescence (XRF) spectrometry. The inorganic elements Si, Ca, Fe, K, Mg, Na, P, Cu, Zn, Mn and Pb, were found in biochars in the ranges 6731-102415.3, 116085.7-241156.7-, 1308-104031.3, 23540-49341.33, 7283.33-14071.67, 291-7818, 3120-183740, 110-1120, 130-11660, 750-6170 and 32.98-131 (mg/kg), respectively. The present results indicate that the concentration of some pollutant contaminants was elevated in the biochars. It can be seen that the concentrations of Si, Al and Ca were higher in sewage sludge biochar (SSB) than in rubberwood sawdust biochar (RWSB) or in the mixes of SSB and RWSB. RWSB had less Na than the other biochars. The Pb element was detected only in SSB or the blends while RWSB did not have it detectably. When a large fraction of SS was co-pyrolysed

with RWS the inorganics in biochar decreased. Furthermore, the major and minor inorganic elements identified in biomass and in pyrolyzed biochar came from the plant biomass: they are present in the plants naturally (Saleem et al. 2020). The results in this current study are similar to prior studies by Deng et al. (2017) and Kończak et al. (2020). According to Deng et al. (2017) the K, Ca, Mg, Na, Si, Fe and Al are the dominant elements in sewage sludge waste char from pyrolysis or co-pyrolysis. According to some previous studies, the inorganic elements often were left in biochar of sewage sludge after pyrolysis because they did not decompose or become volatile at the pyrolysis temperatures in 400-600 °C (Jin et al. 2017; Huang et al. 2017; Wei et al. 2019). The application of biochar for soil improvement can increase crop productivity by reducing toxic elements via electrostatic reactions (Khalid et al. 2020). Remarkably, biochar has many major and minor inorganic elements that are essential nutrients for crop growth and soil health, for example Ca, K, P, and Mg were seen in SSB and RWSB in this study, but some elements are not acceptable for soil application like Cd and Pb. Therefore, the biochar from RWS blended with SS appears very appropriate for soil remediation and might positively influence agricultural crops, because of the balance in inorganic element concentrations. When the biochar is applied to soil, the alkaline substances released from it counteract acidification (Shaaban et al. 2018).

Table 2. Major elements in biochar samples

Element (mg/kg)	Biochar					
	RWS	SS	RWS50:SS50	RWS75:SS25	SC550-30 ^a	CMB600 ^b
Silicon (Si)	6731	102415.3	54572.82	7552.41	14100	30000
Calcium (Ca)	241156.7	116085.7	178621.2	209889.3	140700	62000
Iron (Fe)	1308	104031.3	52669.1	26988.5	287500	N/A
Potassium (K)	23540	49341.33	36441	29982.4	2500	380000
Magnesium (Mg)	7283.33	14071.67	10677.54	8980.76	2700	9000
Sodium (Na)	291	7818	4055.57	2172.11	9500	81000
Phosphorus (P)	3120	183740	122720	71640	73600	53000
Copper (Cu)	110	1120	830	560	N/A	N/A
Zinc (Zn)	130	11660	7780	4530	N/A	N/A
Manganese (Mn)	750	6170	4400	2880	1400	N/A
Lead (Pb)	ND	131	65.57	32.98	N/A	N/A

*In this study, ND and N/A mean not detected and not available, respectively; SC550-30 (SC550-30=TDS: RWW), and CMB600 mean textile dyeing sludge: red wood waste and chicken manure biochar. ^{a,b}Results from Zhou et al. (2021), and (Huang et al. 2021), respectively.

Thermogravimetric analysis

Figs. 1(a-b) show the thermogravimetric analysis curves with peaks, with TGA in (wt%) and DTG also in (wt%), for rubberwood sawdust biochar (RWSB) and sewage sludge biochar (SSB). TGA and DTG were performed to evaluate pyrolytic behavior and biochar resistance to thermal degradation (Reza et al. 2020). As shown in Figs. 1(a-b), three stages were observed in biochar degradation, with weight loss in the first stage over 50-200 °C, in the second stage 200-600 °C, and the third stage 600-1000 °C. The weight loss in first stage was due to elimination of water or moisture. In the second stage, the weight loss was due to degradation of cellulose and hemicellulose. Finally, in the third stage there was degradation of lignin (Naik et al. 2010). The degradation rates of RWSB and SSB were similar to those in Kim et al. (2012) and Reza et al. (2020). Remarkably, the weight loss at higher temperatures is from degradation of inorganic compounds as well as dehydrogenation and aromatization of biochar (Oja et al. 2006). A comparatively low loss of weight is good for a stable biochar with strong carbon-carbon and hydrogen-carbon bonds remaining (Mohanty et al. 2013). Accordingly the biochars from RWS and SS biomasses pyrolyzed at 550 °C were thermally more stable than those from 400 °C or 450 °C, and would be highly efficient and effective for soil remediation applications (Lehmann et al. 2011). In DTG, the highest peak for RWSB was noted at approximately 700 °C, while SSB at this temperature was mostly stable. So, the TGA and DTG graphs clearly showed the mass losses from biochar samples of both RWSB and SSB, with minimum percentages of about 10-20% when the reactor temperatures were increased from 400 to 550 °C, which improved thermal stability of the products. In addition, sometimes the pyrolysis parameters can be less influential as regards the biochar properties (Kim et al. 2012).

pH analysis

Fig. 2 shows the pH values of the RWS, SS, RWS50:SS50 and RWS75:SS25 biochars at 10.02, 8.41, 8.69 and 9.04, from pyrolysis at 550 °C, respectively. It can be seen that the rubberwood sawdust biochar (RWSB) had much higher pH (was more strongly alkaline) than the sewage sludge biochar (SSB) or their blends. The lower pH of SSB might be from acidity of organic oily components. Normally, pH increases with pyrolysis temperature of biochar (Yuan et al. 2011). Moreover, biochar pH increases because of elimination of organic materials while the alkaline salts are retained in biochar (Al-Wabel et al. 2013). However, the biochar surface functional groups such as phenolic, carboxylic, and lactonic groups could be associated with the biochar sorption capacity according to prior studies (Jegajeevagan et al. 2016; Ulusal et al. 2020). pH levels of RWSB and SSB in this study are comparable or similar to previous studies by Angin (2013), Ferreira et al. (2019) and Reza et al. (2020). On the other hand, Han et al. (2021) reported that the low pH of biochar can be relatively influence, because of the hydrogen H⁺ ions engaged the biochar adsorption pour sites. Biochar with higher pH has more potential for use as soil conditioner that reduces heavy metal contaminants. In addition, biochar with suitable pH can enhance the aeration,

moisture and redox potential of weaker soil surface by reducing the pollutants (Chen et al. 2019b). Duku et al. (2011) described that biochar with higher pH is more advantageous for soil health and can limit the utilization of lime in soils, and it also can develop the loamy and sandy properties in soils effectively against clay soils, which was applied in Pennsylvania, Ghana and Mexico. Therefore, the biochar from pyrolysis at an intermediate temperature has much potential for soil use as organic carbon, with good water capacity and exchangeable cation content, and it may decrease the ductile strength of soil surface (Chan et al. 2007).

Recalcitrance and stability

Table 3 shows the carbon sequestration (CS) and recalcitrance of rubberwood sawdust biochar (RWSB), sewage sludge biochar (SSB) and their blends. The calculated R_{50} and CS for biochar samples were in the ranges 0.28-0.54 and 11.03-22.73 (%), respectively. The index (R_{50}) for carbonized biochar is based on a model proposed by Harvey et al. (2012). For soil maintenance functionality and stability, as relates to atmospheric carbon sequestration, biochar materials need to resist biotic or abiotic degradation. The R_{50} index is a measure of the energy required for thermal oxidation at 50% weight loss of biochar. The temperatures determined for R_{50} were obtained from thermograms corrected for ash and water contents, and were found in the range 450-492 °C. It can be seen that our results are similar to Shrivastava et al. (2021). The pyrolysis temperature can influence the biochar recalcitrance which is connected to aromatic C, and with increasing pyrolysis temperature causing loss of N, aromatic C can increase (Spokas et al. 2009). However, the reacting temperatures has a dominating capability to control of the recalcitrance of the biochar nature. Moreover, for carbon sequestration (CS) the R_{50} index is combined with carbon contents from ultimate analysis and proximate analysis. A low pyrolysis temperature tend to give biochar with high C yield, but with small pores, and not resistant to abiotic or microbial mineralization. A higher pyrolysis temperature provides less biochar but it is more stable and recalcitrant (Cheng et al. 2006; Worasuwannarak et al. 2007; Zimmerman 2010). The biochar carbon sequestration capability for $R_{50} > 0.7$ is comparable to soot or graphite (Harvey et al. 2012).

Table 3. Recalcitrant nature of biochar

Type of Biomass	Pyrolysis Temperature °C	Carbon Sequestration (%)			
		Carbon (wt%)	Fixed Carbon (wt%)	R_{50}	CS (%)
RWS	550	86.7	78.26	0.54	22.73
SS	550	24.27	15.04	0.28	11.03
RWS50:SS50	550	55.14	46.63	0.39	16.91
RWS75:SS25	550	71.15	62.42	0.48	19.89

Surface morphology and surface area

Fig. 3(a)-(d) present the SEM micrographs of RWS, SS, RWS50:SS50 and RWS75:SS25 biochars obtained from pyrolysis at 550 °C. The SEM images of biochar samples from rubberwood sawdust biochar (RWSB) and sewage sludge biochar (SSB) and their blends show different surface features. In Fig. 3(a) and 3(b) RWSB and SSB were pyrolyzed singly, while Fig. 3(c) and (d) shows blends. It can be observed that RWSB was rougher than SSB at SEM magnification 500X. The RWSB shows some cylindrical as well as honeycomb shapes. From woody biomass after pyrolysis at a higher temperature the biochar will appear more porous because of thermal degradation of lignocellulosic components. The RWSB surface features in this study are comparable to those in Palamanit et al. (2019) and Sakulkit et al. (2020). As shown in Fig. 3(b), the SSB pores and features are not really clear because of the small particles and sandy character. Hence, the sewage sludge biomass after pyrolysis did not have structural pores, as shown in Fig. 3(b). However, the co-pyrolysis of RWS biomass with SS biomass at various blend ratios gave more porosity than SSB, as seen in Fig. 3(c) and 3(d). Several factors affect the biochar produced, including residence time, temperature, and pyrolysis reactor type etc. (Ahmad et al. 2012). So, the decomposition of sewage sludge (SS) biomass at a higher temperature gave undeveloped pore structure compared to RWSB, because of the lower content of carbon in sewage sludge (Yin et al. 2019). Biochar with high porosity can be applied as adsorbent of heavy metals from soil, because the active sites on biochar can attract and reduce the toxic elements (Wu et al. 2016; Karimi et al. 2020).

The surface area and average pore diameter of rubberwood sawdust biochar (RWSB) and sewage sludge biochar (SSB) were determined by BET, when each biomass feedstock was pyrolyzed at higher temperatures, as shown in Fig. 4. The surface area and average pore diameter of biochar samples were in the ranges 2.15-18.42 (m²/g) and 162.39-217.16 (nm), respectively. The pore volume and pore diameter of SSB were lower than for RWSB or the blends. The specific surface area of RWSB was higher than for SSB, as seen in Fig.4. The surface area and pore volume of RWSB of this study were most similar to those in Sakulkit et al. (2020) and Shrivastava et al. (2021). Regarding SSB, prior studies include Agrafioti et al. (2013) and Deng et al. (2017). Deng et al. (2017) reported that the surface area of sewage sludge biochar was 27.9 (m²/g), while Agrafioti et al. (2013) found that it was 18 (m²/g). However, co-pyrolysis of RWS mixed with SS biomass blended ratios from higher side biochar was strongly influence the lower ratio side of biochar which was effect on the pore volume and surface area also because RWS was more lignocellulosic biomass. Instead of this, the application of higher porous biochar with rich carbon content ($S_{BET} \geq 100 \text{ m}^2/\text{g}$) could be participated to developed the soil porosity by (2 to 40%), to reduce the soil higher bulk density by (3 to 30%) investigated by Fidel et al. (2017) and Mukherjee et al. (2019).

FTIR analysis results

Fig. 5 shows the FTIR results for rubberwood sawdust (RWSB), sewage sludge (SSB) and their blends from pyrolysis at 550 °C. In Fig. 5, several components were present in the samples. Because of this, the biochar samples still contained compounds with C, H and O, despite strong degradation of the lignin component. The FTIR results match ultimate analysis and proximate analysis which were

discussed in earlier sections. In addition, the biochar spectra did not much differ from each other, because of similar elemental compositions in terms of C, H and O contents. The peaks can be described as follows. The first peak was around $3404\text{--}3410\text{ cm}^{-1}$, and was attributed to stretching of OH group and is also associated with biomass dehydration (Camargo and Sene 2014). The second small peak appeared at $2851\text{--}2873\text{ cm}^{-1}$ from C-H stretching vibrations in aliphatic and aromatics structures. The peak at 1605 cm^{-1} was for carbonyl and carboxyl groups in carbohydrates (Elnour et al. 2019), whereas the aromatic ring stretching vibrations of $\text{C}=\text{C}$ were identified at $1559\text{--}1566\text{ cm}^{-1}$ (Bhattacharjee and Biswas 2018). For C-H and CH_2 , the peak at $1428\text{--}1439\text{ cm}^{-1}$ represented stretching vibrations in aliphatics and biochar. The band at $1088\text{--}1120\text{ cm}^{-1}$ was assigned to phenolic OH and aromatics C-O bonds. The weak vibrations of C-H bonds in heteroaromatics and aromatic compounds are visible at $603\text{--}876\text{ cm}^{-1}$ (Elnour et al. 2019; Bavariani et al. 2019).

Hydraulic properties of biochar

Figs. 6 and 7 shows the water holding capacity (WHC) and water releasing ability (WRA) of rubberwood sawdust biochar (RWSB), sewage sludge biochar (SSB) and their blends, produced at 550°C . The water holding capacity (WHC) measurements were modified from (Ulusal et al. 2020), and the present range was $1.01\text{--}3.08\text{ (mL/g)}$. It can be observed that the RWSB had a higher WHC than SSB or the blends, as shown in Fig. 6. The results in this study on biochar were comparable with those in Zhang and You (2013) and Reza et al. (2020). The WHC is significant indicator of the ability of biochar to hold moisturize by cohesion and adhesion forces (Song and Guo 2012). On the other hand, the surface area of biochar not only improves the WHC, but access to the functional groups, porous structure, and oxygen content are also increased (Zhang and You 2013b). The biochar with improved WHC is most effective as adsorbent with micropores that can be saturated with water (Gray et al. 2014). The biochar produced at a lower temperatures has no more porosity than biochar from a higher temperature, and the water may not have access to the pores because of small pore volume, tar blocking the pores, and poor connectivity of the pores (Das and Sarmah 2015).

The water releasing ability (WRA) of rubberwood sawdust biochar (RWSB), sewage sludge biochar (SSB), and blended biochars (50:50 and 75:25) are shown in Fig. 7. The water releasing ability (WRA) and remaining water results were in the ranges $1.19\text{--}52.42\text{ (wt\%)}$ and $47.58\text{--}80.34\text{ (wt\%)}$, respectively. In the graphs the symbol (-Y1) represents the remaining water and (-Y2) represents the released water from biochar. The present experiment allowed 12 hours for the release of water. Comparatively, the released water (%) of RWSB was lower than that of SSB or the blends, because of its lower bulk density and larger micropore volume contributed to water retention (Shaaban et al. 2013). On the other hand, the remaining water (%) is opposite to the release of water (%) by biochars, and the RWSB retained more water (%) than SSB or the blends. Biochar with a large pore volume and specific surface area is suitable for soil surface application to retain moisture over a prolonged period and to decrease the leaching of soil (Lehmann et al. 2006). Among these tested biochars, the RWSB was found to have high potential for filtration as well

as especially for soil amendment, because it has a large pore volume and specific surface areas for interactions with water.

Conclusion

In summary, the innovative carbonized biochars from rubberwood sawdust (RWS) and sewage sludge (SS) and their blends (50:50 and 75:25) were characterized for properties related to soil remediation as well as mitigation of the greenhouse gases. The results showed that the pyrolysis temperature had a large influence on biochar features and structures. The carbon content (86.70 wt%) was higher and oxygen content (7.89 wt%) was lower in rubberwood sawdust biochar (RWSB), whereas the sewage sludge biochar (SSB) had more ash content (65.61 wt%) and less carbon content in ultimate and proximate analysis results. In thermal degradation RWSB had slightly higher mass loss than SSB because of lignocellulosic components, particularly cellulose and hemicellulose. The elements Si, Fe, K, Na and P were higher in SSB than in RWSB, which was confirmed by XRF. The pH of RWSB and SSB was alkaline in the range 8.41–10.02. The carbon quantity in biochar for capturing of CO₂ by carbon sequestrations (CS) method was assessed. The biochar with high porosity and specific surface area, good for soil application, was identified by SEM and BET. Moreover, the obtained functional groups were similar across biochars, assessed by FTIR. The water holding capacity (WHC) and water releasing ability (WRA) were determined for the biochars, complementing pore volume and specific surface, to further assess potential for adsorbent use in soil or water bodies. The biochars were pyrolyzed at the relatively high 550°C temperature, elevating hydrophobicity, increasing pore volume and specific surface area, and improving adsorption capability for pollutant removal. In conclusion, the biochar produced from rubberwood sawdust (RWS) had more potential than sewage sludge (SS) or the blends (50:50 and 75:25) as biochars, because it had a higher pore volume and lower bulk density. It could be applied as anti-pollutant adsorbent in soil amendment or water treatment. Regarding SSB and the blends these could be applied as soil nutrients to replace chemical fertilizers. In the future, when exploring the concept of waste valorizing by producing biochar, these could serve to mitigate environmental pollution problems.

Declarations

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Figures

Figure 1

TGA (a), and DTG (b) of RWS, SS, RWS50:SS50 and RWS75:SS25 biochars from pyrolysis at 550 °C.

Figure 2

pH levels of RWS, SS, RWS50:SS50 and RWS75:SS25 biochars from pyrolysis at 550 °C

Figure 3

SEM photographs of (a) RWS biochar, (b) SS biochar, (c) RWS50:SS50 biochar, and (d) RWS75:SS25 biochar, (all biochars from 550 °C, the right side figures c-d show RWS biochar, while the left side shows SS biochar), and red circles show the pores of particles

Figure 4

Specific surface area and pore diameter of biochar samples pyrolyzed at 550 °C

Figure 5

FTIR spectra of biochars from pyrolysis at 550 °C.

Figure 6

Water holding capacity of biochar samples pyrolyzed at 550 °C

Figure 7

Water retention refers to Y1 axis and water releasing to Y2 axis, for the biochar samples pyrolyzed at 550 °C