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Application of Rice Husk as Water Treatment Agent and Soil Conditioner

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Authors' contributions

This work was carried out in collaboration between all authors. Author TZEL wrote the first draft of the manuscript and prepared the rice husk bio sorbent for water treatment with author NALML. Author MEW designed and supervised the experiment for pot trials whilst. Author NNMR conducted the experiments. Authors SFS and MM designed the chemical treatment and supervised the adsorption study for humic substances. All authors read and approved the final manuscript.

Original Research Article

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ABSTRACT

Aims: Rice husk was chemically treated to be used as bio sorbent for removal of humic substances from peat swamp runoff. The spent bio sorbent was then applied as soil conditioner for enhancing plant growth.
Study Design: Randomized Complete Block Design.
Place and Duration of Study: Universiti Malaysia Sarawak, between September 2012 and August 2013.
Methodology: Rice husk was chemically treated with sodium hydroxide and citric acid. The treated rice husk was subjected to peat swamp leachate to absorb humic substances. It was then applied to grow *Brassica juncea* var. *rugosa L.* in six different treatments, each with six replicates, using Randomized Complete Block Design.
Results: Fourier Transform Infrared spectra of rice husk were characterized by major absorption bands attributable to lignocellulosic compounds. The treatment process

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primarily removed lignin and hemicellulose increasing the binding sites for adsorption. An average of 60-70% of humic content was removed from peat swamp runoff, determined based on the absorbance at 465 nm. The humic-loaded biosorbent was applied to grow *Brassica juncearugosa L*. under six different treatments to examine its effectiveness in enhancing plant growth. These include planting media only (sand and topsoil at a ratio of 3:2), [planting media + fertilizer], [planting media + humic-modified rice husk + fertilizer], [planting media + modified rice husk + fertilizer], [planting media + raw rice husk], [planting media + raw rice husk + fertilizer]. Results showed that plants grown in humic-loaded modified rice husk in combination with fertilizer exhibited significant growth improvement followed by those grown in raw rice husk and fertilizer. Others were relatively less promising suggesting that humic fortified biomass may function as soil conditioner in improving nutrient uptake.

Conclusion: The application of rice husk for water treatment and agriculture is an economically sustainable and environmental friendly approach.

Keywords: Chemically treated rice husk; water treatment; humic substances; adsorption; biosorbents; soil conditioner.

1. INTRODUCTION

The production of paddy has increased over the past many years due to the high demands for food supply. Upon the high production of paddy, a huge amount of by-product, typically rice husk is generated. In Malaysia, approximately 562 600 tonnes of rice husk is produced annually [1]. The rice husk biomass is fundamentally composed of lignin, cellulose and hemicellulose with diverse functional groups such as hydroxyl, carbonyl, carboxylic, ether and etc. They have the ability to form bonding and interaction with ions presents in natural water serving as potential bio sorbents [2] nonetheless the biomass in its untreated state is often not readily functional. It releases organic substances giving rise to high total organic carbon thus low adsorption ability [3]. To enhance their adsorption performance, various chemical modification approaches, involving alteration in the cross-linking of biomass and removal of cellulose and hemicellulose, are incorporated [4]. To date, numerous biomass derived adsorbent has been used to remove organic and inorganic contaminants with removal percentages ranging between 80% and 99% [5-10]. The application does not limit to laboratory study, it has also been extended to pilot and process scale [11-12].

In this study, rice husk was chemically modified to serve as biosorbent, aiding at harvesting humic substances from organic rich water where the spent sorbent was subsequently used to facilitate plant growth. The rationale of the study is that dissolved organic fractions, often known as humic substances, are found in considerable amount in natural water typically peat swamp runoff. The beneficial effects of humic substances in improving nutrient retention, increasing germination and stimulating root growth have been well established [13] nevertheless their presence in water causes characteristic yellowish to brownish color and unpleasant odor. Peat swamp tainted river is an important source of drinking water. In the conventional water treatment system, the humic content is coagulated with alum yielding sludge containing high aluminium, unsuitable for further use. With rice husk bio sorbent, humic compounds can be harvested and converted into value-added product for agricultural purpose as soil conditioner. In this paper, we report the preparation and characterisation of raw and modified rice husk, application of the material for adsorption of humic substances and as soil conditioner for enhancing plant growth.

2. MATERIALS AND METHODS

2.1 Sample Preparation

Rice husk was washed thoroughly under running tap water to remove dirt and foreign matters. The washed rice husk was oven dried at 105ºC and stored in air-tight container for further use.

2.2 Citric acid Treatment

Rice husk was pre-treated with 0.5M NaOH (1:3 w/v) in a water bath at 80ºC for 2 hours. They were washed, oven dried and further treated with 2.0M citric acid at 80ºC for 2 hours. The treated rice husk was then washed to pH 3 and oven dried.

2.3 Functional Groups and Morphological Characterization

2.3.1 Fourier Transform Infrared (FTIR)

All spectra of raw and modified rice husk were obtained on a Thermo Scientific FTIR system (Thermo Nicolet Analytical Instruments, Madison, WI) in triplicates using the KBr disc method with 2 mg sample in 100mg KBr. The spectra range between 4000 and 400cm^{-1} at a resolution of 4cm^{-1}. The spectra were analyzed semi-quantitatively where the relative abundance of absorption bands were compared to identify the changes before and after treatment [14-17].

2.3.2 Scanning Electron Microscope (SEM)

The morphological characteristics were observed using a Scanning Electron Microscope (Model JEOL JSM-6390LA, Japan) with an accelerating voltage of 5kV at ×500 magnification. The samples were coated with thin film of platinum prior to examination.

2.4 Adsorption of Humic Substances

A total of 1 g treated rice husk was agitated with 20mL of water collected from the Asa Jaya River at 130rpm for 15 min. Note that the river receives dark colored runoff from the adjacent peat swamp forest; prior to treatment, the river water was filtered through 0.45µm membrane filter. The mixture was then eluted through a glass column at a flow rate of 1.0mL/min to separate the biomass. The experimental conditions including dosage, contact time and flow rate were optimized using the one-factor-at-a-time approach with six replicates each experiment. The optimization may be alternatively performed according to the full factorial design. Comparatively, it is cheaper and less time consuming than the traditional approach; in addition, the interaction between factors can be systematically evaluated permitting more efficient optimization [18].

The removal efficiency was determined based on the absorbance at 465nm using a UV-Vis spectrophotometer [17]. The wavelength is the characteristic of humic fraction that introduces color to water and absorbs radiation in the visible region. Fundamentally, the absorbance is proportional to the concentration of dissolved organic carbon. If the organic matter is successfully removed, a reduction in absorbance is anticipated. The percentage of absorbance reduction against the absorbance of untreated water is calculated to suggest the average removal percentage. The results are summarized in Table 1.

Table 1. Optimization of operating conditions

Factors	Parameters	Mean absorbance	Average percentage of removal (%)
Dosage (mL)	20	$0.030{\pm}0.001^{a}$	60.12%
	40	$0.044{\pm}0.001^{b}$	41.78%
	60	$0.050{\pm}0.001^{cd}$	33.71%
	80	$0.049{\pm}0.001^{c}$	35.96%
	100	$0.051{\pm}0.001^{d}$	32.98%
Contact Time (min)	15	$0.040{\pm}0.001^{a}$	47.68%
	30	$0.056{\pm}0.001^{b}$	26.86%
	60	$0.031{\pm}0.001^{c}$	59.60%
	90	$0.027{\pm}0.001^{d}$	64.12%
	120	$0.026{\pm}0.001^{d}$	65.50%
Flow Rate (mL/min)	1	$0.025{\pm}0.001^{a}$	67.15%
	2	$0.034{\pm}0.001^{b}$	55.81%
	7	$0.031{\pm}0.001^{c}$	58.82%
	15	$0.032{\pm}0.001^{bc}$	58.04%

** Different letters in mean absorbance of each parameter for different factors indicating significant difference at P = 0.05 using Tukey's test*

2.5 Pot Trials

Pot trials were performed to evaluate the effectiveness of humic-loaded biosorbent as soil conditioner in improving plant growth using Randomized Complete Block Design (RCBD). *Brassica juncea* var. *rugosa L*. were planted into six treatments, each with six replicates, as summarized in Table 2. The planting media was sand and topsoil at a ratio of 3:2. For treatments involving fertilizers, NPK fertilizers were applied in split application with the dosage recommended by Shekhawat et al. [19]. The amount of humic composite applied was estimated and adapted from Rajpar et al. [20]. Note that all treatments were monitored under controlled environment i.e., water, light and pest control. The plant growth parameters including length of stem, leaf area and number of leaves were recorded once every fortnight. At the end of the pot trial, the dry matter and root-to-shoot ratio of harvested *Brassica juncea* var. *rugosa L*. were measured to determine the physiological growth of the tested plants.

Table 2. Treatments for pot trials

Treatment	Details on plant growth media for each treatment
T1	Control (Planting media only)
T2	Planting media + NPK Fertilizer
T3	Planting media + Modified rice husk (with humic)
T4	Planting media + Modified rice husk (with humic) + NPK fertilizer
T5	Planting media + Raw rice husk
T6	Planting media + Raw rice husk + NPK fertilizer

2.6 Data Analysis

The FTIR spectra were analyzed using the peak detection algorithm reported by Sim and Ting [13]. The algorithm produces a peak table representing a summary of the peaks identified including the locations and the corresponding peak areas according to samples. The peak table was square rooted and standardized prior to Principal Component Analysis (PCA) to determine whether untreated and treated rice husk are distinguishable. The peak table can be evaluated, according to variables, to identify the alteration experienced upon treatment. For plant growth parameters, mean values and standard deviations were calculated from replicate measurements and statistical analysis using one-way ANOVA and Tukey's test, at 95% confidence interval, were conducted using SPSS version 21.0.

3. RESULTS AND DISCUSSION

3.1 Characterization of Rice Husk Biosorbents

Potential adsorbents usually contain porous spaces where the adsorption process is typically influenced by their surface area and polarity [21]. Raw biomasses are often not ready to function as biosorbent as their active functional groups are embedded within lignin and hemicellulose. Alkali and acid pretreatments are incorporated to break down lignin and to hydrolyze cellulose and hemicellose, increasing the binding sites. Fig. 1 illustrates the FTIR profiles of raw and treated rice husk. Fundamentally, the spectra are characterized by several major absorption bands attributable to the presence of lignocellulosic compounds at 3400 cm^{-1}, 2920-2850 cm^{-1}, 1700-1600cm^{-1}, 1511cm^{-1} and 1157cm^{-1}. The absorption band at 1157cm^{-1} is the characteristic of glycosidic linkages (C-O-C ring vibrational stretching), commonly found in all spectra of biomasses. The bands at 1700-1600 cm^{-1} and 3400 cm^{-1} are typically assigned to carbonyl and hydroxyl groups whilst the presence of lignin is represented by the band at 1511 cm^{-1}, an indicative of C=C stretching vibrations of aromatic rings [22]. The FTIR spectra, decovoluted with the peak detection approach, produced a peak table allowing multivariate analysis with PCA. The scores plot of PC2 vs PC1 in Fig. 2 shows that untreated and treated samples are noticeably distinguishable implying that the treatment has resulted in considerable changes.

The changes are depicted based on the relative abundance of some characteristic bands of lignocellulosic compounds according to sample types as shown in Fig. 3. The band at 1157 cm^{-1} and 3400 cm^{-1} are markedly reduced suggesting hydrolysis of glycosidic and OH bonds. The absorbance at 1420cm^{-1} and 894 cm^{-1} are indicative of crystalline and amorphous properties of cellulose; it is demonstrated that treated rice husk is represented by a smaller ratio of A1420/A894 indicating an increase in amorphous cellulose due to distortion [23]. In addition, the band of lignin at 1511 cm^{-1} is found to diminish after NaOH pretreatment inferring the degradation of lignin. The band is re-introduced and shifted to 1514cm^{-1} after citric acid treatment, possibly due to the presence of lignin-carbohydrate complex material, known as pseudo-lignin. This compound is a result of the reaction between residual lignin and cellulose/ hemicelluloses in acidic condition [24-25]. Besides, re-condensation and deposition of lignin on the cellulose surface may also explain the reappearance of lignin band in treated rice husk [26]. The band at 1053 cm^{-1} and 1456 cm^{-1}, assigned to methoxyl-O-CH_3, is found abundantly in lignin and hemicellulose [27]; it is likewise diminished after treatment corroborating delignification and dissolution of hemicellulose.

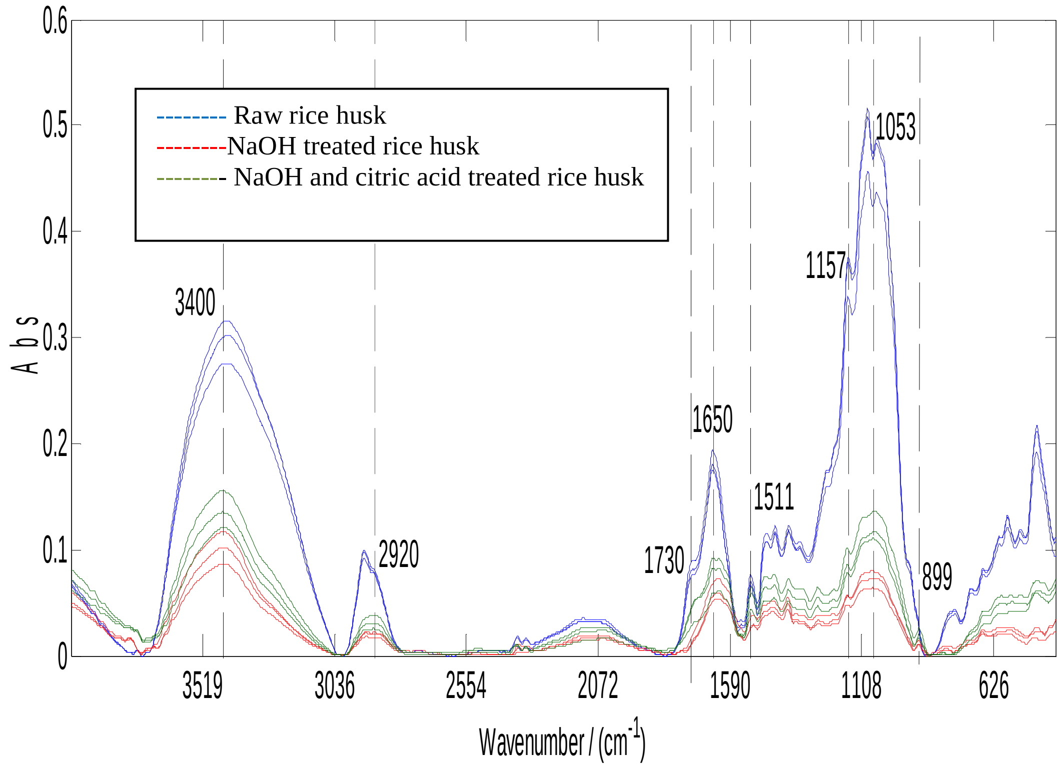


Fig. 1. The FTIR spectra of raw and modified rice husk

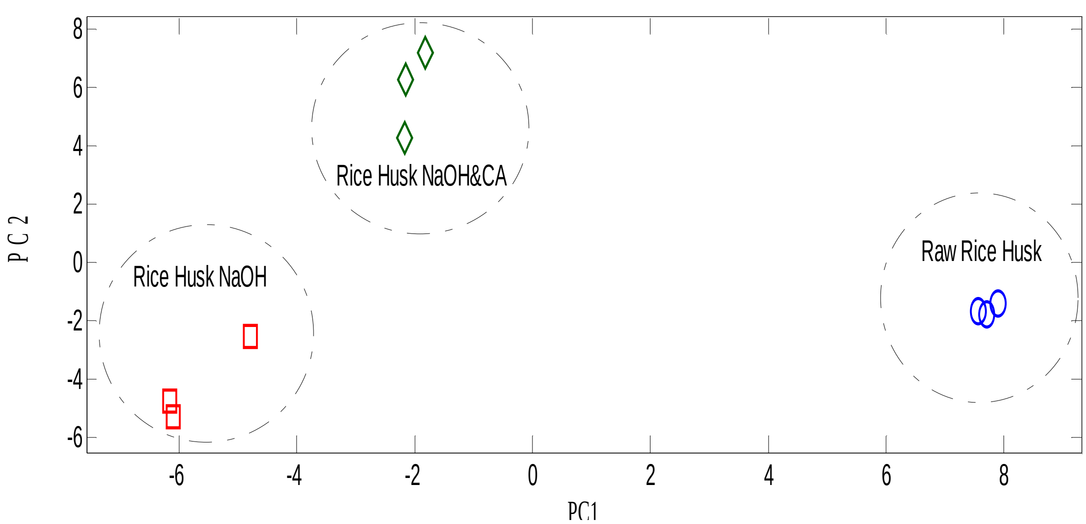


Fig. 2. The PCA scores plot of raw and modified rice husk

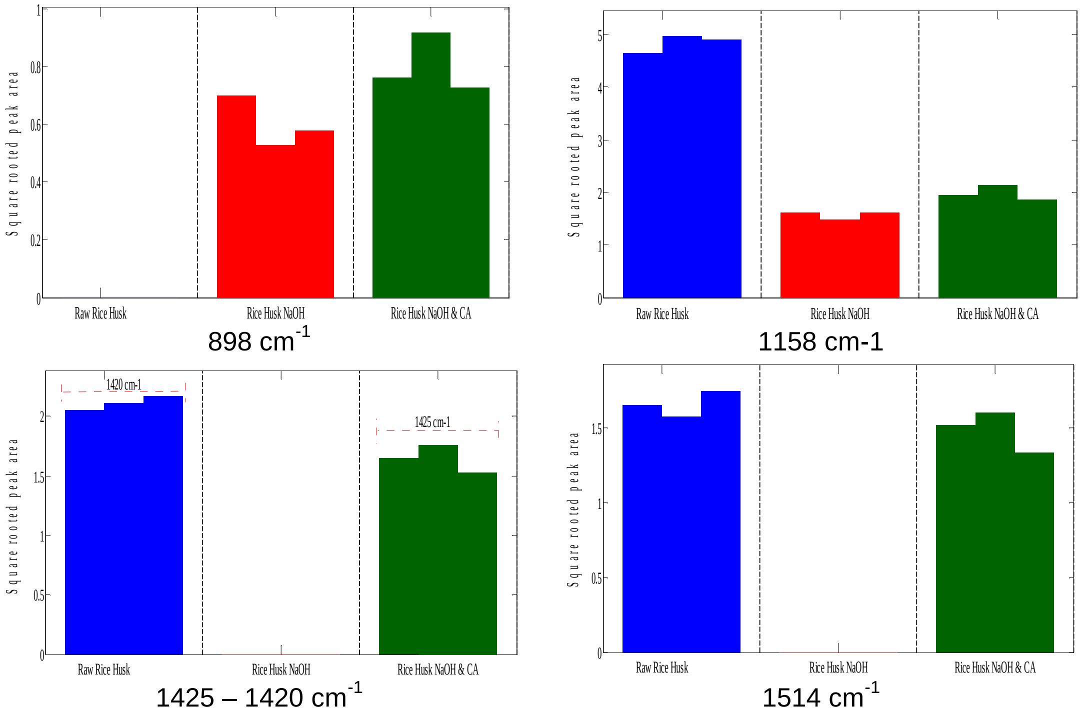
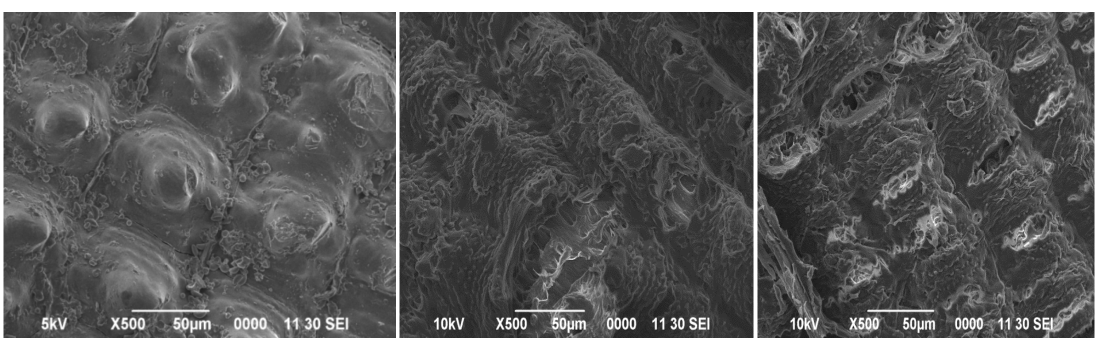


Fig. 3. The relative abundance of some absorption bands before and after treatment

The micrographs of raw rice husk and treated rice husk are shown in Fig. 4. Morphologically, the raw rice husk shows well-defined rigid structure with conical-shaped protrusion on its surface. The protrusions are degraded after NaOH pretreatment where rough surface with minimal fractures is observed suggesting removal of extractives, waxes and oils from the surface. Addition of citric acid to NaOH treated biomass further demonstrates prominent fractured on the outer surface area exposing the inner layer membranes.



(a) Raw rice husk **(b) NaOH treated rice husk** **(c) NaOH and citric acid treated rice husk**

Fig. 4. SEM images of raw and treated rice husk

3.2 Biosorbents for Water Treatment

Treated rice husk was employed to remove humic substances from peat swamp runoff, evidenced by reduction in absorbance. An average of 60-70% of removal is demonstrated using treated rice husk, statistically different from raw rice husk with no adsorption ability ($P = 0.05$) on the contrary introduces additional color suggesting leaching of tannin and lignin compounds.

3.3 Biosorbents for Soil Amendment

Table 3 shows the growth performance of *Brassica juncea* var. *rugosa L.* grown under six different treatments. Statistical analysis indicates that there are significant differences in plants grown in T1 [planting material only] and T4 [planting material + Modified rice husk + humic + NPK fertilizer] throughout the experiment for leaf area, number of leaves and length at 95 % confidence interval (P=0.05). The growth performance of T1 [planting material only], T2 [planting material + fertilizer], T3 [planting material + humic loaded modified rice husk] and T5 [topsoil + raw rice husk] however are not significantly different at each observation period. The results show that under poor sandy soil, no improved plant growth is evidenced even with fertilizer; this may imply that the available nutrients are unable to be taken up efficiently. The plants grown in modified rice husk with humic substances and added fertilizer grew better than plants grown in untreated rice husk, with and without fertilizer. This appears to suggest that humic substances may have served as a carrier to enhance the uptake of nutrients hence promoting its growth in sandy soil.

Table 3. Growth parameters of *Brassica juncea* var. *rugosa L.* measured through 8 weeks

Growth parameters	Treatment	Observations			
		Week 2	Week 4	Week 6	Week 8
Length of leaves (cm)	T1	$0.50{\pm}0.06^{a}$	$1.50{\pm}0.82^{a}$	$2.33{\pm}0.61^{a}$	$3.00{\pm}0.42^{a}$
	T2	$0.50{\pm}0.06^{a}$	$3.60{\pm}0.51^{b}$	$5.48{\pm}0.90^{b}$	$5.68{\pm}0.98^{b}$
	T3	$0.55{\pm}0.14^{a}$	$1.62{\pm}0.62^{a}$	$2.93{\pm}0.56^{a}$	$2.95{\pm}0.54^{a}$
	T4	$1.12{\pm}0.22^{b}$	$6.82{\pm}0.63^{c}$	$8.37{\pm}1.07^{c}$	8.63 ± 1.18^{c}
	T5	$0.55{\pm}0.24^{a}$	$1.18{\pm}0.48^{a}$	$1.18{\pm}0.48^{a}$	$1.98{\pm}1.04^{a}$
	T6	$1.42{\pm}0.21^{b}$	$4.93{\pm}0.37^{d}$	$4.93{\pm}0.37^{b}$	$6.20{\pm}0.80^{b}$
Leaf Area (cm^3)	T1	$0.92{\pm}0.08^{a}$	$2.07{\pm}0.39^{a}$	$2.35{\pm}0.31^{a}$	$2.72{\pm}0.60^{a}$
	T2	$1.07{\pm}0.39^{ab}$	$2.85{\pm}0.58^{a}$	$4.17{\pm}1.55^{a}$	$4.60{\pm}2.01^{ab}$
	T3	$1.17{\pm}0.45^{ab}$	$1.60{\pm}0.28^{a}$	$1.78{\pm}0.33^{a}$	1.78 ± 0.33^{a}
	T4	$1.55{\pm}0.29^{bc}$	$8.87{\pm}1.63^{b}$	$9.38{\pm}2.39^{b}$	$13.02{\pm}2.71^{c}$
	T5	$0.87{\pm}0.19^{a}$	$2.35{\pm}0.61^{a}$	$2.47{\pm}0.53^{a}$	$2.47{\pm}0.53^{a}$
	T6	$1.70{\pm}0.14^{c}$	$6.90{\pm}2.04^{b}$	$7.25{\pm}2.57^{b}$	$7.67{\pm}2.79^{b}$
Number of Leaves	T1	$2.00{\pm}0.00^{a}$	$3.17{\pm}0.98^{a}$	$3.17{\pm}0.75^{a}$	$3.33{\pm}0.52^{a}$
	T2	$2.00{\pm}0.00^{a}$	$4.50{\pm}1.52^{abc}$	$4.17{\pm}0.98^{abc}$	$5.17{\pm}1.47^{ab}$
	T3	$2.00{\pm}0.00^{a}$	$4.00{\pm}0.00^{abc}$	$3.83{\pm}0.41^{ab}$	$3.83{\pm}0.41^{a}$
	T4	$2.33{\pm}0.52^{b}$	$5.50{\pm}0.55^{c}$	$5.17{\pm}0.41^{bc}$	$7.83{\pm}1.83^{c}$
	T5	$2.00{\pm}0.00^{a}$	$3.33{\pm}1.03^{ab}$	$3.67{\pm}0.52^{a}$	$4.33{\pm}0.82^{ab}$
	T6	$2.33{\pm}0.52^{a}$	$4.83{\pm}0.75^{bc}$	$5.33{\pm}1.21^{c}$	$5.83{\pm}0.41^{b}$

** Different letters in each treatment for growth parameters at each observation period indicating significant difference at P = 0.05 using Tukey's test*

Fig. 5 illustrates the dry matter and root-to-shoot ratio of *Brassica juncea* var. *rugosa L.*; plants grown in modified rice husk with humic substances and fertilizer illustrate the highest dry matter percentage that is statistically different from those planted in T2 and T6 (under conditions with fertilizer alone and fertilizer and raw rice husk, respectively). This is in agreement to the findings of Rao et al. [28] that dry matter yield increases with application of humic acid. Other studies further demonstrate that humic substances have the ability to enhance plant growth with less fertilizer; the beneficial effects of combining humic acid and fertilizer was evidenced with improved growth and yield of green mustard [29]. Plants grown in raw rice husk and fertilizer also exhibit encouraging growth though not as good as that with humic composite and fertilizer; the positive performance may be associated to the nature of lignocellulosic biomass as a potent soil stabilizer. After eight weeks, it is evidenced that plants grown in humic and fertilizer are better than that grown in fertilizer alone (T2 and T6). This suggests that the humic loaded biomass may be used as soil conditioner to facilitate nutrients retention nevertheless the beneficial effects in promoting plant growth would require further long-term experimentation and testing on various types of plant species.

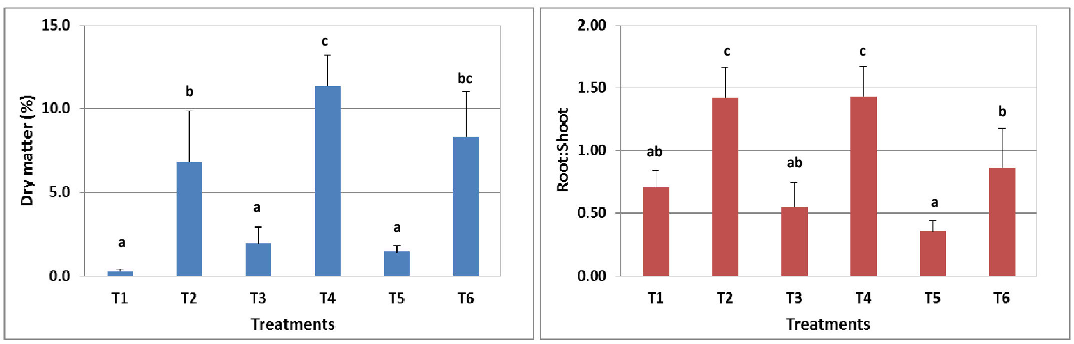


Fig. 5. Dry matter and root-shoot ratio of *Brassica juncea var. rugosa L.* under different treatment

4. CONCLUSION

The application of rice husk for water treatment and agriculture is an economically sustainable and environmental friendly approach. Rice husk was chemically treated, experiencing removal of lignin and hemicellulose hence enhancing the adsorption ability of the material. It was used to remove humic substances from water tainted by peat swamp runoff where the spent sorbent was recycled as soil conditioner. Results show that *Brassica juncea*var. *rugosa L.* grown under rice husk fortified with humic compounds and fertilizer is encouraging.

COMPETING INTERESTS

Authors declare that there are no competing interests.

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