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High quality fuel from food waste - investigation of a stepwise process from the perspective of technology development

Ke Yin*, Ling Li, Apostolos Giannis, Piyarat Weerachanchai, Bernard J.H. Ng, Jing-Yuan Wang

Residues and Resource Reclamation Centre (R3C), Nanyang Environment and Water Research Institute, Nanyang Technological University, 1 Cleantech Loop, CleanTech One, Singapore 637141, Singapore

*Corresponding author. Tel: +6790 4100, Fax: +65 67927319; E-mail: yinke@ntu.edu.sg

Abstract

A stepwise process (SP) was developed for sustainable energy production from food waste. The process was comprised of hydrothermal treatment followed by oil upgrading. Synthetic food waste (SFW) was primarily used as feedstock in the hydrothermal reactor under subcritical water conditions. The produced hydrochars were analyzed for calorific value (17.0 – 33.7 MJ/kg) and elemental composition indicating high quality fuel comparable to coal. Hydrothermal carbonization (e.g. 180°C) would be efficient for oil recovery (> 90%) from food waste, as compared to hydrothermal liquefaction (320 °C) whereby lipid degradation may take place. The recovered oil was upgraded to biodiesel in a catalytic refinery process. Selected biodiesels i.e. B3 and B4 were characterized for density (872.7 kg/m³ and 895.5 kg/m³), kinematic viscosity (3.115

cSt and 8.243 cSt), flash and pour point (30 °C and >126 °C), micro carbon (0.03% and 0.04%), sulfur (both <0.0016%) and calorific value (38917 J/g and 39584 J/g), suggesting similar quality to commercial biodiesel. Fatty acid methyl ethers (FAMES) content was further analyzed to assess the influence of hydrothermal treatment in biodiesel quality, indicating the limited impacts. In overall, the SP provides a promising alternative for sustainable energy recovery through high quality biofuel and hydrochar production.

Keywords: Food waste, waste to energy, hydrochar, biodiesel, FAMES

1. Introduction

Huge amount of food waste (FW) is generated annually worldwide. It has been estimated that one-third of food produced (around 1.3 billion tonnes) is wasted during production, processing, distribution, consumption, and disposal [1]. In Singapore, the amount of food waste was increased by 50% from 542,700 tonnes in 2006 to 788,600 tonnes in 2014 [2]. The main challenge of food waste management is its rapid putrescibility under ambient conditions due to high moisture content and biodegradable nature [3].

Currently, food waste is managed through various methods such as recycling, recovery, incineration, landfill, anaerobic digestion, composting, etc. [4]. Recycling of food waste is rare due to its easy decay [3]. Incineration has been widely applied to reduce the waste volume and recover energy from waste materials. However, the high water content of food waste consumes most energy for evaporation during combustion [5]. Similarly, the water content of food waste poses a challenge for landfilling, which leads to leachate accumulation and then pollution to the surrounding environment [6]. Traditional bioconversion technologies have been widely explored

for treatment of food waste e.g. anaerobic digestion and composting [7, 8, 9]. But, the bioconversion processes requires long period and high land occupation.

Hydrothermal treatment is the physico-chemical conversion of feedstock at certain temperature (160-600°C) and pressure (5-40 MPa) under subcritical or supercritical water conditions [10, 11]. It is attractive for food waste conversion due to: 1) high moisture content (70-90%), 2) versatility of chemistry, and 3) enhanced reaction rate and efficient separation [11]. The hydrothermal processes (carbonization, liquefaction, gasification) are controlled by temperature and pressure generating certain products such as hydrochar, bio-oil and gaseous compounds [12].

Food waste consists of several constituents including carbohydrates, lignin, lipids/fats, proteins, extractives and inorganic compounds. Carbonaceous solid product or hydrochar is one of the main products during hydrothermal carbonization of food waste, which can be used as fuel, catalyst, fertilizer, etc. dependent on the feedstock and process [10, 13]. Hydrothermal treatment of food waste also promises potential liquid or gas fuel production [11, 13, 14, 15, 16]. It has the advantage over other thermal processes as it is performed at much lower temperature.

In this study, a stepwise process (SP) for the treatment of food waste was developed. The hydrothermal conversion of food waste followed by oil upgrading is a sustainable approach for energy recovery. The produced hydrochars and biodiesel were characterized to evaluate their feasibility for fuel application.

2. Materials and methods

2.1. Synthetic food waste (SFW) preparation and characterization

SFW was prepared containing cabbage (3%), cooked chicken and pork meat (6%), rice and noodles (18%), cooking oil (10%), and water (63%). The food waste composition was empirically determined according to the literature [17, 18, 19]. All ingredients were obtained from local food supermarket in Singapore. The detailed preparation of the SFW is shown in Table S1. In brief, raw rice and noodles were cooked in a microwave oven for 10 and 6 minutes, respectively. Chopped cabbage, chicken and pork were separately boiled with certain amount of water in the microwave oven for 4 minutes. Cooking oil was pre-heated at simulated cooking temperature of 180 °C. In the end, all components were mixed and homogenized in a blender.

SFW was characterized for moisture content, total solids, elemental analysis, lipid determination, and calorific value. The moisture of the SFW was 82% (Table S2). High water content ensured a relative high pressure which is favorable for the hydrothermal process. The calorific value of SFW was about 28 MJ/kg, due to the oil and carbon content. Other elements in SFW such as N, H and O were comparable to literature reported values, indicating a representative sample preparation [18].

2.2. Hydrothermal experiments

Hydrothermal (batch) experiments were conducted in hydrothermal reactors (100 mL and 2 L) (Parr, USA). The reactor was equipped with heating oven, holding stander, probes and functional ports on top for process control, e.g. thermocouples, pressure gauge, gas inlets/outlets, liquid collection, cooling coil inlet and outlet, stirrer, etc. (Fig. S1)

In a typical batch experiment, certain amount of SFW was loaded into the vessel, purged with nitrogen, properly capped, and oven heated for a period of time (20 min up to 2 h). Then, the vessel was cooled down by the cooling coils at room temperature until the pressure was

lower than 10 psi and/or the temperature was below 60°C. The gas valve equipped at the top cover released the gaseous products in a gas bag for further analysis. Afterwards, the cover was open to collect the solid/liquid products. The products were vacuum-filtered through a filter paper (45 µm). Both solid and liquid fractions were subjected to oil extraction with solvents. For oil extraction, it was followed the amended method by Karagoz et al. [20]. In brief, oil was extracted from hydrochar with acetone/hydrochar ratio (w/w) at 10:1 in a sealed container, agitated with magnetic stirrer for 10 hours to obtain the absorbed oil. The liquid was extracted with ethyl acetate (v/v=1:1) to obtain the suspended oil. The extracted oils were mixed and the recovery rate by weight was measured. All hydrochar and oil samples were subjected to detailed characterization.

2.3. Analytical techniques

Hydrochar (before and after oil extraction) was oven dried at 105°C for 24 hours (Memmert UFB500, Germany). The elemental analysis was determined in the Elemental Analyzer (Germany). The calorific value of hydrochars was tested using a bomb calorimeter (IKA, Germany). The gaseous samples were analyzed in gas chromatography GC-TCD-TCD (7890B, Agilent Technologies, USA). However, the overall gas production was low and consisted mainly of CO₂. The liquid after oil extraction was analyzed for its chemical characteristics i.e. COD, NH₃-N, PO₄³⁻, and pH. COD ammonium-nitrogen and phosphates were determined using Hach test kits at spectrophotometer (Hach, USA). The pH value of the liquid was determined with a pH meter (Horiba). The calorific value of collected oil was measured using a calorimeter (IKA, Germany). All analyses were conducted in duplicates.

2.4. Oil upgrading

The oil upgrading system (Newton and Stockers, Singapore) was integrated with the hydrothermal reactor. The upgrading system was installed for 1) automatic collection of hydrothermal liquid driven by pressure from the hydrothermal vessel, and 2) oil separation and reforming. The received liquid was layered shortly after due to various hydrophobic fractions. On top, it was floated the lipid product while the wastewater was collected in the bottom. The wastewater was completely drained, while the lipid fraction was retained in the reactor. Afterwards, certain amount of methanol well mixed with catalyst (i.e. homogenous catalysts including H_2SO_4 and KOH , and heterogeneous catalyst CaO) was introduced by vacuum. The esterification/transesterification reaction lasted for a designed time. Heating system with oil circulation was equipped for temperature control during biodiesel reaction. A continuous agitation was employed via an overhead stirrer. Biodiesel was collected after the end of the reaction, filtered and stored at 4°C for further analysis (Table 1).

The characterization of biodiesel including density (at 15°C), kinetic viscosity (at 50°C), flash point, pour point, micro carbon residue, water by distillation, and sulphur, was made according the ISO standard operation procedures. GC-FID (6790A, Agilent Technologies, USA) was used for quality analysis of the biodiesel. In brief, splitless mode was adopted for inlet setting, under an operation temperature of 250°C . The detector was adjusted to 230°C . Oven temperature was programmed from 50°C until 230°C . GC grade Helium gas was used as carrier gas. Gas flowrate was at 1.0 L/min while solvent delay was set of 4.7 min . The resulted biodiesel samples were denoted as B1, B2, B3, B4, and B5 depending on the feedstock, catalyst used, and experimental conditions.

3. Results and discussion

3.1. Characterization of hydrochar

Elemental composition of hydrochars was determined after hydrothermal treatment (Table 2).

Carbon content (%) was increased from 46% to 74% with temperature increase, while the initial carbon content in SFW was around 51%. The minor carbon deficit in the hydrochars produced at low-temperature hydrothermal treatment could be ascribed to the carbon release into the liquid phase, since the COD value of the hydro-broth was measured as high as 84 g/L (Table S3). The elevated COD value probably indicated partitioning of solid sample into the liquid phase due to chemical transformation and integration [21]. Moreover, the C/N ratio was raised with temperature increment. It is well known that C/N ratio of organic waste is highly associated with heating profile, and hydrothermal carbonization favored its rise because of N release into the liquid phase [22]. On the contrary, there was a loss of O and H content in the hydrochar when temperature was increased. As a matter, O and H release was promoted at higher temperature due to gasification effect, with generation of H₂ and CO₂ [11, 23]. It should be noted that oil recovery rate was higher at lower temperature since high temperature promoted oil degradation. Hydrochars produced from hydrothermal process at different temperature were measured for their heating value as shown in Table 3. The hydrochars presented elevated heating value compared to original dry SFW, mainly ascribed to oil absorption and C/H ratio. The hydrochars attained hydrophobicity due to carbonization, during which fixed carbon was developed. The hydrochars produced at 180°C possessed C content and heating value of 47-48% and 33.7 MJ/kg, respectively, comparable to those of lignite or coal, providing a promising solid fuel, rather than soil conditioner [24]. Hydrochars after oil extraction showed a nearly 30% drop in their heating value (Table 3). It seems that reduced heating values could debate oil recovery from

the produced hydrochars. Hydrochars produced at higher temperature posed similar calorific value even though the increment in C content. Other mechanisms could be responsible for this observant result. Under high temperature of hydrothermal treatment, the subsistent pressure could hinder the formation and/or growth of pores inside the hydrochars impeding oil adsorption. In addition, significant gasification could take place incurring 1) poor oil quality due to complexation, 2) loss of elements with energy storage like H, and 3) hydrochar mass reduction [25]. The above reasons could offset the accumulation of calorific value in the hydrochar at high temperature. Therefore, oil recovery after hydrothermal treatment should be associated with hydrochar oncoming utilization and oil yield.

3.2. Oil upgrading to biodiesel

Oil upgrading to biodiesel was conducted using uncooked cooking oil, cooked cooking oil, and recovered oil from produced hydrochars. Several catalysts (both homogeneous and heterogeneous) were applied for biodiesel production including alkaline and acidic catalysts. Standard method to analyze fatty acid methyl esters (FAME) was established based on David et al. [26] by pre-run different concentrations of the standard mixtures. The name of each peak was assigned with a format as $C(x):(y) n(z)$, where “ x ” (4-24) represents the carbon number on the chain, “ y ” (0/1/2) represents the $-C=C-$, and “ z ” represent the number of C from the end of carbon chain. The biodiesels produced from the different resources using several catalysts and temperatures (Table 1) were subsequently subjected to GC-FID analysis. In general biodiesel production could be affected by several operation conditions including volume, temperature, time, agitation, catalyst, and the characteristics of the primary lipids. The low recovery rate of sample B1 using alkaline catalyst was likely associated with the poor mixing particularly in the

beginning [27]. Saponification could occur in the presence of concentration gradient of alkaline, when KOH was just introduced (in large-piece solid form) and agitation was initiated [28]. Also, the selection of catalyst is critical since alkaline catalyst request a stringent low acidity of the original lipids. High acidity e.g. over 1% could ruin the whole process by promoting significant saponification reaction. The presence of water could compromise the biodiesel production as well [29]. Original oil contains negligible fatty acid (Fig. 1). However, hydrothermal treatments of food waste generated a liquid broth with low pH values (Table S3), suggesting an increased acidity of the recovered oil which may possess complex composition [24]. Pre-washing of recovered oil hereby was necessary before oil upgrading to achieve a high quality biodiesel production.

Fig. 1 shows the GC-FID chromatographs of biodiesel products, indicating a consistent composition among them. The peaks presented in all chromatographs were consistent owing to high quality biodiesel production. The last chromatograph is just reference of used cooking oil. Impurities were negligible from all products since no miscellaneous peaks were presented. All products are characterized with six major peaks, standing for C14:0, C16:0, C18:0, C18:1, C18:2, and C18:3, respectively. The six fractions were one-to-one correspondent to six fatty acids before the transesterification, as myristic acid, palmitic acid, stearic acid, oleic acid, linoleic acid, and linolenic acid, respectively. As the six fatty acids are the dominant acids in soybean and palm oil, which have been mixed to obtain the vegetable oil for the study, each of the identified FAMES was dedicated to their respective precursor fatty acids.

Fig. 2 presents the fractionation of different components among the biodiesels. It seems that C16:0 and C18:1 was the two main fractions accounting 80% or more in the biodiesel, attributed to the abundance of such precursors in the vegetable oil. B5, being derived from the

NTU waste cooking oil, showed a higher fraction of C16:0 than the rest, possibly due to the presence of a higher fraction Palmitic acid from its original resource. More interestingly, B4 which was derived from hydrothermal treatment (at 180 °C) shows no substantial differences on the distribution of FAME fractions as compared to the others. This indicates that hydrothermal process (i.e. carbonization) has not significantly modified the structure of glycerides and, thus, it has minor effect on the subsequent oil esterification/transesterification.

Table 4 presents the biodiesel B3 and B4 fuel specification and properties compared to international standards. The results showed that B3 and B4 were well complied with the US and EU biodiesel standards. B3 biodiesel had lower flash point temperature possessing excellent feature for engine starting and ignition, but safety issues in storage and transportation should be considered. B4 had higher flash point that could cause misfire, ignition delay and carbon deposits in the combustion chamber. The low micro carbon residue could provide better combustibility and lower deposit in the engine. The low sulfur content could reduce emissions of particulate matter from diesel engines and reduce attrition between moving parts of diesel injector pump and fuel injectors. The viscosity of B3 biodiesel was in the range of biodiesel standard whereas B4 biodiesel possessed slightly higher viscosity than the standards. The high viscosity of B4 could be due to the incomplete reaction during biodiesel production, whereby significant amount of monoglycerides and diglycerides were present and mixed with FAMEs with no further transformation. In consideration of the calorific value, both biodiesels had much higher values.

4. Conclusion

SP (hydrothermal treatment and oil upgrading) was developed for high quality fuel production. It was highlighted that the produced hydrochars had similar quality to coal, while biodiesel met the international standards. According to SP: 1) the caloric value of hydrochars was much higher before oil extraction, 2) oil had significant merit, 3) hydrothermal carbonization had minor influence on biodiesel quality, 4) higher hydrothermal temperature/pressure could compromise the biodiesel recovery. In overall, the stepwise hydrothermal treatment and oil upgrading could be considered as an alternative approach to treat food waste comparing to conventional bioconversion or other thermal processes.

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ACCEPTED MANUSCRIPT

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Tables and Figures

Table 1. Experimental conditions of the upgrading process for the extracted oil from SFW.

Sample	Description	Vol. (mL)	Catalyst (%)	Methanol (mL)	Time (h)	Temp (°C)	Mixing (rpm)	Recovery (%)	HHV (MJ/Mg)
B1	Cooking oil	100	KOH (1%)	100	2	60	200	30	40.0
B2	Cooking oil	50	98% H ₂ SO ₄ (20%)	50	17	60	200	80	39.8
¹ B3	Cooking oil	500	CaO (8%)	854.4	3	65	100	100	38.9
B4	HT@180 °C	150	98% H ₂ SO ₄ (10%)	150	10	80	200	91	39.7
¹ B5	HT@180 °C	500	CaO (8%)	854.4	3	65	100	10	39.7

¹: Methanol dosage was higher for B3 and B5 with heterogeneous catalyst.

Table 2. Characterization of hydrochars generated from different hydrothermal experiments.

Hydrothermal temperature (°C)	Reaction time (min)	Sample mass (g)	Oil recovery (%)	Char			C/N
				C (%)	H (%)	O (%)	
140	120	30.0±0.03	98.8±0.5	46.2±0.2	7.3±0.03	35.6±0.5	5.1±0.9
160	120	30.0±0.02	96.0±3.6	49.8±0.4	8.0±0.1	31.9±2.0	5.1±1.4
180	120	41.2±6.68*	74.8±10.7	48.7±0.3	7.1±0.01	30.9±0.1	5.1±0.1
180	20	1008.0±2.83	92.0	46.7±1.0	3.9±0.1	24.7±1.6	6.4±0.1
320	20	200.0±0.05	75.8±6.1	74.6±1.0	6.4±0.85	9.6±2.5	14.3±2.5

*: Higher deviation shown by sample mass of 41.2 g was due to the unexpected low mass recovery for one of the samples after thermal experiment (@180 °C) from its original preparation.

Table 3. Measured heating value of the hydrochar under different experimental conditions.

Samples	Hydrothermal temperature (°C)	Calorific value (HHV, MJ/kg)		
		Hydrochar before oil extraction	Hydrochar after oil extraction	Oil
Synthetic food waste	Untreated	28.3±0.7	17.0±0.4	39.6
	180	33.7±0.6	21.3±0.1	39.8
	320	32.5±0.0	N.A.	37.2

Table 4. Physicochemical characteristics of biodiesel production from the stepwise system.

Test	Method	Unit	Result		ASTM biodiesel STDD 6751	EU biodiesel STD EN 14214 for vehicle	EU biodiesel STD EN 14213 for heating oil
			¹ B3	¹ B4			
Density @15 °C	EN ISO 12185	kg/m ³	872.7	895.5	--	860-890	860-900
Kinematic Viscosity @50 °C	EN ISO 3104	cSt	3.115	8.243	1.9-6.0	3.5-5.0	3.5-5.0
Flash Point (PMCC)	EN ISO 2719	°C	30	>126	130 min	120 min	120 min
Pour Point	EN ISO 3016	°C	+ 6	+ 6	--	--	--
Micro Carbon Residue	EN ISO 10370	% wt	0.03	0.04	0.05 max	0.30	0.30
Water Content	EN ISO 12937	mg/kg	1953	2684	500	500	500
Sulfur content	EN ISO 8754	% wt	<0.0016	<0.0016	0.02% max	0.01%	0.01%
Calorific Value	DIN 51900, DIN 51900- 1/2/3	J/g	38917	39584	NA	NA	≥35000

¹: B3 and B4 refer the sample in Table 1.

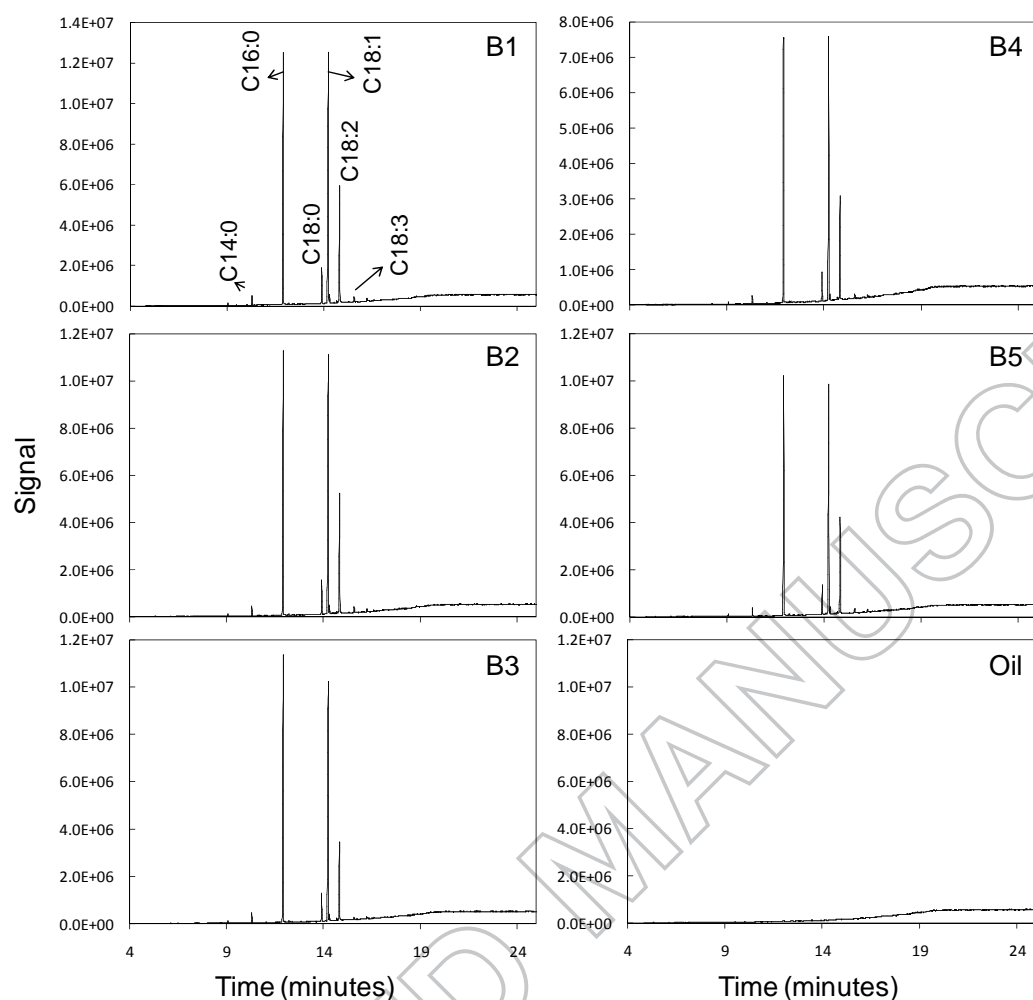


Fig. 1. Gas chromatography of biodiesel products from oil refinery system (samples B1-B5).

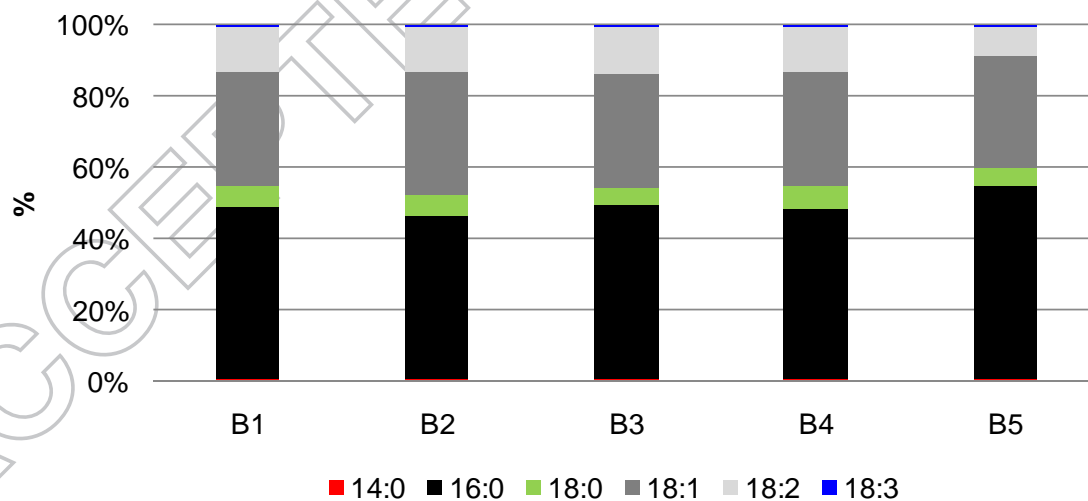


Fig. 2. Fractional distribution of different carbon chains in the biodiesel products (samples B1-B5).

Supplemental tables and figures

6

7 Table S1. Synthetic food waste (SFW) preparation

Components	Theoretical weight (g)	Cooking time (min)	Percentage by weight (%)
Rice	121.22	10	12.12
Noodle	60.60	6	6.06
Pork	30.30	4	3.03
Chicken	30.30	4	3.03
Cabbage	30.30	--	3.03
Oil	100.00	--	10.00
Water	627.28	--	62.73
Total	*1000.00		

*: loss after blending was determined at ~4%.

8

9 Table S2. SFW characterization

No. of analysis	H ₂ O%	C%	H%	O%	Caloric value (J/g dry)
1	81.95	52.48	8.54	29.19	
2	81.84	50.20	8.40	31.49	
3	81.74	51.16	8.62	29.99	
Average	81.84	51.28	8.52	30.22	28286

10

11 Table S3. Hydrothermal broth characterization under different treatment conditions.

Processes	Temperature (°C)	Volume (mL)	Reaction time (h)	Liquid analysis			
				COD(ppm)	NH ₃ -N (ppm)	PO ₄ ³⁻ (ppm)	pH
HTC	180	45	2	25700	91.8	188.4	4.3
	180	1000	1/3	84350	416.0	120.4	NA
	220	45	2	21250	138.7	254.0	3.7

12

13



Fig. S1. Laboratory setup of the SP.

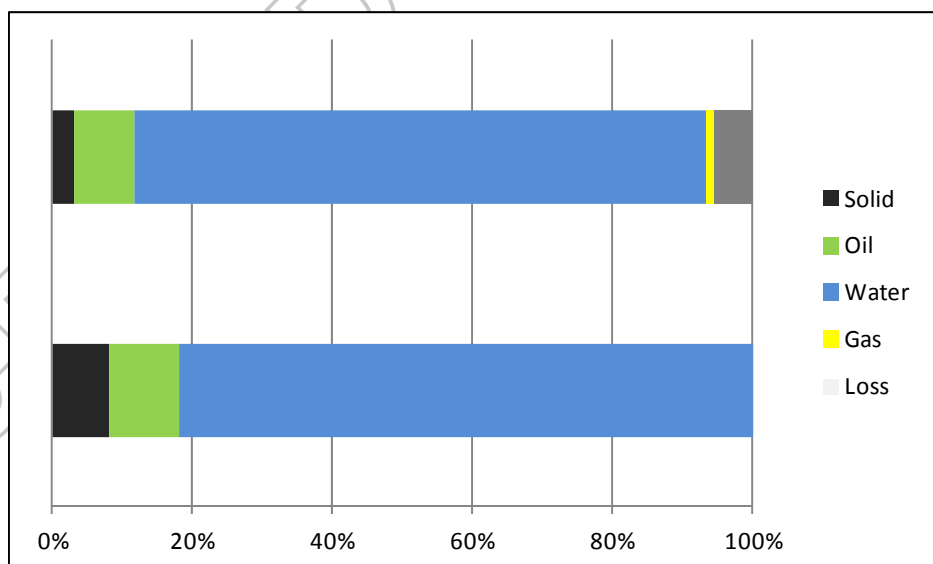


Fig. S2. Yield of hydrothermal product vs. original composition (low bar: components from original SFW and high bar: the components after hydrothermal reaction (320 °C in 20 min)).