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Characterization of biomass residues and their amendment effects on water sorption and nutrient leaching in sandy soil

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HIGHLIGHTS

- Two distinctive biorefinery residues were effective as soil amendments.
- Biorefinery residues could improve water holding capacity of sandy soil.
- Biorefinery residues could improve ammonium and phosphate leaching of sandy soil.
- There is correlation between residue SSA and amendment efficiency.

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ABSTRACT

In this study, we evaluated the efficiency of two types of biomass residues (fermentation residues from a bioethanol process, FB; brown mill residues from a papermaking process, BM) as amendments for a sandy soil. The characteristics of these residues including specific surface areas, morphologies and nutrient sorption capacity were measured. The effects of biorefinery residues on water and nutrient retention were investigated in terms of different particle sizes and loadings. The results indicated that bio-based wastes FB and BM were able to significantly improve water and nutrient retention of sandy soil. The residues with larger surface areas had better water and nutrient retention capability. Specifically, in the addition of 10% loading, FB and BM was able to improve water retention by approximately 150% and 300%, while reduce 99% of ammonium and phosphate concentration in the leachate compare to the soil control, respectively.

1. Introduction

The use of low-cost crop residues (e.g. sugarcane bagasse residue after sugar extraction) to produce second-generation biofuel (e.g. ethanol) is a promising sustainable approach to partially offset current petroleum-dominant energy market (Farrell et al., 2006; Balat et al., 2008; Geddes et al., 2011). As a result, a large number of biorefinery residues will be produced in newly-built biorefinery processes. Meanwhile, large quantity of bio-based wastes from current biomass related processes (e.g. pulp and paper processes) also need better utilization instead of burning. One of the promising approaches to use bio-based waste is as a low-cost soil amendment. As reported in previous literature, the addition of organic matters improved water holding capacity of a soil (Johnson et al., 2007). In addition, the removal of bio-based waste from agricultural fields or forests to biomass processing locations instead of leaving in the crop field had adverse impact on soil quality and productivity, and led to accelerating evaporation, water and nutrient losses (Lindstrom, 1986; Blanco-Canqui and Lal, 2009). It is essential to return part of biomass residues back to soil as approximately 41% of biomass should be kept in all major land use areas in order to prevent soil erosion according to previous studies (Holt, 1983; Lindstrom, 1986).

Productivity of sandy soils, characterized by low water and nutrient holding capacity, is limited by the lack of available water as well as nutrients that are required by plant growth (Andry et al., 2009). Meanwhile, in arid area, it is imperative to use water efficiently because of water shortage. Synthetic hydrophilic polymers have been investigated as soil amendment materials to retain water and nutrient in arid area. It has been reported that hydrophilic polymers such as polyacrylic acid and polyacrylamide gels

Abbreviations: FB, fermentation residues from a bioethanol process; BM, brown mill from a papermaking process; WRV, water retention value.

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were able to retain water up to 500 times of their weight (Holliman et al., 2005). These synthetic polymers could improve water retention in sandy soils (Aldarby et al., 1992), therefore facilitated the growth of plants (Flannery and Busscher, 1982; Johnson, 1984). However, despite the superior water retention capacity, their wide applications as soil amendments have been limited by a variety of factors such as non-renewable, non-biodegradable, low salt tolerance, possibility of releasing toxic residues and high cost (Holliman et al., 2005; Andry et al., 2009). Bio-based materials such as manures, starch, pristine plant fiber, cellulose (including carboxymethylcellulose, CMC), and chitosan have been studied as soil amendments as well. The drawbacks for their use include low retention, high price, low salt tolerance and their competitiveness with food (Mamilov et al., 2001; He et al., 2002; Andry et al., 2009; Radwan et al., 2012).

The use of biomass residues (biorefinery and pulping residues) as soil amendments not only adds an avenue to biomass processing industries but also offsets the negative impacts of biomass residues' removal on soil properties. Johnson et al. reported that corn stove fermentation residues were capable to improve properties of severely-eroded soil such as water stable aggregates and decreased bulk density without adverse impacts on crop growth (Johnson et al., 2007). Galvez et al. claimed that bioethanol by-products led to N_2O emissions and larger increases in soil respiration, N availability, and enzymatic activity in comparison with other amendments such as sewage sludge and composts (Galvez et al., 2012). (Gell et al., 2011) reported that no crop phytotoxicity was significant after seven-day application of bioethanol residues.

The impact of biorefinery residues and their characteristic difference on water and nutrient retention capacity of sandy soil remains unclear. Therefore, in this study, we aim to evaluate the efficiency of biomass residues as sandy soil amendments according to their characteristics, particle size ranges, and loading levels. These two distinctive biomass residues (fermentation residues (FB) from a cellulosic bioethanol process using sugarcane bagasse as raw materials; brown mill residues (BM) from waste stream of a papermaking process) are different in compositions (lignin-dominated or cellulose-dominated), particle sizes, and specific surface areas. The understanding for the correlation between their characteristics and water and nutrient retention capacity of sandy soil is very beneficial for the large-scale use of bio-wastes as soil amendments in the field in the future.

2. Material and methods

2.1. Materials

Fermentation sugarcane bagasse residues (FB) were collected from waste stream of a bioethanol pilot plant. The collected residues were placed in a sieve (US standard test sieve #270) and washed with warm tap water until effluent became clear and then dried in an oven at 70 °C for 24 h. Brown mill residues (BM) were collected from waste stream after screening in a papermaking process using slash pine as the raw material (Buckeye Technologies, Perry, FL, USA). BM residues were dried at 70 °C for 24 h and then milled in a laboratory mill (Model 4, Thomas Willey, Swedesboro, NJ, USA) equipped with a screen with a mesh opening size of 2 mm. Both FB and BM were separated into three size ranges (A: 0.297–0.5 mm, B: 0.178–0.297 mm, C: 0.089–0.178 mm) by using a set of ASTM standard test sieves (#35, 50, 80 and 170) and a Octagon 200 test sieve shaker at an amplitude of 8 for 15 min. The soil was collected from Hastings, Florida. It is classified as Ellzey fine sand series (sandy, siliceous, hyperthermic, Arenic Endoaqualf) (Baillie, 2001). Boric acid, sodium hydroxide, sodium tetraborate, sulfuric acid, phenol, ethylenediamine tetraacetic acid disodium salt dehydrate, sodium nitroprusside, ammonium chloride, antimony potassium tartrate, ammonium molybdate, ascorbic acid, ammonium persulfate, potassium phosphate monobasic were purchased from fisher scientific (USA) and used as received. Sodium hypochlorite solution (Clorox House bleach) was purchased in local grocery store (The Clorox Company, Oakland, California, USA). The nutrient solution at a concentration of 3000 ppm was prepared by dissolving anhydrous ammonium chloride and potassium phosphate monobasic in autoclaved deionized water and stored at 4 °C to prevent microbial growth.

2.2. Soil and leachate samples preparation

Soil (150 g as dry weight equivalent) and FB or BM (1%, 3%, 5%, and 10% dry weight of soil) were manually mixed in a beaker before loading to soil columns. Basically, nutrient solution was transferred to columns loaded with soil and FB/BM mixtures and the leachates were collected (for detailed description of soil column and the leachate collection procedure, please refer to Supplementary information). All leachate samples were then stored at 4 °C in the refrigerator and the pH is kept less than 2 by adding sulfuric acid (H_2SO_4) before analysis. The soil–FB or BM residue mixtures were temporarily stored in zip-bags and used for water retention value (WRV) analysis.

2.3. Biomass residues characterization

2.3.1. Composition analysis

Compositions of biomass residues (FB and BM) were analyzed according to the National Renewable Energy Laboratory (NREL) method (Sluiter et al., 2008). Monomer sugar contents after cellulose hydrolyzation were measured by High Pressure Liquid Chromatography (Agilent Technologies HPLC 1200 series, Santa Clara, CA, USA) equipped with BioRad Aminex HPX-87H column (Hercules, CA, USA). The acid soluble lignin was determined by UV–Vis spectroscopy (Beckman DU800 UV/Vis Spectrophotometer, Brea, CA, USA). Both acid insoluble lignin and ash content were obtained by gravimetric analysis.

2.3.2. Specific surface area measurement

Specific surface areas (SSAs) of FB and BM at different particle sizes were determined by N_2 sorption isotherms on a NOVA 1200 series volumetric gas adsorption instrument (Quantachrome, FL, USA). The sample was loaded in a specific glass cell and the cell was submerged in liquid nitrogen. The densities of all the samples were pre-measured by a multipycnometer (Quantachrome, FL, USA) and used as a parameter for further SSA analysis. The specific surface area was calculated by multipoint nitrogen adsorption in a relative pressure range of 0.05–0.2 P/P_0 in accordance to BET method developed by Brunauer et al. (1938).

2.3.3. Scanning electron microscopy (SEM) for morphology analysis

Scanning electron microscopy (SEM) with a field emission gun (FEI XL-40 FEG-SEM, operating voltage of 30 kV, FEI, Hillsboro, Oregon, USA) was used to examine the morphologies of both biomass residues FB and BM. All samples were coated with platinum before scanning.

2.3.4. Fourier transform infrared spectroscopy (FTIR)

Fourier transform infrared spectroscopy (FTIR) was used to investigate the sorption of nutrient ions on biomass residues (FB and BM). FTIR spectra were recorded on Spectrum BX spectrometer from PerkinElmer (Massachusetts, USA) (for detailed sample preparation and testing parameters, please see Supplementary information).

2.4. Water retention value (WRV)

Relative water retention values (WRVs) for both soil alone as control and soil-residue mixture at different sizes (size groups A, B, and C) and loading levels of FB and BM (1%, 3%, 5%, and 10%) were measured according to a modified TAPPI Method UM256 "water retention value" (TAPPI, 1991). In each column (as described in Section 2.2. soil and leachate samples preparation), approximately 15 g of soil or soil mixture was collected and then loaded into a stainless steel centrifuge holder equipped with a pre-weighed membrane filter (pore size 0.45 μm) and a fine metal mesh located on its perforated bottom. The holder was located inside a 50 mL centrifuge tube (detailed schematic in Supplementary information, Fig. S1). Each sample was subjected to centrifugation at 900 G's for 30 min at room temperature, where the operating speed (2600 RPM) was calculated according to Eq. (1). After centrifugation, each sample with membrane filter together was transferred to a pre-weighed aluminum tray and the gross weight of wet sample with tray was recorded immediately. After that, it was dried at 50 °C for 24 h until constant weight was reached. The gross weight of dry sample with tray was recorded as well. The WRV of each sample was calculated according to Eq. (2).

Equation 1. Relative centrifuge force G's (ratio of acceleration on sample to acceleration due to gravity).

$$G's = \frac{4\pi^2 W^2 r}{3600 g_0} \quad (1)$$

where W is the revolutions per minute, r is radius of centrifuge, g_0 is the acceleration due to gravity (9.8 m s^{-2}).

Equation 2. Water retention value (WRV) (ratio of total water content to dry weight of sample).

$$WRV = \frac{(W_1 - W_3) - (W_2 - W_3)}{W_2 - W_3} \quad (2)$$

where W_1 is the gross weight of wet sample, tray and membrane filter after centrifugation, W_2 is the gross weight of wet sample, tray and membrane filter after drying, W_3 is the weight of tray and membrane filter.

2.5. Nutrient solution concentration analysis

Ammonium (NH_3) concentration in leachates was determined by the AQ-2 Discrete Automated Analyzer (AQ2, Seal Analytical, Mequon, WI, USA) according to USEPA colorimetric method 350.1 (O'Dell, 1993a). Phosphorus concentration was determined by the AQ-2 Discrete Automated Analyzer (AQ2, Seal Analytical, Mequon, WI, USA) according to EPA method 365.1 (O'Dell, 1993b).

2.6. Statistical analysis

All experiments were carried out in triplicate. Analysis of variance (ANOVA) was used to analyze the results of water retention values and nutrient solution concentrations. General linear model with a pairwise comparison, Tukey test under a 95% confidence interval ($\alpha = 0.05$) in Minitab 16 software package was used. Different letters shown in Tukey method column in both Table 2 represent significant difference in either WRV or nutrient retention ($\alpha = 0.005$).

3. Result and discussion

3.1. Fiber compositions

Lignocellulose is generally composed of three main components 15–30% of lignin, 30–45% of cellulose and 10–15% of hemicellulose, which are combined together to form a firm and compact network structure (Ververis et al., 2004). The compositions of biomass residues FB and BM are summarized in Table 1. FB had much higher percentage of lignin (50%) than original lignocellulose because most of carbohydrates were converted to ethanol through hydrolysis and fermentation processes while lignin could not be utilized through biorefinery process and left as residues (Geddes et al., 2011). BM was exclusively made of cellulose (80%) while lignin occupied as low as 8.5% in the residue. This indicated that most of lignin was removed through papermaking process (Hubbe et al., 2007).

3.2. Specific surface areas (SSAs)

The specific surface areas (SSAs) of FB and BM are illustrated in Fig. 1. We observed that particle size ranges (A: 0.297–0.5 mm; B: 0.178–0.297 mm; C: 0.089–0.178 mm) had no significant effect on SSAs for both FB and BM. This indicated that bulk sizes might not affect specific surface areas which were determined by both surface area and density. In addition, the residues had a large aspect ratio, which resulted in a larger deviation for the use of screen size as classification standard. Previous research also claimed that different types of fibers passed through the same test sieve might have distinctive SSAs (Driemeier et al., 2011). As shown in Fig. 1, the SSA of BM (76 m^2 g^{-1} of the average SSA) was more than twice that of FB (32 m^2 g^{-1} of the average SSA). The larger SSA of BM might be derived from cellulose fibers, which occupied as high as 80% in this residue. FB with a greater percentage of low-aspect-ratio lignin resulted in a reduced SSA, which agreed well with other researchers (Spence et al., 2010). In fiber-dominant BM residues, cellulose crystallinity was greatly reduced and a large number of fibrils with larger SSA were produced during the process to remove lignin (da Silva et al., 2011).

3.3. Morphologies of biomass residues

The morphologies of FB and BM were examined by the scanning electron microscope (SEM). As shown in Fig. 2(a) and (c), in general, BM had smaller size and larger aspect ratio than those of FB, which agreed well with the result of specific surface areas. FB was made of irregular lignin bundles with a diameter larger than 50 μm and small openings were observed at the end of these bundles (Fig. 2(a and b)), which might facilitate water and nutrient transfer. BM was composed of uniform and platy fiber bundles

Table 1
Compositions of BM and FB residues.

	Lignin%	Cellulose%	Hemicellulose%	Ash%
BM	8.5 ± 2.9	80.4 ± 2.4	10.4 ± 0.6	0.8 ± 0.0
FB	49.8 ± 1.7	40.1 ± 0.4	9.5 ± 1.5	0.7 ± 0.2

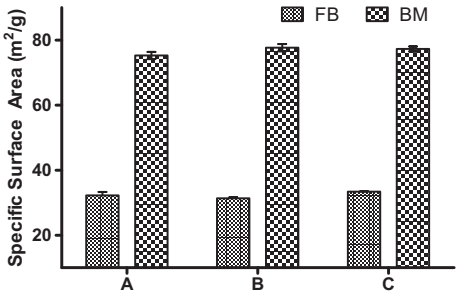

Fig. 1. BET specific surface area (SSA) analysis of FB and BM, three size classes: A: 0.297–0.5 mm B: 0.178–0.297 mm C: 0.089–0.178 mm.

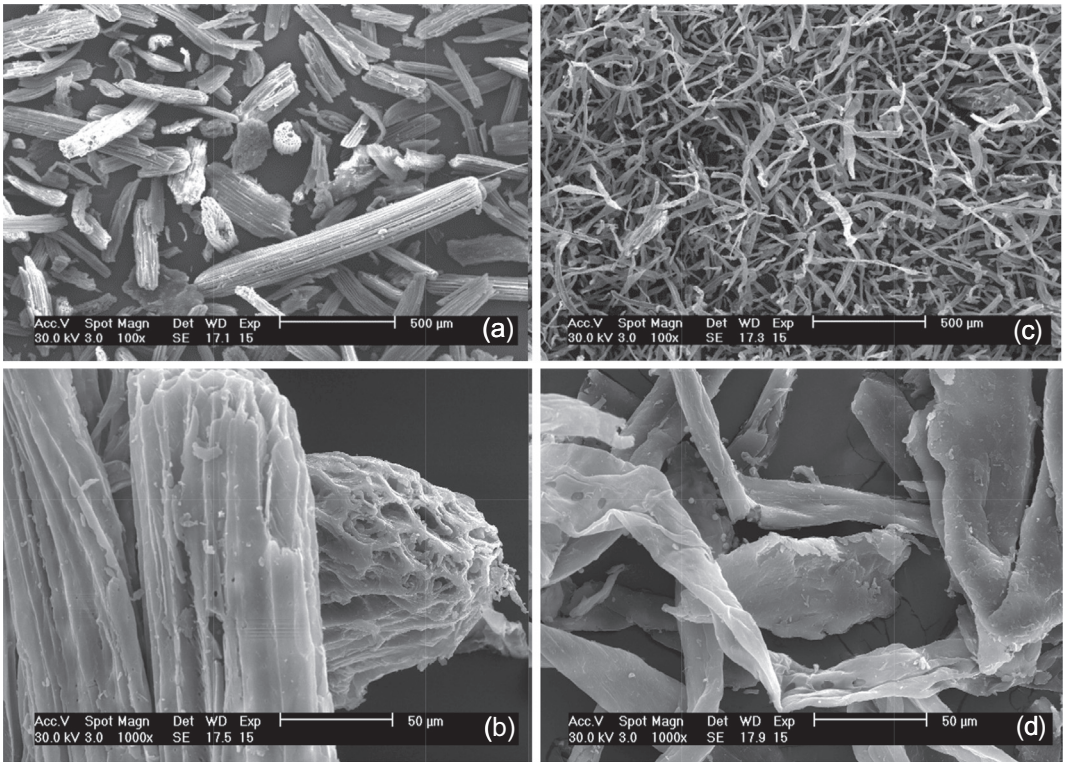

Fig. 2. Scanning Electron Microscopy (SEM) images of biomass residue, FB and BM (a and b, FB with 100X and 1000X magnification respectively; c and d, BM with 100X and 1000X magnification respectively).

with a width of several micrometers but a large aspect ratio (Fig. 2(c and d)).

3.4. Nutrient ions sorption

In order to study the sorption of nutrient ions (ammonium and phosphate) onto FB and BM residues, FTIR was used to characterize untreated and nutrient-solution-treated FB and BM samples. Many changes in peaks and vibrations were presented, which can prove the sorption of the nutrient ions onto the FB and BM. Detailed discussion and the FTIR spectra can be found in the supplementary information (Supplementary information: Result and Discussion 3.4. and Fig. S2).

3.5. Water retention values (WRVs)

The addition of merely 1% FB and BM improved WRV by approximately 10% and 35% in comparison with the control soil, respectively (Fig. 3(a)). Statistical analysis of WRV of FB (see Supplementary information Table S1) showed that there was no significant difference of WRV between the control and all three fiber size groups in the addition of 1% of FB. Addition of 1% BM from all three size groups significantly improved WRV in comparison with the soil control and there was no significant difference of WRVs among three size groups. These results agreed well with the previous SSA results. It indicated that the residues with larger SSAs had higher WRVs.

As shown in Fig. 3(b), the WRV of the soil was increased by approximately 55%, 75%, and 150% with the addition of 3%, 5%, and 10% of FB, respectively. The addition of 3% and 10% of BM, the WRV of sandy soil was doubled, even tripled comparing with the soil control. Statistical analysis (Table 2) also indicated that the WRV of soil-residue samples was significantly improved in the addition of 3%, 5%, and 10% of FB or BM. The difference of WRV between FB and BM was attributed to their differences in surface properties and compositions. As previously mentioned, BM had larger SSA than that of FB, which resulted in more available sites for holding water. Laird and his coworkers stated that

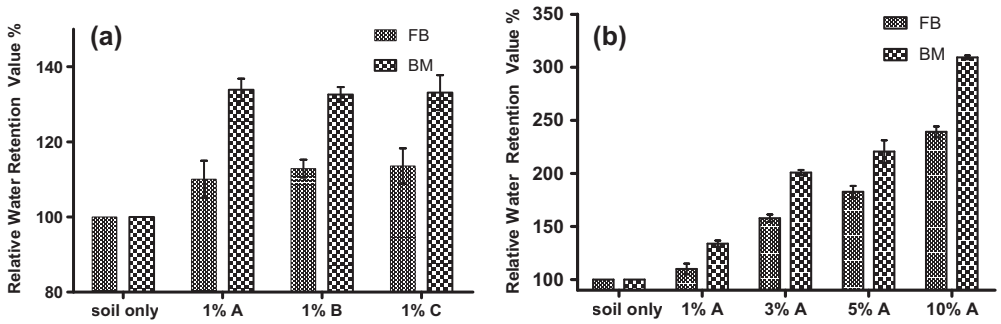

Fig. 3. a – Effects of fiber sizes on the relative water retention value (WRV) of FB- and BM- soil mixture in the presence of 1% residue, with soil only control as 100% and three size classes: A: 0.297–0.5 mm, B: 0.178–0.297 mm, C: 0.089–0.178 mm; b – Effects of different fiber loadings on the relative water retention value (WRV) of the FB- and BM-soil mixtures, with soil only control as 100% and one size class: A: 0.297–0.5 mm (based on 1%, 3%, 5%, and 10% loadings).

Table 2
Effects of fiber loadings on the relative WRV%, ammonium retention% and phosphate retention% of residue–fiber mixtures and corresponding statistical analysis.

Sample	Relative WRV%	Tukey method 95%	Relative NH4 retention%	Tukey method 95%	Relative P retention%	Tukey method 95%
Soil only	100	A	100	A	100	A
FB 1% A	110.07 ± 8.57	A B	37.60 ± 3.18	B	46.48 ± 2.31	B
FB 3% A	157.90 ± 5.93	C	2.56 ± 0.29	C	2.91 ± 0.94	C
FB 5% A	182.76 ± 9.62	D	1.04 ± 0.18	C	0.32 ± 0.09	C
FB 10% A	239.28 ± 8.68	E	0.70 ± 0.08	C	0.30 ± 0.15	C
BM 1% A	133.86 ± 5.18	B C	15.16 ± 0.84	D	22.43 ± 2.08	D
BM 3% A	200.84 ± 4.31	D F	2.08 ± 0.58	C	0.41 ± 0.10	C
BM 5% A	220.70 ± 18.44	D E	1.63 ± 0.25	C	0.40 ± 0.01	C
BM 10% A	309.46 ± 3.08	G	1.24 ± 0.37	C	0.37 ± 0.03	C

biomass amendment with greater SSA had improved water and nutrient retention of soil and biomass mixture (Laird et al., 2010). FB had about 5 times of lignin content of BM but less cellulose content. Cellulose was highly hydrophilic due to the existence of a large number of hydroxyl groups and carbonyl groups. The surfaces of BM residues were greatly fibrillated through pulping process (Hubbe et al., 2007), which could further facilitate water absorption of BM in soil as well. In FB, cellulose fibers were removed from residue surfaces and surfaces in the presence of large amount of hydrophobic lignin possibly reduced water retention. The results agreed well with several previous studies, that stated plant fiber residues with larger percentage of cellulose or smaller percentage lignin had greater water sorption or water retention in comparison with the samples with less cellulose (Johnson et al., 2007; Pejic et al., 2008; Said et al., 2009; Spence et al., 2010; Driemeier et al., 2011).

3.6. Nutrient reduction in leachate

The soil nutrient retention by incorporating FB and BM was studied in terms of different fiber types, particle size ranges, and residue loadings. The effectiveness was evaluated by nutrient concentration in the leachate, which was presented in Fig. 4(a–d). In general, soil mixture with FB and BM demonstrated excellent nutrient absorption capacity for both ammonium and phosphate ions. The addition of 1% FB with the particle size range of 0.297–0.5 mm in the soil could reduce ammonium and phosphate concentration in the leachate to about 40% and 60% of the soil control (Fig. 4(a)), respectively. While the addition of 1% BM with the same particle size range in the soil reduced ammonium and phosphate concentration to approximately 15% and 30% of the control (Fig. 4(c)). The fiber size ranges did not significantly affect the nutrient retention for both FB and BM (Supplementary information Table S1, Fig. 4(a and c)), which was similar as the WRV study. This can be explained by the identical SSAs in three different size ranges. Kithome, Li and their coworkers claimed that one of the dominating factors of ionic sorption onto coir and activated carbon was the SSA (Kithome et al., 1999; Li et al., 2010).

The residue loadings significantly affected the nutrient retention for both ammonium and phosphate ions (Table 2, Fig. 4(b and d)). As shown in Fig. 4b, in the addition of 3%, 5%, and 10% FB or BM in the soil, the ammonium concentration in the leachate were reduced to about 3%, 0.9%, and 0.7% for FB and 2.5%, 2%, and 1.5% for BM as compared with that of the soil control respectively. The phosphate concentration changes in the leachate followed the similar trend (Fig. 4(d)). When the loading levels of two residues

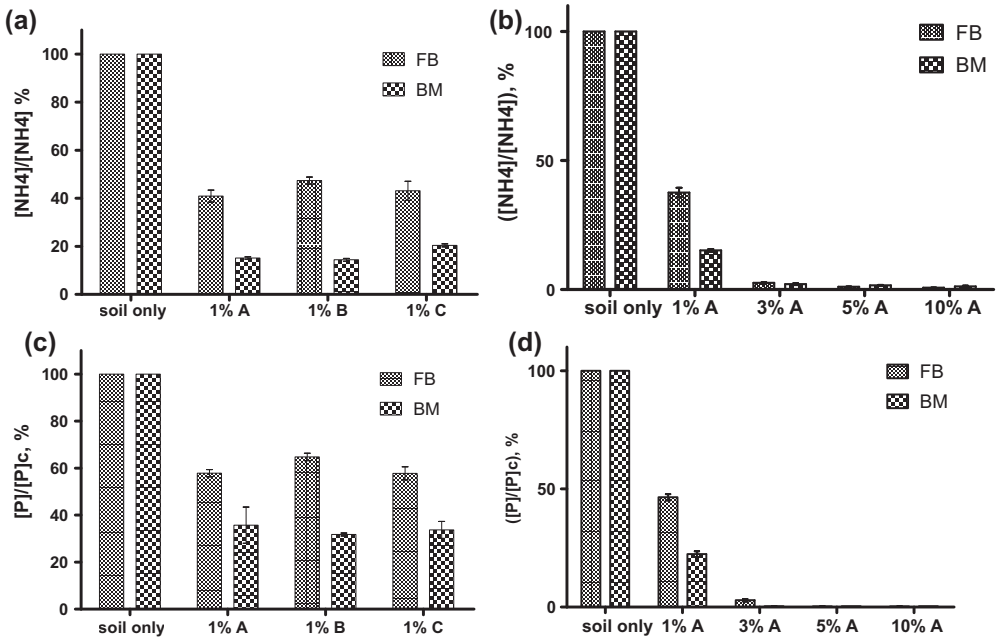

Fig. 4. a – Effects of different fiber sizes on the relative ammonium ion concentration in the leachate of FB- and BM-soil mixture in the presence of 1% residue and with three size classes: A: 0.297–0.5 mm, B: 0.178–0.297 mm, C: 0.089–0.178 mm; b – Effects of different fiber loadings on the relative ammonium ion concentration in the leachate of FB- and BM-soil mixture with one size class: A: 0.297–0.5 mm (based on 1%, 3%, 5%, and 10% residue loadings); c – Effects of different fiber sizes on the relative phosphorus concentration in the leachate from FB- and BM-soil mixture in the presence of 1% residue, with three size classes: A: 0.297–0.5 mm, B: 0.178–0.297 mm, C: 0.089–0.178 mm; d – Effects of different fiber loadings on the logarithmic relative phosphorus concentration in the leachate from FB- and BM- soil mixture, with one size class: A: 0.297–0.5 mm (based on 1%, 3%, 5%, and 10% residue loadings). All results was calculated based on 100% for soil control sample.

were higher than 5%, nutrient concentration in the leachate was extremely low.

4. Conclusions

Bio-based wastes, FB and BM were able to significantly improve water and nutrient retention of sandy soil. In 1% and 10% loading levels, FB and BM was able to improve WRV by approximately 10% and 35% to 150% and 300%, while reduce 50–99% of ammonium and phosphate concentration in the leachate compare to the soil control, respectively. The ability of FB and BM on water and nutrient retention was correlated with their characteristic properties especially specific surface areas and surface functional groups. The results of this study will be beneficial to biorefinery, pulping, and agricultural industries.

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Appendix A. Supplementary material

Supplementary data associated with this article can be found, in the online version, at http://dx.doi.org/10.1016/j.chemosphere.2013.12.088.

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