

STUDY OF BIOOIL PRODUCTION FROM SEWAGE SLUDGE OF A MUNICIPAL WASTEWATER TREATMENT PLANT BY USING HYDROTHERMAL LIQUEFACTION

by

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To overcome the problem of rapid depletion of natural energy reserves and consequent pollution caused by them, this work explored the possibility of utilizing sewage sludge biomass to produce biooil using hydrothermal liquefaction pathway. In this study, effect of different reaction parameters such as reaction temperature, residence time, and sludge-to-water ratio on solid biomass conversion and bioyield and its higher heating value were investigated. Although maximum conversion of (99.7%) and highest biooil yield (22.01 wt.%) was achieved at 330 °C, however optimum temperature was chosen as 300 °C which produced conversion efficiency and yield of biooil very close (98.07% and 21.5 wt.%, respectively) to what was obtained at 330 °C as lower temperature is beneficial for overall economy of the process. Similarly, a residence time of 60 minutes and sludge-to-water ratio of 1:6 was screened to be producing optimized yield of biooil. The higher heating value of different fractions biooil was much improved (30.18 MJ/kg of acetone phase and 38.04 MJ/kg of dichloromethane phase) as compared to that of raw feedstock (12.74 MJ/kg). Carbon balance performed on the products indicated that maximum amount of carbon went to biooil phase (53.4 wt.%). However, a significant portion of carbon was lost (33.9 wt.%) due to the limitation of experiments at lab scale which involves evaporation and drying to reach final products. The Fourier transform infrared spectroscopy spectral analysis of different biooil phases showed that it was mainly made up of alcohols, alkane, ketones, aldehydes and carboxylic acids.

Key words: hydrothermal liquefaction, sewage sludge, biooil, water soluble oil, biochar, higher heating value

Introduction

Extensive use of natural reserves of fossil fuels results into not only rapid depletion of these resources but also causes day-by-day increase in environmental pollution. At the same time, massive increase in population, urbanization and industrialization also result in increase

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in energy demand [1]. As estimated, population of world, by 2050, will be 9 billion and almost 99% of this increase is expected to be coming from developing countries [2]. This rapid growth in population implies a multifold increase in energy consumption in these areas and hence the quest to find alternative sources of energy is even more pressing in future. These alternatives must not only be abundant in terms of sustainability but also renewable (having positive effects on the environment). In this context, waste biomass seems to be promising raw material for energy production as the utilization of waste biomass serves the two-pronged strategy of producing alternative energy on one side and on the other hand, efficiently handling the waste material which is anticipated to rise with population growth.

Sewage sludge (SS), a waste biomass, poses serious threats to environment if not handled properly. For example, considerable amounts of chemicals including sulfur, chlorine, nitrogen, heavy metals present in SS may cause eutrophication [3]. Human sewage, owing to high organic content, is one of the main reasons for pathogenic bacteria and viruses in seawater which disturb aquatic life and ultimately impact human health when these infected seafood is consumed by humans [4]. The SS contains high amounts of organics (including carbohydrates, lipids, proteins, *etc.*) and is an ideal feedstock to be used for biofuel production [5]. It is a biomass which is obtained as a waste from wastewater treatment plant and contains a high percentage of moisture content. Urbanization, mass migration and exponential growth in housing sector causing a continuous increase in sludge production [6], and hence its effective utilization is inevitable.

In Pakistan, open fields are used for rural waste and sewerage system is utilized for disposal of urban excreta, whereas the remaining waste deposited along roadside, into water-ways, or deposited as solid waste [7]. There are many environmental problems associated with these practices such as bad odor, production of secondary pollutants *i.e.* dioxins during incineration or burning of waste, *etc.* These evil practices further add to the existing environmental problems connected with this sludge and hence increase problem severity.

Production of fuels from this waste biomass as a potential replacement of conventional fossil fuels would not only be a low cost alternative but also renders an environmentally friendly as well as effective utilization of this waste [8]. Direct combustion and similarly pyrolysis are not the options for such a biomass because of its very high moisture content. However, a thermal conversion process of biomass into energy dense product in aqueous media, known as hydrothermal conversion process, has been the subject of many studies on SS [9].

Supercritical water gasification reaction, being on commercial scale, does not create products in liquid state or solid state and hence the existing infrastructure for solid and liquid fuel utilization cannot be used [10]. Hydrothermal liquefaction (HTL) of SS produces a range of products in different phases with a focus to produce liquid product. However, large quantities of solid (consisting of biocoke and ash) and gaseous products are also produced hence giving a variety of products.

Although there has been number of studies performed on HTL of SS in different parts of the world, as mentioned previously, yet the composition of SS varies around the world depending upon the eating and living habits of inhabitant [11]. There is not a single study on the SS liquefaction in Pakistan, as per author's knowledge, which demands not only thorough investigation of the SS produced in Pakistan, but also the understanding of its subsequent liquefaction behavior.

Current study focuses on utilization of sludge obtained from a local municipal sewage treatment plant in Pakistan. After comprehensive analysis of raw SS feedstock, HTL was performed with varying conditions of temperature, residence time, and biomass-to-solvent ratio

in sub-critical regime of water. Conversion of raw SS and liquid product selectivity was better than most of the studies performed at similar conditions of variable parameters [5]. Liquid products were characterized using proximate analysis, elemental analyzer, FTIR, and total organic carbon analyzer. Carbon balance was also reported to show the total carbon lost during the liquefaction process as volatiles. Hence, this study serves as a foundation for future studies on HTL of SS produced in Pakistan.

Materials and methods

Collection and preparation

The raw SS sample in wet form was collected from a local municipal sewage treatment plant (from the wastewater treatment plant of Bahria Town, Rawalpindi, a city in Pakistan). Raw SS (having 78% moisture) was dried in sunlight in open trays by covering them with a thin cloth to avoid dust accumulation. Dried SS was stored in sealed polythene bags for further use.

Characterization of sewage sludge

Moisture content of sludge sample was measured by using ASTM E871 method, ash content using ASTM E1755-01, volatile matter using ASTM E-872 and fixed carbon is the resultant of the summation of percentage moisture, ash, and volatile matter subtracted from 100. Ultimate analysis of raw SS sample was performed using CHNS analyzer (FLASH 2000 CHNS/O Analyzer by ThermoFisher Scientific) and based on the elemental analysis, higher heating value (HHV) which was calculated using DIN 51900 standard. The results of proximate and ultimate analysis of SS sample are shown in tab. 1.

Table 1. Proximate and ultimate analysis and HHV of raw SS sample

Proximate analysis [wt.%]							
Moisture		Ash		Volatile matter		Fixed carbon	
8.82		40.91		43.18		7.8	
Ultimate analysis [wt.%]					HHV [MJkg ⁻¹]		
C	H	N	S	O	12.74		
31.09	4.8	13.94	0.74	49.41			

Hydrothermal liquefaction process

Dried SS sample (as per standard mentioned previously) was put into a mini bench top reactor (4560 Parr Instrument Company) to perform HTL experiments batch wise. Schematic depiction of the experimental procedure is shown in fig. 1. The main parts of HTL system includes reactor vessel with volume 1 L, stirrer, pressure and temperature gauges. In a typical experimental run, measured amount of distilled water and dried sludge was loaded into reactor. Reactor was sealed and purged with N₂ after loading and pressurized with N₂ to 1.0 MPa so that the residual air inside reactor is removed. The experiments were performed at different reaction temperatures (270 °C, 300 °C, and 330 °C) for different sludge to moisture content ratios (1:2, 1:4, and 1:6) and for three different residence times (30 minutes, 60 minutes, and 120 minutes). As the temperature was raised, pressure of the reactor increased due to vapor pressure of solvent as well as thermal breakdown of larger molecules of feedstock to smaller units. After the

desired residence time at set temperature, heating was stopped, the reactor was quenched with cold bucket of water (to bring temperature to about 100 °C) and then left to cool down to room temperature (about 45 minutes). It is important to mention that the heating and cooling time of the reactor was not included in the residence time. After cooling of the reactor, gases were vented before opening the autoclave.

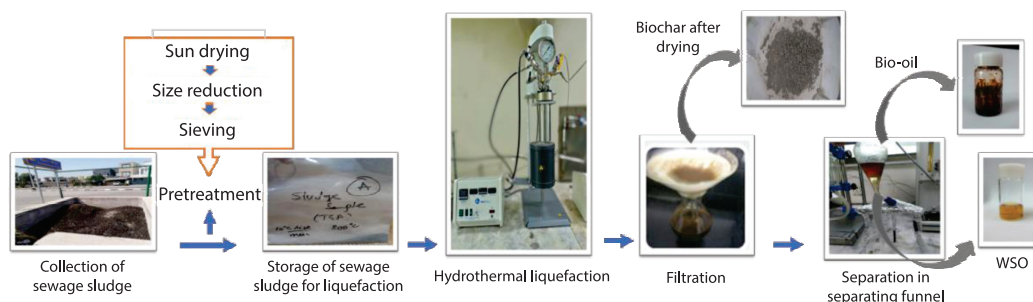


Figure 1. Schematic diagram of experimentation stage showing step by step procedure adopted for this study

Products separation and characterization

After venting the reactor, it was opened, and the mixture of solid and liquid products was separated using Whatman No. 41 filter paper. The solid cake left on the filter paper was rinsed with acetone to separate organic phase present in solid residue and dried in the drying oven at 105 °C till constant weight. Filtrate was taken to separating flask so that organic oil phase is separated from aqueous phase having water-soluble oil (WSO) dissolved in it. The two-phases are separated using liquid-liquid extraction. The reactor was rinsed using dichloromethane (DCM) and residual products (liquid and solid) was added to separating flask. To have a maximum separation and to standardize the process, a time of 1 hour was provided before the phases were collected separately. Top layer consisting of aqueous phase with WSO in it was dried in drying oven at 105 °C till constant weight and the bottom layer having organic fractions dissolved in DCM was dried at 70 °C. Residual mixture of oil which adhered to the components of reaction was finally washed with acetone and mixed with the organic fraction for maximum recovery of biocrude.

Conversion of the solid SS into liquid and solid products and overall losses were measured using:

$$\text{Yield of bio-oil/WSO [wt.\%]} = \frac{\text{Mass of daf bio oil/WSO}}{\text{Mass of daf sewage sludge}} \times 100 \quad (1)$$

$$\text{Yield of biochar [wt.\%]} = \frac{\text{Mass of daf solid residue}}{\text{Mass of daf sewage sludge}} \times 100 \quad (2)$$

$$\text{Losses [wt.\%]} = 100 - (\text{sum of yields of bio-oil, WSO and solid residue}) \quad (3)$$

Biooil and WSO were analyzed in aforementioned elemental analyzer, and HHV of the respective phases were calculated using DIN 51900 standard. Carbon content measured via elemental analyzer was used for carbon balance. Chemical functionalities of the products and raw SS were obtained using a Perkin Elmer Spectrum Fourier transform infrared FTIR spectrometer with a resolution of 4 1 per cm in the range of 4000-400 1 per cm.

Results and discussion

Effect of different reaction parameters

Effect of reaction temperature

Most important parameter of HTL process is reaction temperature as it is critical parameter in controlling ionic properties of water which helps dissolving the biomass components. Ionic product of water increases with the increase in temperature and reaches its maximum value at 300 °C [12] and this is one of the reasons for achieving high conversion at 300 °C in many studies [13, 14].

The temperature values investigated in this study are 270 °C, 300 °C, and 330 °C, for fixed sludge-to-water (ss-to-water) ratio of 1:6 and a residence time of 60 minutes (these values of ss-to-water ratio and residence time were reached after screening study). With the increase in reaction temperature from 270-300 °C, yield of biooil increased from 17-21.5 wt.%. A further increase in temperature to 330 °C has increased biooil yield marginally to 22.01 wt.% as shown in fig. 2.

Conversion of the solid biomass also increased with temperature as evident by the decrease in biochar yield. From 270-300 °C, yield of biochar significantly decreased from 21.61-1.5 wt.% and further increase in temperature decreased the biochar yield to 0.62 wt.%.

Effect of temperature on the conversion of solid biomass can be explained considering the two observations. First is that the maximum conversion in this study, like the previous studies, is obtained at 300 °C which emphasizes on the ability of water to perform as an efficient solvent in dissolving solid biomass and reaction intermediates which promotes the liquid product yield. Aforementioned dissolving power of water at high temperature is associated with the ionic product of water. Second is that most of the organic part of the biomass had been converted to products at 300 °C as indicated by the very small amount of biochar yield. Further increase in temperature to 330 °C was not expected to improve biooil yield. However, it helped us improve our understanding of the reaction mechanism. For example, although the decrease in WSO yield from 14-10 wt.% and the decrease in yield of biochar from 1.5-0.62 wt.% was observed as the reaction temperature was increased from 300-330 °C, the yield of biooil however increased slightly from 21.5-22.01 wt.%. This was because the total losses during the reaction, which included gases and lighter fractions that could not be recovered during separation and drying process, increased from 63-67.37 wt.% as shown in fig. 2. The ability of water to perform as a solvent at increased temperature is used to depolymerize larger molecules of solid biomass, as well as to dissolve the reaction intermediates which helps retarding the recombination reaction, has also been reported earlier [15].

Effect of reaction time

Previous studies on HTL exhibit that with increase in residence time, conversion of the feedstock initially increased and after certain increase of residence time conversion started decreasing. Initially biomass is dissolved in the solvent at high temperature and gets converted

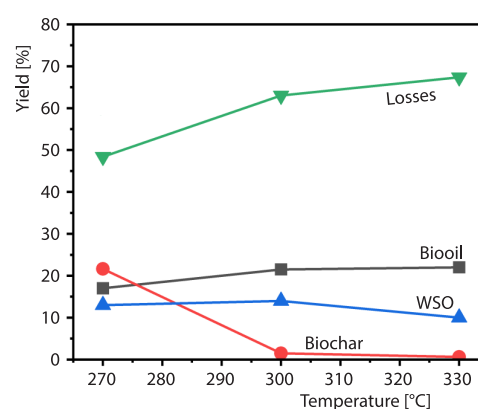


Figure 2. Effect of reaction temperature on yield of biooil, biochar, WSO, and losses (reaction time: 60 min, ss-to-water ratio: 1:6)

into biooil, biochar, gas, and reaction intermediates. As the reaction time is increased, lack of reactivity of solvent with reaction intermediates plays its part and repolymerization of the reaction intermediates forms biochar and hence a decrease in biooil yield. At the same time, some biooil components also get decomposed which results a decrease in biooil yield and an increase in gas yield [16, 17].

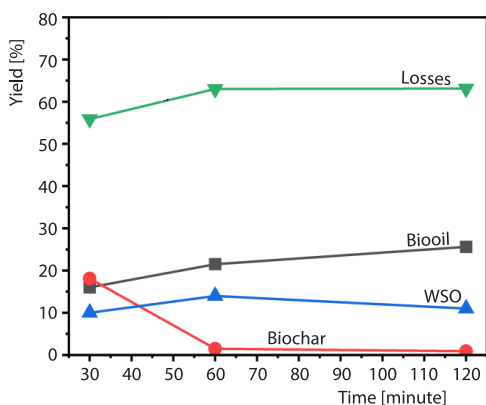


Figure 3. Effect of residence time on yield of biooil, biochar, WSO, and losses (temperature: 300 °C, ss-to-water ratio: 1:6)

tion reaction, which causes the increase in biochar yield, was not significant even up to 120 minutes. Thus, the increase in biooil yield came from the decrease in WSO yield which was 14 wt.% at 60 minutes and decreased to 11 wt.% at 120 minutes reaction time. The highest yield for all reaction times was of losses which consisted of gas which was not quantified in this study as well as lighter compounds which evaporated during post liquefaction protocol including separation and drying. Losses also increased with the reaction time up to 60 minutes (from 55.88 wt.% at 30 minutes to 63 wt.% at 60 minutes) and then became stagnant as reaction time was increased to 120 minutes which indicates that the decomposition of raw material did not occur significantly after 60 minutes reaction time.

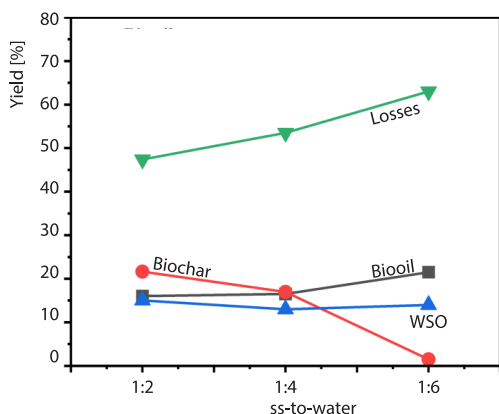


Figure 4. Effect of ss-to-water ratio on yield of biooil, biochar, WSO, and losses (temperature: 300 °C, reaction time: 60 minutes)

In this study, the effect of residence time was examined at a fixed temperature (300 °C) and ss-to-water ratio (1:6). As the reaction time was increased from 30-60 minutes, conversion of the biomass increased which was indicated by the increase in yield of biooil (16 wt.% at 30 minutes to 21.5 wt.% at 60 minutes) and that of decrease in yield of biochar (18.12 wt.% at 30 minutes to 1.5 wt.% at 60 minutes) as shown in fig. 3.

However, with further increase of reaction time to 120 minutes, as opposed to previous studies where conversion decreased after a certain increase in reaction time, conversion did not decrease as yield of biochar remained constant (63.1 wt.%) and yield of biooil increased (25.01 wt.%). This shows that the repolymeriza-

Effect of sludge-to-water ratio

The effect of ss-to-water ratio on HTL products yield and conversion efficiency was investigated with fixed reaction temperature of 300 °C and reaction time of 60 minutes. As SS-to-water ratio was increased from 1:2 to 1:4 to 1:6, the conversion efficiency increased steadily as depicted by the decrease in biochar yield from 21.61 wt.% (at 1:2) to 16.98 wt.% (at 1:4) to 1.93 wt.% (at 1:6) and yield of WSO phase remains almost unchanged. The yield of biooil increased, respectively from 16 wt.% to 16.5- 21.5 wt.%. Similar results have been reported by Yang *et al.* [15]. In HTL, SS-to-water ratio is one of the

important parameters as it defines the concentration of solid in the media to be used as a solvent. As SS-to-water ratio was increased from 1:2 to 1:4 to 1:6, the amount of water increased and solid concentration decreased, which promoted solvolysis reaction leading to hydrolytic degradation of SS and thereby conversion efficiency increased. Since dissolution plays an important role to initiate subsequent degradation, at high solid concentration of SS, the relative interactions among fragments of reactants became less effective, thus reducing the dissolution of SS components [18].

The effect of SS-to-water ratio on yield of biooil, biochar, WSO, and losses during the reaction at temperature of 300 °C and at residence time of 60 minutes has been presented in fig. 4.

Ultimate analysis and HHV

Ultimate analysis of the HTL products and subsequent HHV for reaction temperature of 300 °C, reaction time of 60 minutes and SS-to-water ratio of 1:6 has been presented in tab. 2. The HHV of the product biooil increased as compared to raw feedstock because of improvement in elemental composition.

Table 2. Ultimate analysis and HHV of raw feedstock, different phases on biooil and biochar (obtained at temperature: 300 °C, reaction time: 60 minutes, ss-to-water ratio: 1:6)

Sample	Ultimate analysis [wt.%]					HHV [MJkg ⁻¹]
	C	H	N	S	O	
Raw SS	31.09	4.80	13.94	0.74	49.41	12.74
Acetone phase	60.79	8.41	11.99	0.51	18.30	30.18
DCM phase	75.66	9.99	7.62	0.26	6.47	38.04
WSO phase	24.12	6.83	10.28	1.85	56.92	12.12
Biochar	27.66	2.99	3.81	0.36	65.18	7.04

Carbon and hydrogen content of raw feedstock was low (31.09 wt.% and 4.80 wt.%) and oxygen content was high (49.41 wt.%). However, carbon content of different biooil phases was higher (acetone phase 60.79 wt.% and DCM phase 75.66 wt.%) while that of WSO phase was lower (24.12 wt.%) than raw feedstock (31.09 wt.%). Similarly, oxygen content of acetone phase and DCM phase was low (18.30 wt.% and 6.47 wt.%, respectively) while that of WSO phase was higher (56.92 wt.%). The hydrogen content of all biooil phases was higher (acetone phase 8.41 wt.%, DCM phase 9.99 wt.% and WSO phase 6.83 wt.%) as compared to that of raw feedstock (4.80 wt.%). This increase in carbon and hydrogen content and decrease in oxygen content affects the HHV a great deal as compared to nitrogen and sulfur content which do not impact HHV significantly as per DIN 51900 standard used for the calculation of HHV. Liquid-liquid extraction process employed in this study divided the biooil into oxygen rich WSO oil phase which created an affinity with water due to the presence of hydrogen bonding and the remaining biooil (DCM phase) had the lowest oxygen and hence highest carbon containing species and hence the highest HHV. The intermediate phase which was solubilized in acetone during rinsing of the reactor and other parts had intermediate amount of carbon and oxygen content and hence intermediate HHV. Carbon content of biochar is very low showing almost complete conversion of the organic part into different products. Very high oxygen content of biochar is a bit strange but that may be because the amount of oxygen has been calculated based on difference method.

Carbon balance

To understand the distribution of carbon content into different products during HTL, determination of amount of carbon was performed on all three products (biooil, biochar, and WSO) and carbon losses was calculated by subtracting the sum of all three product from the feedstock carbon content. Overall carbon balance for the optimized reaction conditions (reaction temperature of 300 °C, residence time of 60 minutes, and SS-to-water ratio of 1:6) has been presented in fig. 5 which shows that the highest amount of carbon percentage (53.4 wt.%) was found in biooil phase. This indicates that HTL of SS at the reaction conditions optimized

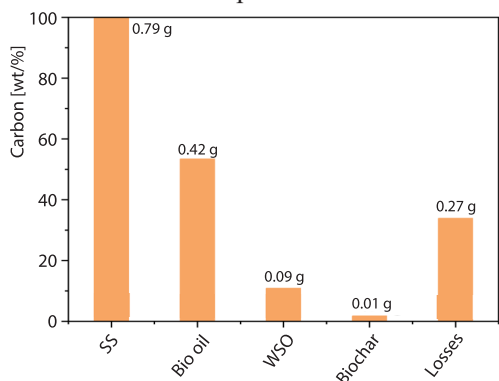


Figure 5. Distribution of carbon present in raw ss into biooil, WSO) biochar, and losses (temperature: 300 °C, reaction time: 60 minutes, ss-to-water ratio: 1:6)

in this study shows promising results. However, carbon content of the losses fraction was found to be significant (33.9 wt.%) during this HTL reaction. Since the yield of losses fraction was huge (63 wt.%), the carbon content of this fraction is not as big as it seems. Moreover, losses fraction is only found in laboratory experiments (in post liquefaction handling of the products including separation and drying), industrial scale HTL (because of the precise control and recycling of the solvents) is not expected to have such a high yield of losses fraction. The lowest percentage of carbon was found to be in biochar (1.5 wt.%) which shows almost complete conversion of carbon from the SS to different products.

The FTIR analysis

Figure 6 shows the FTIR spectra of the raw SS, biooil obtained via DCM rinsing as well as via acetone rinsing and the WSO phase produced at temperature of 300 °C, reaction time of 60 minutes and ss-to-water ratio of 1:6. Raw SS showing the presence of many different types of functional groups where carbon is associated with hydrogen, with oxygen, with nitrogen, and with other carbon. Similarly other functional groups like –OH (1418 1 per cm), NO (1540 1 per cm), and S = O (1015 1 per cm) were also found indicating heterogenous nature of the raw feedstock. However, in different phases of biooil (DCM phase, acetone phase and WSO), nitrogen containing and sulfur containing species were not found which implies that these components might have ended up in biochar as reported earlier [19] or it might have been lost during separation process. The bands around 2840-2950 1 per cm in DCM and acetone phases correspond to the stretching vibration of alkyl C–H. However, the broad band at 3262 1 per cm in WSO phase represent stretching vibrations of –OH groups. This shows the segregation of the biooil into hydrophobic (DCM phase with the presence of C–H groups) and hydrophilic (aqueous phase with the presence of –OH groups) while separation was performed in separating funnel. The bands at 1635 1 per cm and 1652 1 per cm were consistently found in raw feedstock and in all phases of biooil correspond to the stretching vibrations of C=C groups indicate the presence of C6 rings [20]. The small peak at 1157 1 per cm and 1097 1 per cm in DCM phase and at 1166 1 per cm in acetone phase can be assigned to C–O–C ether groups which was not observed in WSO [21, 22].

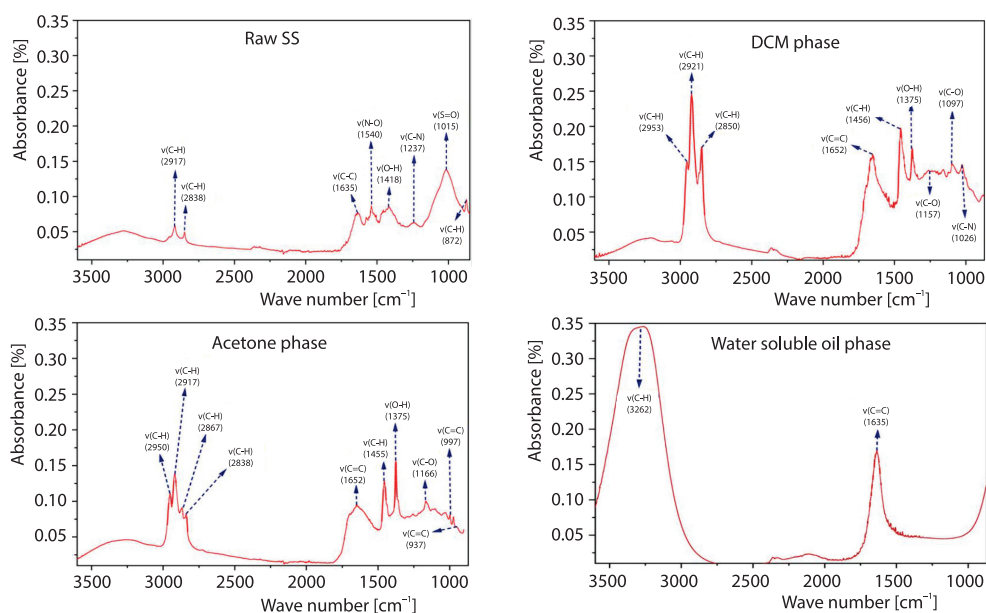


Figure 6. The FTIR spectra of raw SS and different biooil fractions including DCM phase, acetone phase and WSO phase (obtained at temperature: 300 °C, reaction time: 60 minutes, ss-to-water ratio: 1:6)

Conclusion

This study emphasizes the utilization of SS as a potential biomass for biofuel production as it helps solving the issues of energy production using renewable feedstocks as well as efficient disposal of SS simultaneously. This work investigated the biooil production potential of SS as a waste biomass using HTL as a thermal conversion route. The SS in this work has been found to be a favorable feedstock for biooil production via HTL mainly because of its very high inherent moisture content. Through screening of different reaction parameters, reaction temperature of 300 °C, a residence time of 60 minutes and SS-to-water ratio of 1:6 produced high quality biooil in terms of its HHV with almost complete conversion (98.07%) and high yield (21.5 wt.%). Product separation process which included liquid-liquid extraction and drying of biooil increased the losses fraction up to 62.57 wt.% having carbon content of 33.9% of the original feedstock. Since this product separation protocol is not adopted at large-scale production, these losses are not expected there.

Contribution

Authors Hassan Zeb and Muhammad Asif Hussain equally contributed to this work.

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