

BIOENERGY STUDY ON GRASS (*Pennisetum Hordeorides* Lam.)

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Abstract

Hydrothermal treatment is one of the environmentally friendly methods for the conversion of biomass into valuable end products such as biofuel, hydrochar and biochar. In this research work, hydrothermal treatment of grass (*Pennisetum hordeorides* Lam.) was carried out at 100°C to 140 °C for 20 min to 40 min as the preliminary step to provide bioenergy products such as bioethanol and biochar. The maximum reducing sugar content of liquid fraction, 43.46 mg/mL was achieved after hydrothermal treatment at 110 °C for 30 min and further yeast fermentation of reducing sugar was accomplished to obtain 2% by volume of bioethanol. Carbonization of solid residue after hydrothermal treatment was conducted for biochar at different temperatures (300 °C, 400 °C and 500 °C) and times (0.5 hr, 1 hr, 2 hr, 4 hr, 8 hr). Characterization of grass such as cellulose, hemicellulose, and lignin contents before and after hydrothermal treatment was conducted. High heating value (HHV) and energy densification were evaluated for energy values of biochar. The maximum HHV (26.78 MJ/kg) and energy densification (1.28) had resulted for the carbonization temperature of 500 °C for 2 hr. The changes in surface morphology and functional groups in raw grass, HTG and biochar were observed by Scanning Electron Microscopy (SEM) and by Fourier Transform Infrared Spectroscopy (FTIR).

Keywords: hydrothermal treatment, bioethanol, carbonization, biochar

Introduction

Transformation of biomass to energy is a sustainable solution to reduce greenhouse gas emissions and the secondary as well as tertiary biomass can be substituted for fossil fuels. Bioenergy is related to the process heat (thermal energy used for residential, commercial, or industrial applications), biopower (conversion of biomass into electric power intended to stationary application), and biofuels (chemicals derived from biomass designated for transportation purpose) (Brown, 2014). Bioethanol and biochar are regarded as the bioenergy providing substances with the significant characteristics of structural composition, rich in carbon and renewable content (Kandasamy et al., 2021). Several technologies to convert biomass into useful products are mainly categorized to thermochemical (non-catalytic) route and biological (catalytic) route (Garba, 2020).

Thermochemical biomass conversion processes involve combustion (heating the biomass in the presence of oxygen), gasification (partial biomass oxidation at 700 °C - 900 °C), pyrolysis (heating the biomass at 300 °C - 900 °C under the absence of oxygen), torrefaction (low-temperature pyrolysis) and carbonization (two types - flash and hydrothermal processing) (Kumar et al., 2020). Hydrothermal treatment has been extensively studied as a first step for a biorefinery due to its environmentally friendly advantages over other treatments (del Río et al., 2020). This co-production of high value-added products and bioethanol allows the reduction of capital costs involved in the lignocellulosic biomass processing (Du et al., 2020, Aristiz´abal-Marulanda & Cardona, 2021).

The present study intended to explore the potential bioenergy of grass (*Pennisetum hordeorides* Lam.). Bioethanol and biochar were prepared by further processing of liquid and solid portions resulting from hydrothermal treatment of grass stems. Fermentation of liquid portion and carbonization of solid residue were carried out for bioethanol and biochar, respectively. Scanning Electron Microscopy (SEM) and Fourier Transform Infrared (FTIR) analyses of grass stems before and after hydrothermal treatment and biochar were conducted for the surface morphology and

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functional groups present. Proximate composition of grass stems before and after hydrothermal treatment, strength of bioethanol, and high heating value (HHV) of biochar were analysed.

Materials and Methods

Raw Materials

Grasses (*Pennisetum hordeorides* Lam.) as shown in Figure (1) were harvested from the campus of East Yangon University, Yangon Region. *Saccharomyces cerevisiae* – baker's yeast was purchased from Supershell Chemical Dealer, 27th street, Pabedan Township.



Figure (1) Grass (*Pennisetum hordeorides* Lam.)

Methods

Processing of Bioethanol and Biochar

Grass stems were cleaned, and chopped into small pieces and then air-dried for 2 hr until the moisture content was 10-12%. Hydrothermal treatment of crushed grass stems was conducted in a 100 mL, polytetrafluoroethylene (PTFE) lined stainless steel synthesis reactor. The amount of biomass and distilled water were added into the reactor with solid to liquid ratio of 1:9 (w/v). Then the reactor was securely sealed and placed in a hot air oven. The treatment was conducted at various temperatures of 100 – 140 °C and heating times (20 - 40 min). After the hydrothermal treatment was completed, the reactor was cooled and the broth was subjected to vacuum filtration for the separation of liquid and solid residue. Fermentation of liquid fraction was carried out using *Saccharomyces cerevisiae* – baker's yeast for production of bioethanol. pH of the liquid portion was maintained at pH 5.6 and 3 g/L of yeast was used in fermentation. After fermentation, bioethanol was separated by distillation. The strength of bioethanol was measured by portable alcohol meter (ATAGO AL-21 Alpha Handheld Alcohol Refractometer, 0 to 21%). Meanwhile, the solid residue was carbonized in a muffle furnace at various temperatures of 300 °C, 400 °C and 500 °C for 0.5 hr, 1 hr, 2 hr, 4 hr, and 8 hr, respectively. The resulting solid residue biochar was washed with water for several times and dried.

Determination of Reducing Sugar Content of Liquid Fraction during Hydrothermal Process

Reducing sugar content of liquid fraction resulting from hydrothermal treatment was determined by Lane and Eynon's method (Pearson, 1976). The liquid was titrated with standard Fehling solutions A and B using methylene blue as indicator.

Characterization of Grass Before and After Hydrothermal Treatment

The air-dried biomass was pulverized in a grinder into -16 mesh size particles. Proximate composition such as extractive matter, lignin content, holocellulose, hemicellulose and cellulose content of grass before and after hydrothermal treatment was determined.

Determination of Extractive Matter

The procedure for determination of extractive matter was conducted according to the method of ISO 14453:2014. Extractive matter present in the samples was determined by carrying out solvent extraction with acetone in Soxhlet apparatus at 90°C for 2 hr. The sample was taken out, dried and weighed.

Determination of Acid Insoluble Lignin

Acid insoluble lignin of the extractive free sample was determined based on the Klason Method. 72% H₂SO₄ was used for hydrolysis of biomass for lignin until complete hydrolysis was assured by further hydrolysis with 4% H₂SO₄. The solid residue was collected, dried and weighed for acid insoluble lignin.

Determination of Holocellulose, Hemicellulose and Cellulose Content

Holocellulose content in extractive free sample was determined by TAPPI T9-wd -75. Extractive free sample was hydrolyzed with a mixture of acetic acid and 80% sodium chlorite solution at 90°C for 1 hr. The complete hydrolysis was assured by adding of a mixture of acetic acid and 80% sodium chlorite solution. The filtered residue was dried and weighed for holocellulose. Holocellulose obtained from above was further hydrolyzed with 17.5% sodium hydroxide solution. The residue was neutralized with 1M acetic acid to pH 7, dried and weighed for hemicellulose. Cellulose content can be calculated by subtracting hemicellulose content from the holocellulose content.

Characterization of Biochar

Volatile Matter content (VM), ash content and Fixed Carbon content (FC) were analyzed by ASTM-D1762-84 method for its high heating energy value and energy densification value.

Determination of Volatile Matter (VM)

Volatile matter was determined by igniting the sample in a muffle furnace at 850°C for 10 min. The weight loss was the amount of all volatile matter.

Determination of Ash Content

The dried sample was incinerated in a muffle furnace at 750 °C for 6 hr. The residue was weighed for ash content.

Determination of Fixed Carbon Content (FC)

The fixed carbon of samples was calculated by the subtraction of the sum of ash and volatile matter contents from 100.

Determination of High Heating Value (HHV)

The HHVs of samples were estimated using the following equation of Kieseler et al., 2013.

$$\text{HHV (MJ/kg)} = 0.4108\text{FC} + 0.1934\text{VM} - 0.02111\text{Ash}$$

Determination of Energy Densification

Energy densification was calculated with the ratio of HHV of biochar and HHV of biomass described by Fan et al., 2018.

Scanning Electron Microscopy (SEM) and Fourier Transform Infrared Spectroscopy (FTIR) Analyses

Changes in surface morphology and the functional group of raw grass, HTG and biochar were analyzed by Scanning Electron Microscopy (SEM) (EVO-18, ZEISS, Germany) and by Fourier Transform Infrared Spectroscopy (FTIR) (IR Tracer-100, SHIMADZU, Japan) at the Department of Chemistry, West Yangon University, Htantabin Township, Yangon Region.

Results and Discussion

The hydrothermal treatment of grass stem was conducted for the purpose of energy providing intermediate products by varying the treatment temperature and time. Figure (2) and Figure (3) show the effect of temperature and time on the reducing sugar content of liquid fraction. Maximum amount of reducing sugar, 43.46 mg/mL was obtained by the hydrothermal treatment at 110°C for 30 min. Table (1) presents the proximate composition of grass stems before and after hydrothermal treatment. Cellulose and lignin contents in raw grass (*Pennisetum hordeoides* Lam.) were ranged to the similarity of those contents of switchgrass and mixed grass (27-35% cellulose, 12-21% hemicellulose, 15-20% lignin) cited in Williams et. al., (2017) whereas hemicellulose content was different from that of the value mentioned in literature. High cellulose content represents the potential component for the conversion into biochar (Waliszewska et al., 2021). After hydrothermal treatment, degradation of biomass was proved by increase in lignin content of 23.12% and cellulose content of 23.98%, respectively in solid residue as shown in Table (1). Simultaneously, release of reducing sugar was observed in the liquid fraction with the evidence of decrease in hemicellulose content of 13.21% found in solid residue. Soluble fractions of cellulose and hemicellulose in liquid fraction may consist of six carbon sugars (hexose) and five carbon sugar (pentose). As a result, hexoses were fermented to ethanol and pentoses were resistant to fermentation that was processed by baker's yeast. A few microorganism strains were confirmed for fermentation of pentose sugars (Liu, 2005). The strength of bioethanol by yeast fermentation of liquid fraction resulting from hydrothermal treatment was found to be 2% by volume. This result could be compared with the conventional treatment method such as acid hydrolysis using concentrated sulphuric acid followed by yeast fermentation. 3.83 % by volume of bioethanol was achieved by conc: H₂SO₄ treatment (3.4% conc: H₂SO₄, at 100°C for 86 min) in which 61.08 ± 7.84 mg/g of reducing sugar was resulted (Soe Soe Than, 2014). Harsh acid treatment of grass stem reached to release more reducing sugar that gave slightly higher strength of bioethanol when compared to novel method of hydrothermal treatment.

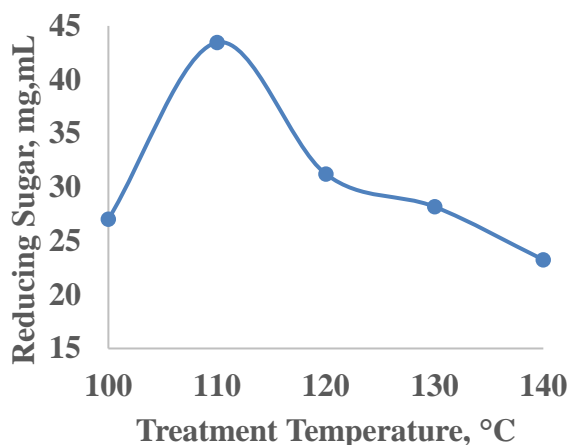


Figure (2) Effect of Treatment Temperature on Reducing Sugar Content

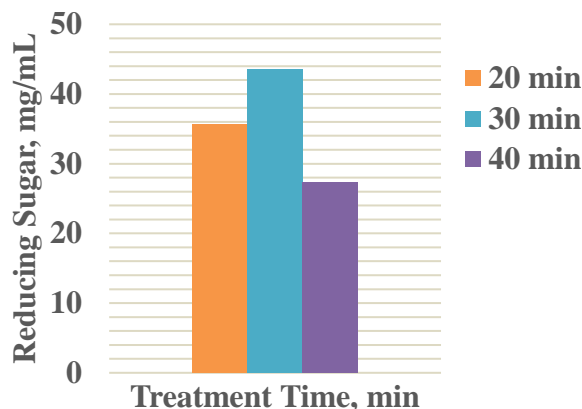


Figure (3) Effect of Treatment Time on Reducing Sugar Content

Table (1) Composition of Grass Before and After Hydrothermal Treatment

Composition (%)	Grass Stem (dry basis)	
	Raw grass	Solid residue after hydrothermal treatment (HTG)
Lignin	19.72	24.28
Holocellulose	60.7	65.15
Hemicellulose	27.18	23.59
Cellulose	33.53	41.57

Figures (4) and Figure (5) depict the effect of carbonization temperature and time on High Heating Value (HHV) of biochar obtained from cellulose and lignin rich residue after hydrothermal treatment (HTG). HHV can be supposed to the energy content of biomass which is an essential parameter for the assessment of a combustion system. HHV is determined, based on the results of proximate analysis of ash content, volatile matter content, and fixed carbon content. The highest result was observed at carbonization temperature of 500°C for 2 hr. Thus, the higher temperature of carbonization from 300°C - 500°C would increase HHV from 22.57 MJ/kg to 26.78 MJ/kg for a residence time of 2 hr. The longer residence time (4 hr to 8 hr) caused slight decrease in HHV of biochar. It may be due to increase in ash content of biochar with longer carbonizing time. According to the experimental results, the increment of carbonization temperature (300-500°C) for time (0.5 – 8 hr) was observed with gradually decrease in the amount of volatile matter and increase the ash content. When compared to HHV of raw grass, HTG, and biochar as shown in Figure (6), HHV of biochar was significantly large. It was due to increase in cellulose and lignin contents in solid residue by hydrothermal treatment.

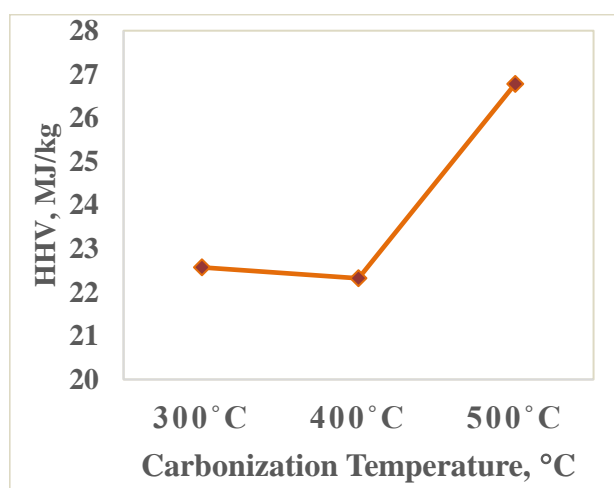


Figure (4) HHV of Biochar at Different Carbonization Temperature for 2hr

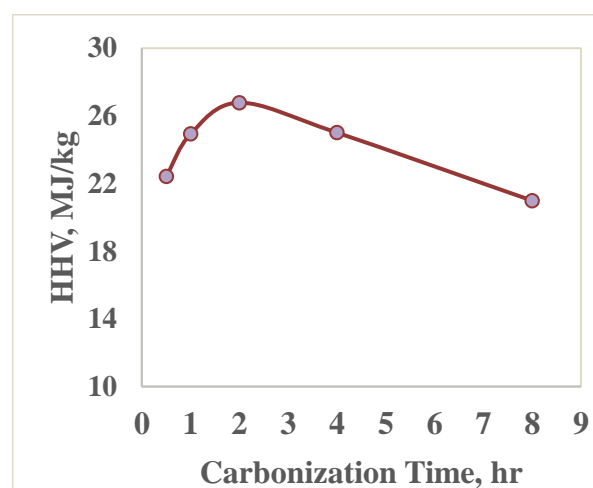


Figure (5) HHV of Biochar at Different Carbonization Time at 500°C

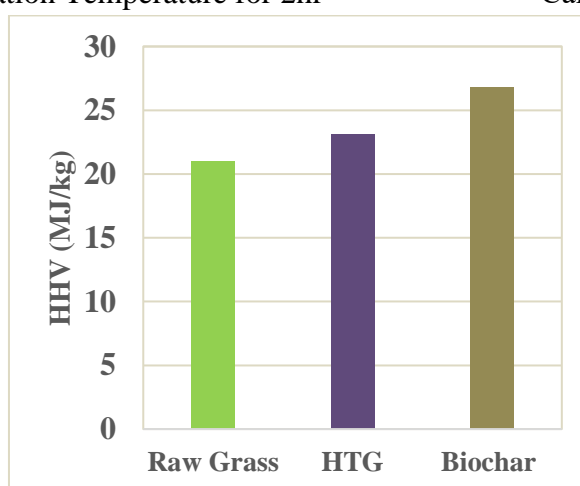


Figure (6) HHV of Raw Grass, HTG and Biochar

As can be seen in Figure (7), the highest energy densification was observed as 1.28 at the carbonization temperature of 500°C for 2 hr. Zhang et al., (2015) and Liu et al., (2018) also stated that the conversion of biomass to biochar needs proper carbonization temperature and time. If the carbonization temperature and/or residence time is higher than the suitable carbonization condition, most of the organic compounds are susceptible to change into ash. On the other hand, biomass could not be completely converted to the volatile compounds and underwent decomposition of organic compounds. The observed results were consistent with the statement of Mäkelä et al., (2015). It has been reported that higher temperature led to increase the degradation of hemicellulose, cellulose, and lignin which was accompanied by decarboxylation of biomass components. Consequently, a desirable decrease in oxygen content of the solid enabled increasing energy densification in biomass. HHV and energy densification are important assessment to evaluate the success of biochar produced from biomass (Elaiwu and Greenway, 2019 and Fan et. al., 2018). The maximum amount of HHV and energy densification indicate the destruction of low energy chemical components with the production of high energy components.

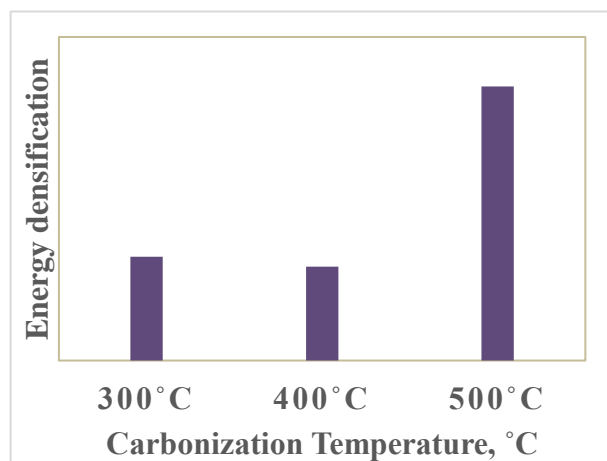


Figure (7) Energy Densification of Biochar

The surface morphology changes in raw grass, HTG and biochar were studied by Scanning Electron Microscopy (SEM). As shown in Figure (8), raw grass exhibited a smooth surface structure, but it was evident with fragmentation and fibrillation by the appearance of a higher number of droplets like structures in solid residue after hydrothermal treatment (HTG). After carbonization, the surface of biochar became rough and porous when compared with that of HTG after hydrothermal treatment. It may be due to the stresses gained by the hydrothermal treatment and carbonization. Yang et. al., (2015) and Nizamuddin, (2017) reported that the decomposition of hemicellulose, cellulose and lignin can attribute to the coarseness of the surface of the biomass. It may be associated with the removal of hemicellulose by hydrothermal treatment and the droplets observed on the surface of HTG could be due to the increase of some residues accompanied by lignin modification. Under the carbonization condition, the small pores and fragments were gradually formed on the surface of biochar because of the release of volatile matters. Moreover, the regular pore structures were found in biochar because elevated temperature and increased residence time enhanced the decomposition of the cellulose and hemicellulose as well as a small amount of lignin components (Fan et al., 2018).

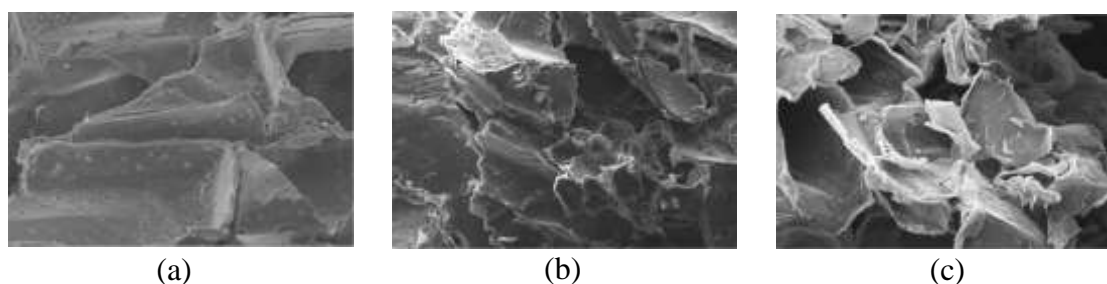


Figure (8) Scanning electron microscopy images for (a) Raw grass, (b) HTG (c) Biochar

FTIR spectra of biomass before and after hydrothermal treatment and biochar (after carbonization of solid residue) are depicted in Figure (9). The absorption band within the range of $3,500\text{--}3,000\text{ cm}^{-1}$ indicated that the stretching of O–H groups of hydroxyl and carboxyl groups present in grass stems before and after hydrothermal treatment and biochar. It can also be found that the peaks between $2000\text{--}1660\text{ cm}^{-1}$ of C–H stretching vibration for the methylene groups, the absorption band nearly 1500 cm^{-1} of C=C stretching vibration for the aromatic compound, the peak at 1732 cm^{-1} and 1248 cm^{-1} of C=O and C–O bond in acetyl ester groups

exhibited for before and after the treatment of grass. The significant appearance of absorption bands at 1600 cm^{-1} and around $1480 - 1350\text{ cm}^{-1}$ indicated the C-H of alkane group and C=C of aromatic groups in HTG and biochar. When compared with FTIR spectra of grass before and after hydrothermal treatment, the intensity of peaks around 3390 cm^{-1} became slightly diminished for HTG. This was due to the disruption of hydrogen bonds in cellulose (Buranov and Mazza, 2010). The appearance of a band around 2900 cm^{-1} ($2,920\text{ cm}^{-1}$, 2899 cm^{-1}) in HTG distinctly indicated diminishing intensity than that of raw grass. Kumar et al., (2009) reported the statement that the band around 2900 cm^{-1} was ascribed to C-H stretching vibration within the methylene portions of the cellulose. The bands at 1732 cm^{-1} and 1248 cm^{-1} for HTG showed a relative decrease in the intensity than that of raw grass. Nitsos et al., (2021) stated that the corresponding components of acetyl ester units might be present in hemicellulose and also described that decrease in the intensity of bands after hydrothermal treatment. It indicated the dissolution of hemicellulose from biomass and its deacetylation (Pandey, 1999). The absorption band around 1515 cm^{-1} indicates the presence of the aromatic ring in lignin (Colom et al., 2003). Nongthombam et al., (2017) observed that a small increase in the intensity of the peak at 1515 cm^{-1} could be attributed to the enhancement of the lignin concentration in the treated biomass samples. In the case of biochar, the intensity of the peaks at 3396 cm^{-1} , 2900 cm^{-1} , 1732 cm^{-1} and 1248 cm^{-1} were greatly diminished than that of HTG because of the degree of deoxygenation and dehydration during carbonization and after carbonization. The intensity of C=C and C-O stretching vibration was weakened after carbonization, indicating that lignin was partially decomposed (Fan et al., 2018).

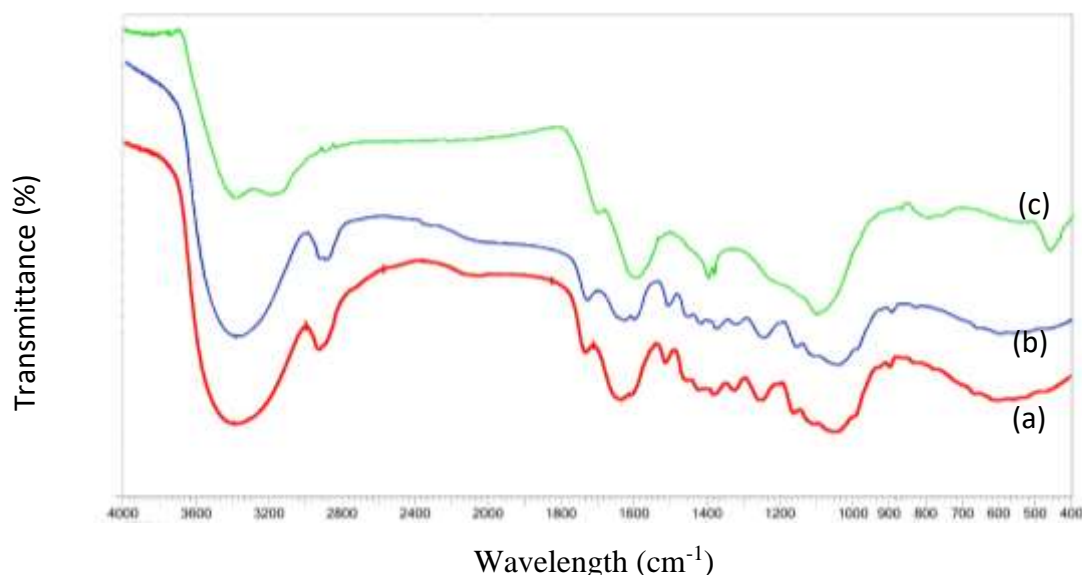


Figure (9) Fourier Transform Infrared (FTIR) Spectra for (a) Raw Grass, (b) HTG (c) Biochar

Conclusion

Hydrothermal treatment is an environmentally friendly method and it can discharge zero net waste. In this research, the potential bioenergy such as bioethanol and biochar from grass (*Pennisetum hordeoides* Lam.) was studied using hydrothermal treatment followed by fermentation of liquid fraction and by carbonization of solid fraction. Hydrothermal liquefaction of grass into soluble fractions of cellulose and hemicellulose was carried out at different temperatures and different times. The effective hydrothermal treatment at 110°C for

30 min gave for maximum amount of fermentable sugar without any further treatment method of biomass. It was also evident that changes in composition of grass such as cellulose, hemicellulose, and lignin contents were observed for raw grass and HTG. 2% by volume of bioethanol was accomplished by direct fermentation of liquid fraction using Baker's yeast. Carbonization of HTG at 500°C for 2 hr presented the maximum HHV and energy densification for the prepared biochar from grass. HHV (26.78 MJ/kg) of biochar prepared from grass can be compared with the HHV of lignite (~16.077MJ/kg), subbituminous coal (~23.26 MJ/kg) and bituminous coal (grade C) (~25.05MJ/kg) (Schweinfurth, 2009). Therefore, hydrothermal treatment method can support the process conversion of biomass into bioenergy.

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