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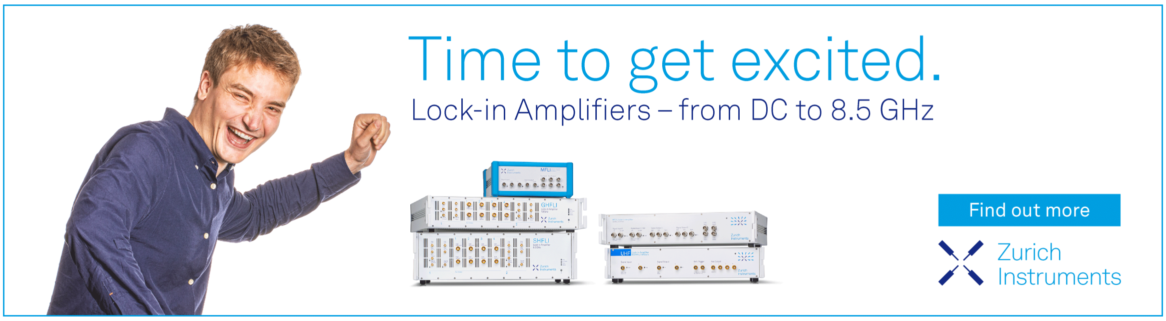

Bioenergy Derived from PABR Sludge through Hydrothermal Liquefaction: Effects of Temperature

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Abstract. Presently, clean and renewable energy sources, that don't have any adverse impacts on the environment, are the basic requirement for sustainable development. In this context, bioenergy generation from sludge through hydrothermal liquefaction (HTL) has been recognized as a promising approach since it reduces the waste volume, effectively utilizes the resources, recovers energy, and eliminates pathogens. In addition, this approach can solve both energy crisis and sludge management problems by introducing a sustainable pathway of bioenergy generation. The present work investigated the effect of temperatures on HTL products (biochar and biocrude) of sludge derived from a photo anoxic baffled reactor (PABR). The FTIR analysis indicated the biochar samples contain aliphatic amine, aromatic nitrile, and n-methyl amino compounds, which may be used to make fertilizers. On the other hand, biocrude consisted of hydrocarbon, esters, aldehydes, and ketone compounds. Additionally, the peaks for biocrude sample at 280 ℃ is similar to biocrude samples heated at 260°C with long-chain alkyl hydrocarbons (at wavenumber ranges from 2916-2936 cm^{-1}, 2845-2975 cm^{-1}, 1360-1390 cm^{-1}, 1415-1475 cm^{-1}), but the peak intensity for the biochar sample increased around 10 to 25% for all the functional groups due to the increases of temperature. So, the optimum thermal condition for producing high-quality biochar and biocrude could be achieved at 280 ℃ temperature. Thus, the sludge-to-energy approach would be a sustainable pathway for achieving the UN's sustainable development goal (SDG 7).

INTRODUCTION

In recent years, an alarming issue for the survival of future generations is the rapid growth of the economy which leads to high energy consumption, greenhouse gas emission, environmental pollutions and consequently the climate change that leads to global warming (Hasan et al., 2018; Kabir et al., 2022). The reserves of fossil fuel are depleting rapidly due to this intensive energy demand consumed by industry, transportation, and household (Pittman et al., 2011). This process of generating energy from fossil fuels is unhealthy to the environment. Considering this scenario, to fulfil energy requirements of the current and future era, developing renewable energy sources is an utmost need. In this context, the 'Waste to Energy' approach could unfold in a threefold beneficial way by diminishing waste and producing energy in a cost-effective way (Iqbal and Kang, 2021).

With the rapid growth of population and urbanization, huge amounts of waste are generated which becomes a big challenge and will be a nightmare for developing countries where there is inadequate waste/sludge/wastewater management system (Liu et al., 2021). However, faecal sludge or sludge containing wastewater when disposed to open places or to natural water bodies in an inappropriate manner, hampers the environment tremendously. So, the faecal sludge as well as wastewater management is a noteworthy concern in developing countries that impedes them to obtain sustainable development goals (SDGs) (Khalekuzzaman et al., 2018b). Within this context, management of wastewater/sludge by using profitable biomass conversion techniques has significant health and environmental benefits, and is fundamental for the sustainable development of all communities, considering that wastewaters contain usable resources, energy and nutrients (Ludovico Spinosa and Puja Doshi, 2020).

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Wastewater treatment by different baffled reactors has drawn attention other than the conventional aerobic processes because of zero consumption of energy during the treatment process (Khalekuzzaman et al., 2018a). Recently, Hasan et al. 2021 has referred to a specially designed Photo-anoxic baffled reactor (PABR) from which a microalgal-bacterial consortium can be harvested. The harvested microalgae-bacteria (sludge) during the wastewater treatment process has paved an energy rejuvenating way for bioenergy generation. Microalgae (MA) are now considered 3rd-4th generation biomass with the major advantages of organics-rich lignin free cellular structure which can easily be converted to bioenergy (Hoque et al., 2020). Moreover, Cultivation of microalgae brings along some advantages as they use the nutrients from the treated effluent of PABR for their growth (Mehedi Hasan et al., 2021). Furthermore, MA is not yet standalone biomass for energy conversion since the production and harvesting of MA is energy intensive and expensive processes (Islam et al., 2022). On the contrary, sludge extracted from the PABR effluent, can be a potential replacement of MA if it can be effectively converted to bioenergy in the form of biofuels like biochar and biocrude by hydrothermal liquefaction (HTL) (M. Hasan et al., 2021).

The HTL is a thermochemical conversion technique of wet biomass into biochar and biocrude oil (Suali and Sarbatly, 2012). There are mainly four types of thermochemical conversion which are pyrolysis, steam reforming, transesterification, and hydrothermal liquefaction (Goyal et al., 2008). Among all these processes HTL has secured its attention to the researchers during the past decade due to its several beneficial attributes over the other thermochemical approaches. Since the feedstocks, like microalgal biomass or sludge, have a high fraction of water around 80%-90%. On the other hand, traditional thermochemical processes, like gasification and pyrolysis, are not economically viable since they consume huge energy during the dry process of feedstock (Patil et al., 2008). However, the HTL process can escape this energy-consuming drying step which makes it more sustainable and economic in sludge operation. Primary requirement of HTL is water which behaves like a catalyst and solvent since it is in subcritical state for HTL reaction (Patrick et al., 2001). Other advantages are that HTL can be operated at moderate temperatures (200-374□C) and pressure (2-20 MPa) where pyrolysis requires higher temperature conditions (400-450□C) (Gollakota et al., 2018; Jazrawi et al., 2015). The HTL is the low-cost process which has operational flexibility and at the same time enhances the capability of getting high yield with critical attributes such as lesser heteroatomic content, formation of low biochar and energy recovery. Therefore, HTL may be used to produce bioenergy such as biocrude and biochar, and reduce the negative impacts on the environment associated with sludge.

Although several studies were carried out on the htl of sludge, microalgae, and other organic wastes. However, very little knowledge was available on bioenergy generation from pabr sludge. Since pabr sludge contains microalgal-bacterial consortium, it would be a novel prospect to analyze the thermal effects on htl products like biocrude and biochar. Hence, the present research objectives are: (1) characterization of biocrude and biochar driven from pabr sludge and compare them microalgal driven biocrude and biochar; (2) to investigate the effects of temperature.

METHODOLOGY

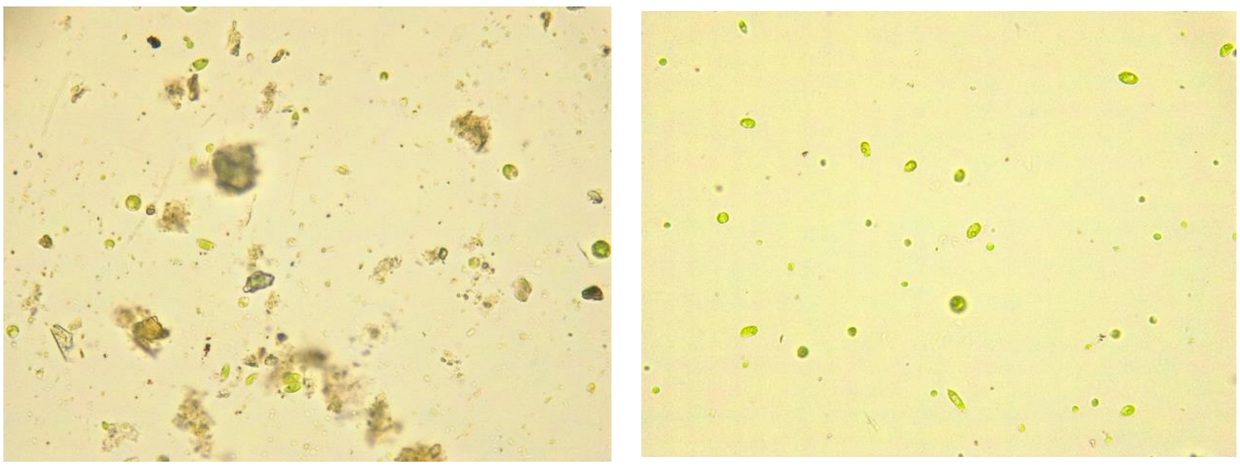
HTL is an appropriate pathway for converting wet wastes into bioenergy, such as biocrude and biochar. In the present study, three samples, in which two sludge samples and one microalgal biomass, were used for the HTL process at two different temperatures. However, microalgal biomass was used to compare the functional groups of biocrude and biochar derived from sludge samples.

Collection of Sludge

The sludge samples were collected from Photo Anoxic Baffled reactor (PABR) at chamber-1 (Ch-1) and chamber-2 (Ch-2). The dimensions of PABR and other operating parameters for wastewater treatment was described in the previous study (M. Hasan et al., 2021). However, PABR is very effective for removing both organic waste and nutrient, as microalgal-bacterial symbiosis act on it and the sludge accumulated at the bottom of each chamber is suitable for bioenergy generation. The microscopic view of microalgal-bacterial symbiosis in sludge samples is shown in Fig. 1 (a), and the proximate and elemental analysis are presented in Table 1.

Collection of Microalgae

Microalgal co-culture, dominated by Chlorella vulgaris and Scenesdesmus, was used in the present study which was cultivated in photobioreactor (PBR) made of locally available transparent plastic containers having capacity of 8 L. However, PABR effluent was used as a nutrients source for microalgae cultivation process. And the PBR is very effective for removing nutrients from PABR effluent and producing microalgal biomass which further used for bioenergy generation. The characteristics of PABR effluent and other operating parameters of microalgae cultivation and harvesting process was presented in the earlier study (Mehedi Hasan et al., 2021). The microscopic view of microalgal co-culture is shown in Fig. 1 (b) and the proximate and elemental analysis are presented in Table 1.



(a) (b)

FIGURE 1. Microscopic view at 40X magnification, (a) microalgal-bacterial symbiosis of sludge, and (b) microalgal biomass.

TABLE 1. Characterization of sludge and microalgal biomass.

Components	**Microalgae**	**Sludge**
Proximate Analysis		
Total solids (mg/L)	12.2 ± 0.6	13.5 ± 0.3
Moisture (mg/L)	87.8 ± 0.6	86.5 ± 0.3
Volatile solids (mg/L)	10.4 ± 0.4	11.1 ± 0.5
Ash content (mg/L)	1.8 ± 0.4	2.4 ± 0.5
Ultimate Analysis		
C (wt %)	23.2± 0.2	29.7 ± 0.3
H (wt %)	3.3 ± 0.1	3.9 ± 0.2
N (wt %)	3.9 ± 0.2	1.7 ± 0.1
O (wt %)	32.5 ± 0.5	29.7 ± 0.4

Hydrothermal Liquefaction Processes

The experiment was performed in a hydrothermal synthesis reactor, which made of 316 grade stainless steel with a capacity of 25 ml and working pressure 5 MPa. There is a ppl liner having capacity of 25 mL occupied inside the reactor. The purpose of ppl liner is to hold the microalgal biomass and sludge samples inside the reactor with high temperature and pressure. The heat inside reactor distribute equally due to uniform wall thickness (Khalekuzzaman et al., 2020).

In hydrothermal liquefaction processes, each experiment was used around 15 ml biomass into the reactor for bioenergy conversion. The reactor was then taken into the muffle furnace and cooked at two different temperatures (260 and 280 ºC) where retention time was 60 minutes and heating rate of 60 ºC /min. When the desired retention time was reached, the reactor was taken out from muffle furnace and kept it into the water bath for cooling at room temperature. In order to collect the final product, the gases stored at the reactor headspace were carefully released and dichloromethane (DCM) was added into the HTL reactor for extraction purposes (Islam et al., 2022). Then the liquefaction products collected into the conical tube and shacked them through a vortex to form a homogeneous mixture. However, in order to separate the solid and liquid phase, the conical tube was centrifuged at 4000 rpm for 10 min (Md. R. Hossain et al., 2022). And the phase separation of HTL products shown in Figure-3, where the aqueous phase stays at the top, biochar stays in the middle, and the organic phase dissolved in dichloromethane stays at the bottom of the tube.

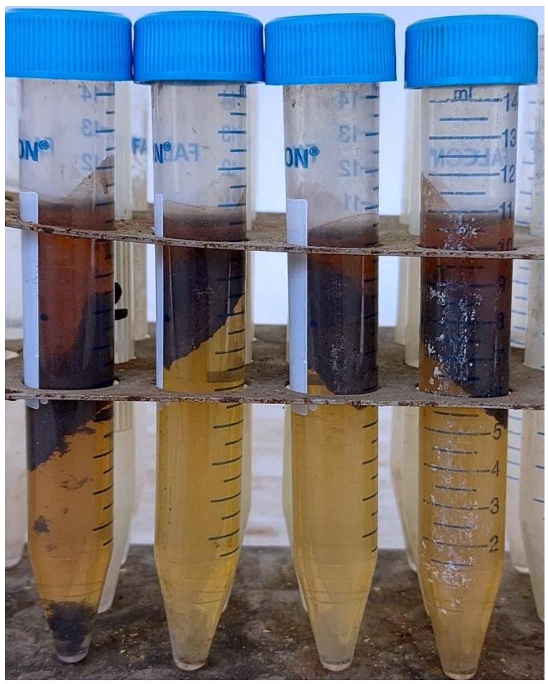


FIGURE 2. Phase separation of HTL products.

This research mainly focused on the obtained biochar and biocrude generated from PABR sludge and microalgae. So, the organic phase containing biocrude was separated by a pipette, moved into pre-weighed petri dishes, and kept in a desiccator for a few hours to evaporate DCM (M. R. Hossain et al., 2022). And the biochar samples were dried at 100 °C for at least 24 h before being weighed (He et al., 2018). Finally, FTIR analysis was carried out of the biochar and biocrude samples.

RESULTS AND DISCUSSION

Fourier Transform Infrared Spectroscopy (FTIR) Analysis

The distribution of functional groups existing in the biochar and biocrude were studied by FTIR spectroscopic analyses (Ansari et al., 2017). The peaks and vibrations (stretching and bending) found in the various wavelengths suggested the following active groups like C-H Stretch, $(CH_2)_4$-C, -CH_2/-CH_3 Stretch, N-H, C-O-C, C=C, C=O, C-N, and N=O were presented in the biochar and biocrude samples. However, it was observed from IR spectra that all samples were contaminated for water vapor (3440- 3950 cm^{-1}) and CO_2 contamination (at 2250 cm^{-1} to 2450 cm^{-1}).

After eliminating the water vapor and CO_2 contamination, IrAnalyzer software was used for manual baseline correction. Finally, the peaks of the spectrums were analyzed to determine the active functional groups.

Characterization of biochar

The biochar driven from microalgae and sludge samples at two different temperatures (260 ℃ and 280 ℃) was described by FTIR spectrums and shown in Fig. 3 and Table 2. However, the biochar from microalgal biomass at two different temperatures has shown almost similar peaks, but the absorbance intensity of the peaks is different. And the peak intensity of biochar at 280 ℃ is much higher than the of biochar at 260 ℃, which indicates that the quality of biochar is enhancing due to the increase of temperature. On the other hand, a contrasting result was observed for sludge-driven biochar, where the significant change was noticed on these peak intensities, which were around 2.5 times more intense for Ch-1 biochar at 260 °C than biochar at 280 °C. Moreover, the biochar generated from sludge samples (Ch-1 and Ch-2) at two different temperatures showed almost similar peaks, but the changes were noticed compared to biochar from microalgal biomass. And the absorbance intensity of the peak of different functional groups for sludge derived biochar was much stronger than the microalgal derived biochar (M. Hasan et al., 2021). The peak ranges from wavenumber 2900-2975 cm-1, indicating the functional groups of C-H alkyl (Jena et al., 2012), but it was totally absent in all the biochar samples in both temperatures.

All biochar samples derived from microalgae and sludge have shown the peaks wavenumbers 1500-1580 cm-1 and 1640-1690 cm-1, which representing the existence of N=O and C=C stretching of functional groups of p-nitrophenol compounds (Annenkov et al., 2015). However, the microalgal biochar samples have shown a strong peak at wavenumbers 1000-1100 cm-1 for Si-O/C-O, which indicating the presence of outer cell membrane (Si-O-C) of microalgal biomass (Mahapatra and Ramachandra, 2013). On the contrary, the sludge biochar samples have shown a medium peak for Si-O/C-O at wavenumbers ranges from 1000-1100 cm-1, indicating that the sludge samples collected from PABR might contain microalgal cells (Khalekuzzaman et al., 2020). Moreover, the sludge biochar samples have also shown peak for C-N stretching at wavenumbers 1250-1310 cm-1, whereas microalgal biochar samples have also shown peak for N-H stretching at wavenumbers 860-900 cm-1, which verified the presence of aliphatic amine, aromatic nitrile, and n-methyl amino compounds. Hence, it is suggested that the biochar may be applied to make fertilizers as it contains nitrogen compounds (Khalekuzzaman et al., 2019). Similarly, other researchers observed the same functional groups in the biochar samples from FTIR spectrums (Lu et al., 2018; Vardon et al., 2011).

TABLE 2. FTIR spectrums band assignments of biochar samples

Functional Groups	**Spectra range**	**Strength of Spectra ranges of biochar**					
		Microalgae		**Ch-1**		**Ch-2**	
	(cm^{-1})	**260 ºC**	**280 ºC**	**260 ºC**	**280 ºC**	**260 ºC**	**280 ºC**
C-H Stretch	2900-2975	Variable	Variable	Variable	Variable	Variable	Variable
C=O	1700-1755	Variable	Variable	medium	Variable	Variable	Variable
C=C, trans	1640-1690	Strong	medium	medium	Strong	Variable	Strong
N-H	1560-1640	--	--	--	--	--	--
N=O	1500-1580	medium	Strong	Strong	Strong	medium	Strong
C-H bending	1350-1480	medium	medium	Variable	Variable	Variable	Variable
C-N Stretch	1250- 1310	--	--	Variable	Variable	Variable	Variable
C-O, Si-O Stretch	1000-1100	Strong	Strong	medium	medium	medium	medium
N-H Deformation	860-900	Strong	medium	--	--	--	--

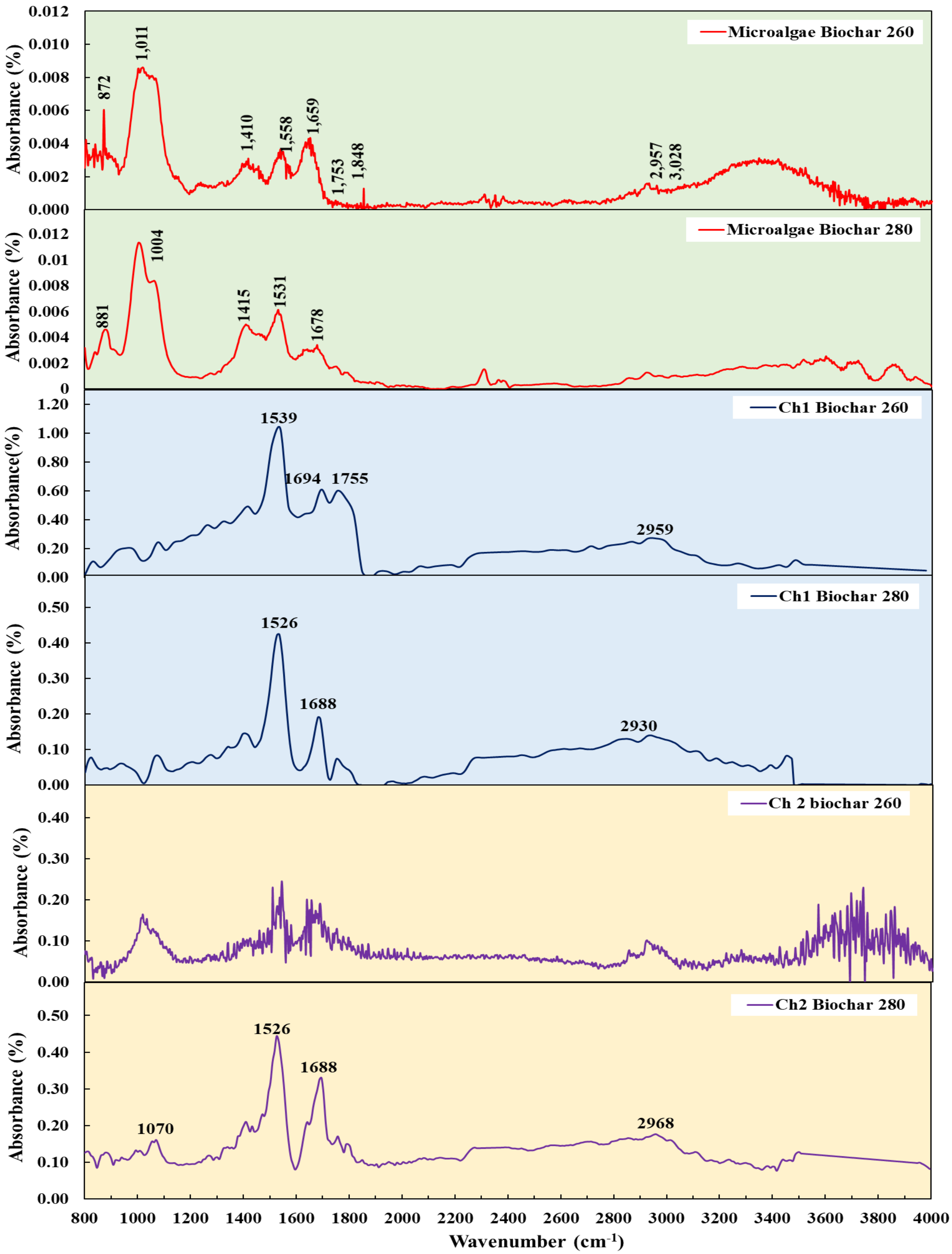


FIGURE 3. FTIR absorbance spectroscopy on biochar samples after hydrothermal liquefaction.

Characterization of biocrude

The FTIR analysis of biocrude samples driven from microalgal biomass and sludge at two different temperatures (260 ℃ and 280 ℃) are presented in Fig. 4 and Table 3. Both samples have demonstrated different strength of wavenumbers corresponding to different functional groups. All the biocrude samples, except microalgal biocrude at 260℃, have shown strong strength peaks at wavenumber 2916-2936 cm-1 for (CH2)4-C and at wavenumber 2845-2975 cm-1 for C-H stretch, which indicating the biocrude is suitable for biodiesel conversion (Khalekuzzaman et al., 2019). However, the absorbance (at 2927 cm-1) of sludge biocrude (Ch-1) at 260℃ is 42 times more intense with respect to microalgal biocrude at 260℃. At the same time, sludge biocrude (Ch-1) at 280℃ is 32 times more intense when compare it with microalgal biocrude at 280℃. In addition, the peak for COO (at wavenumber 1720-1770 cm-1) suggested that aldehydes, ketones and aliphatic esters may exist in both biocrude samples (Ponnuswamy et al., 2013). The IR spectrum showed C=O stretching (at 1640-1800 cm-1), which indicating the presence of alcohol, phenol and -COOH group in the biocrude samples (Guo et al., 2015). Finally, the results suggested that the sludge biocrude is better than the microalgal biocrude.

The peaks for Ch-1 biocrude sample at 280°C is similar to biocrude samples heated at 260°C with long chain alkyl hydrocarbons (at wavenumber ranges from 2916-2936 cm-1, 2845-2975 cm-1, 1360-1390 cm-1, 1415-1475 cm-1), but the peak intensity for the biochar sample increased around 10 to 25% for all the functional groups due to the increases of temperature. And similar result was observed for Ch-2 biocrude sample. However, the peaks at 1640-1800 cm-1, and 1070-1135 cm-1 represented the C=O, and C-O stretching of aromatic carboxylic acid (Annenkov et al., 2015). Many researchers were observed similar chemical compounds in the biocrude sample (Kovac et al., 2002; Vardon et al., 2011).

TABLE 3. FTIR band assignments for biocrude samples

Functional Groups	**Spectra range**	**Strength of Spectra ranges of Biocrude**					
		Microalgae		**Ch-1**		**Ch-2**	
	(cm^{-1})	**260 ºC**	**280 ºC**	**260 ºC**	**28 0ºC**	**260 ºC**	**280 ºC**
$(CH_2)_4$-C	2916-2936	Medium	Strong	Strong	Strong	Strong	Strong
C-H Stretch, Alkyl	2845-2975	Medium	Strong	Strong	Strong	Strong	Strong
COO(Esters)	1720-1770	Strong	--	Strong	Strong	Medium	Medium
C=O	1640-1800	Variable	Variable	Variable	Variable	Variable	Variable
N=O	1500-1580	--	Strong	--	--	--	--
CH_2/CH_3 Stretch	1415-1475 1360-1390	Variable	Strong	Strong	Strong	Strong	Strong
C-O-C	1100-1300	Strong	Medium	Medium	Medium	Variable	Variable
C-O, Si-O Stretch	1070-1135	Strong	Strong	Strong	Strong	Strong	Strong
C-S=O	1020-1060	Strong	Variable	Strong	Medium	Medium	Strong

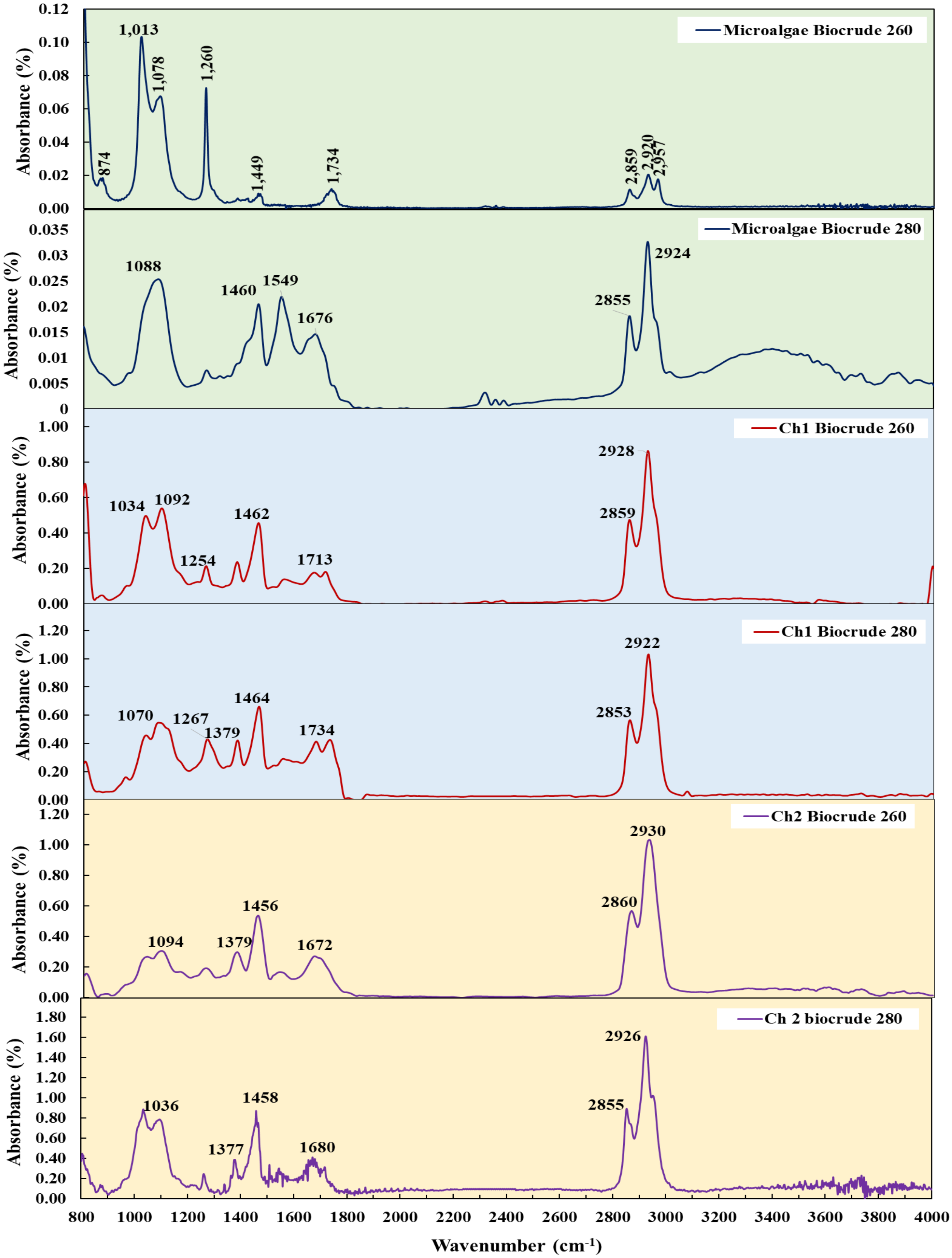


FIGURE 4. FTIR absorbance spectroscopy of biocrude samples after hydrothermal liquefaction.

CONCLUSIONS

This study found that the sludge biocrude quality obtained at 280 °C was better than that of the sludge biocrude obtained at 260 °C. The peak intensity for the biochar sample increased around 10 to 25% for all the functional groups due to the increase in temperature. On the other hand, a contrasting result was observed for sludge-driven biochar, where the significant change was noticed on these peak intensities, which were around 2.5 times more intense for ch-1 biochar at 260 °C than at 280 °C . The FTIR analysis indicated the biochar consisted of an aliphatic amine, aromatic nitrile, and n-methyl amino compounds, and biocrude consisted of hydrocarbon, esters, aldehydes, and ketone compounds. This study also concludes that the PABR sludge biocrude is better than the microalgal biocrude. Thus, the sludge-to-energy approach would be a sustainable pathway for achieving the UN's sustainable development goal (SDG 7).

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