



Conversion of Citrus Fruit Peel into a Value-Added Product, Bio-Oil

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ABSTRACT

The present study aimed to investigate the bio-oil from the blended citrus fruit peel by hydrothermal liquefaction process. Huge amounts of fruit peel waste are disposed of in the open environment without any proper management. Such fruit peels are considered a potential bio-resource to be converted into economically valuable products like bio-oil. Since the citrus fruit peel is a rich source of moisture content, a hydrothermal liquefaction process was introduced to produce bio-oil from cellulose, and lignocellulose. The experimental design against temperature, time, and biomass concentration optimization was carried out which was confirmed by the ANOVA *f* and *p* test that reveals time and temperature influenced the bio-oil yield drastically. As the time and temperature rise more than 60 min and 280°C, the volatile substance present in the biomass converts itself into solid residue which has a negative impact on bio-oil production, compared with biomass concentration. The maximum yield of bio-oil was recorded as 29.4% at 280°C at 60min reaction time and 80g/200mL concentration as optimized parameters. The GCMS reveals the presence of hydrocarbons and alkanethiol which are flammable and hold the standards of commercial transportation fuel but hold nitrogen and oxygen-containing compounds to pull down the fuel standards. Thus, the produced bio-oil can be blended with the transportation fuel after the upgradation process for efficient results.

INTRODUCTION

Increases in population and waste generation are two interrelated things that are directly proportional to each other (Supangkat & Herdiansyah 2020). Statistical data reveals that the waste generation in the world will rise to 73% by 2050 (The World Bank 2024). As the waste generation increases, to ensure a clean environment the generated waste has to be managed efficiently in a traditional manner to discard waste in a bare land named landfills which may cause various hindrances to mankind and the surrounding ecosystem. Landfills can cause air pollution by releasing a high amount of greenhouse gas into the environment resulting in global warming, and water pollution by discarding the heavy metals into the ground water which results in various health issues. This also pollutes the soil and makes the land unfit causing communicable diseases, eye infections, skin infections, dust allergies, gastrointestinal tract infections, etc. in the nearby population (Parvin & Tareq 2021, Siddiqua et al. 2022). However, waste management awareness among the common man, the 3R concept is booming and reducing the percentage of landfill usage for waste disposal. But a wiser way of utilizing the waste is to convert it into value-added products like bio-oil, biomaterials, and biochemicals, which must be opted for by the population to decrease and efficiently utilize the waste (Bharathiraja et al. 2017, Sadh et al. 2018). Some of the popular methods used in current days to convert waste are thermochemical conversion and biochemical conversion methods (Nanda et al. 2013, Aboagye et al. 2017, Kim et al. 2015). In this study thermal method of treatment has been chosen. Though the thermal method includes various techniques like pyrolysis, gasification, hydrothermal process, etc. hydrothermal

process is been used here since the waste utilized under this method does not need a pretreatment process, thereby saving energy and time consumption.

The waste chosen for treatment here is citrus fruit peel, a lignocellulose waste that has a production rate of 180 billion tons every year globally. Such waste can be efficiently converted into value-added products by the hydrothermal liquefaction process since the process holds several advantages in treating wet biomass, using low temperatures, and reducing energy consumption (Vo et al. 2016). The hydrothermal liquefaction process is well-studied for processing feedstocks from lignocelluloses wet waste which leads to oil products in a mixture of hydrocarbons (gasoline/ jet/ diesel range) (Snowden-Swan et al. 2017). The Hydrothermal Liquefaction process also undergoes a significant thermodynamic change (supercritical phase) at high temperature (200-350°C) and pressure up to 25 MPa for wet biomass which leads to weakening and ionization of H bonds into hydronium (CHO) and hydroxide (OH-) ions (Beims et al. 2020). Hydrothermal Liquefaction influences acid-catalyzed hydrolysis and degradation leading to the breaking down and reforming of biomass to cellulose, hemicellulose, lignin, and ultimately to biocrude with the higher energy content of 30-35 MJ/kg (Ruiz et al. 2013). Malins et al. (2015) demonstrated the effect of different catalysts in the efficient production of biocrude from sludge and Vardon et al. (2011) carried out Hydrothermal Liquefaction processes with sludge, manure, and algae anaerobically and analyzed various parameters of biocrude. The investigation carried out by Hariram et al. (2023) also utilized avocado fruit seed to extract bio-oil by trans-esterification process. Lignocellulose waste is the major contributor of cellulose, hemicellulose, lignin, and pectin that contribute to producing value-added products by break down mechanism of biomolecules by hydrothermal liquefaction process at temperature ranges from 200°C-450°C and pressure ranges from 5-20 MPa (Baruah et al. 2018, Dimitriadis & Stella 2017). The optimum parameters like temperature, the concentration of biomass, and time were experimentally studied to produce the bio-oil. Gas Chromatography and Mass Spectrometry) GCMS and physicochemical proximate analysis methods were used to analyze the bio-oil. Thus, this research aims to provide a basic understanding of the effect of the Hydrothermal Liquefaction process on the citrus fruit peel in producing the bio-oil.

MATERIALS AND METHODS

Sample Collection

The citrus fruit peel was collected from the canteen of "Aarupadai Veedu Institute of Technology, Chennai". The collected biomass (orange peel lemon peel and sweet lime

peel) was made into coarse powder. This fresh sample consists of 78% of moisture content. This also contains 87.4% carbon, 11.2% of nitrogen, 1.1% of hydrogen, and 30.9% of oxygen.

Hydrothermal Liquefaction Process

The hydrothermal liquefaction process was carried out in a thermal autoclave reactor of a capacity of 5 L with an operating temperature of 350°C and a heating rate of 10°C/min. The biomass was loaded in the desired volume to the reactor vessel along with distilled water as solvent. The holding time of the reaction was set as 1 hour with 700 rpm as the speed of rotation. The layer separation method was used for extracting the bio-oil using hexane as solvent. An equal volume of hexane solvent was added to the bio-oil in a separating funnel for the separation of the organic layer from the residue. The bio-oil was further purified by a rotary vacuum evaporator from which the organic layer was separated using a distillation process. The same conditions were repeated in triplets to minimize the error. The bio-oil yield was calculated by weighing the weight of the bio-oil against the weight of the biomass taken for the hydrothermal liquefaction process.

Design of experiment (DOE)

The optimization study was carried out and verified by using the ANOVA p-test. The experiment was carried out with 3 optimizing parameters – temperature (210°C, 220°C, 240°C, 260°C, 280°C, 300°C and 310°C), time (50 min, 60 min, 120 min and 130 min) and biomass concentration (10 g/200mL, 20 g/200mL, 40 g/200mL, 50 g/200mL, 60 g/200mL, 80 g/200mL, 100 g/200mL, 110 g/200mL). The ANOVA p-test analysis was used to confirm the effect of 3 variables on the yield and experimental fitness after 16 runs of the experiment listed in Table 1.

Bio-oil Characterization

The bio-oil produced was characterized by GCMS and property analysis, to find the compounds present in it and ensure the efficiency of the bio-oil. GCMS analyses the compounds present in the bio-oil produced from citrus fruit peel (orange peel, lemon peel, and sweet lime peel). The chromatography utilized here was Agilent 7890 GC outfitted with an Agilent 7683B auto-injector, an HP-5 section, and a flame ionization detector. Helium gas served as a carrier with a flow rate of 1 mL/min and an inlet temperature of 250°C. The start temperature of the detector starts from 50°C per 2 min and rises by 10°C/min. The electron input and scan range were found to be 70eV and 35-335 amu. Property analysis was carried out to find the kinematic viscosity, calorific

value, density, and TAN (Total Acid Number) number to find the efficiency of the bio-oil.

RESULTS AND DISCUSSION

Effect of Parameters on Hydrothermal Liquefaction Process

The effect of the parameters on bio-oil yield was analyzed (Table 1). The bio-oil production was found to increase as the temperature rose from 200°C to 260°C and started to

decrease as the temperature rose further to 300°C. From the above analysis, the optimum temperature for thermal liquefaction was found to be 280°C (Fig. 1). The same protocol was implemented to optimize the time. 4 different timings were taken for the process namely – 50 min, 60 min, 120 min, and 130 min. The maximum yield was recorded at 60 min. The above analysis infers, that the optimum time for the best conversion of citrus peel into bio-oil was recorded as 60 min (Fig. 2). To optimize the concentration, the same process was carried out with 20 g/200mL, 40 g/200mL,

Table 1: Design of the experiment.

Experimental run	Variables			Response
	Temperature (°C)	Time (min)	Biomass concentration (g/200mL)	Bio-oil Yield %
1	220	60	20	18.2
2	240	60	40	21.8
3	260	60	60	26.7
4	280	60	80	29.4
5	300	60	100	29.2
6	220	120	20	12.3
7	240	120	40	15
8	260	120	60	16.1
9	280	120	80	19.7
10	300	120	100	21
11	210	50	110	23
12	210	50	10	10.3
13	210	50	50	13.5
14	310	130	110	21.6
15	310	130	10	2
16	310	130	50	4.7

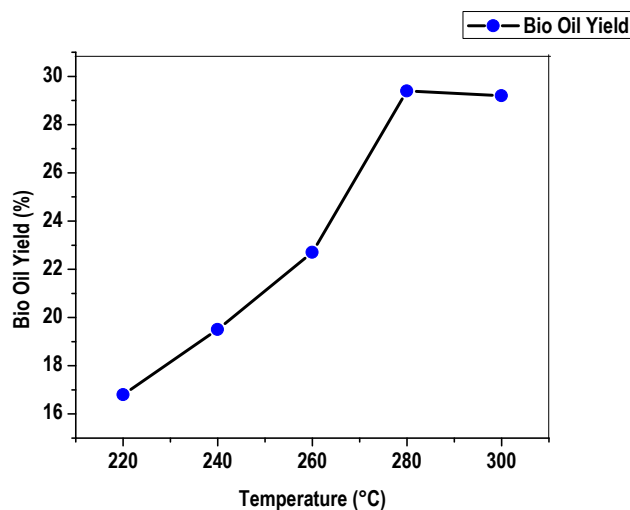


Fig. 1: Mean value of response with temperature.

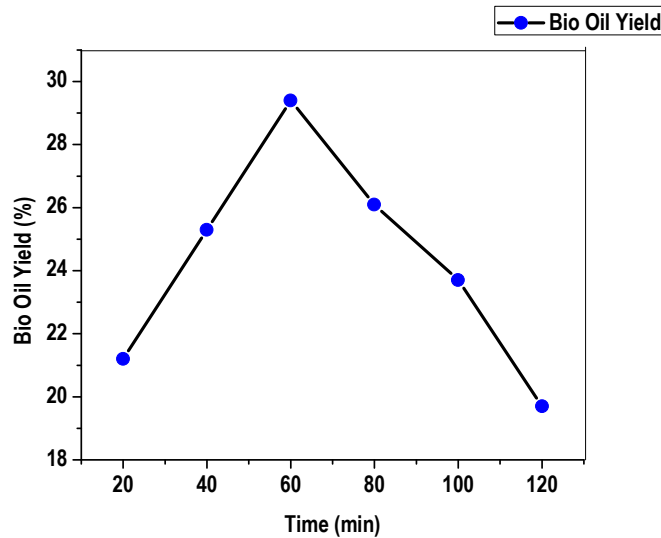


Fig. 2: Mean value of response with time.

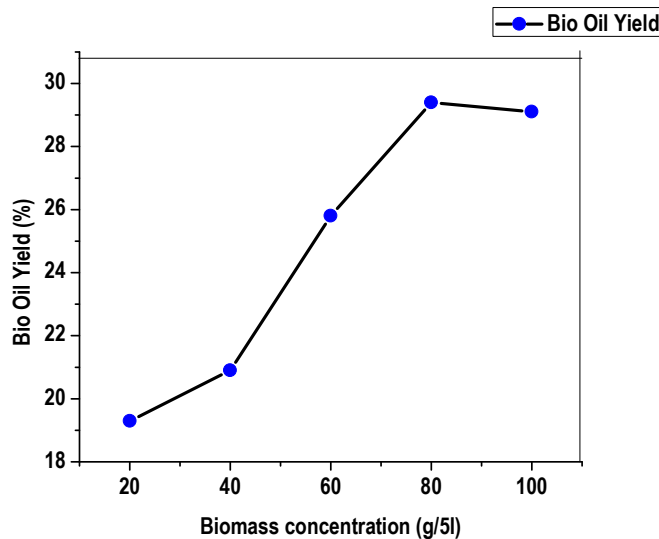


Fig. 3: Mean value of response with biomass concentration.

50 g/200mL, 60 g/200mL, 80 g/200mL, 100 g/200mL, and 110 g/200mL. Among the different concentrations, 80 g/200mL recorded the highest yield of bio-oil and was noted as an optimized concentration (Fig. 3). Thus at 280°C, 60 min, and 80 g/200mL concentration the yield of bio-oil was recorded as 29.4 yield %. Along with bio-oil, biochar was obtained as the by-product which can be used as a bio-fertilizer. Orange peel requires 275°C for effective conversion of biomass into bio-oil due to the presence of elemental carbon (Billar & Ross 2011, Dandamudi et al. 2019, Reddy et al. 2016). The yield recorded by microalgae was found to be higher than the citrus peel biomass due to the presence of lipid content in the microalgae that enhances oil

production at greater temperatures. However, the citrus peel produces the bio-oil at a lesser reaction temperature since the biomass is a rich source of lignocellulose, cellulose, and lignin. On further increasing the temperature, the gasification process is favored and increases the biochar yield. Thus, low temperatures are best suited for lignocellulose-rich biomass for bio-oil production (Mohan et al. 2006). The maximum yield range of biocrude was recorded between 25 to 28% at a temperature of 200 to 275°C and minimum biocrude yield was obtained between 4.4 to 9.5% at a temperature of 275°C, respectively in hydrothermal liquefaction process with orange peel as biomass (Divyabharathi & Subramanian 2021). High volatile matter present in the citrus peel promotes

bio-oil production and less lignin content reduces the biochar yield out of the hydrothermal liquefaction process (Divyabharathi & Subramanian 2021).

ANOVA Evaluation For the Response of Bio-Oil

ANOVA evaluation recorded for the response of bio-oil reveals in Table 2, that the temperature and the reaction time of the hydrothermal liquefaction process significantly affect the bio-oil yield compared with the concentration of the biomass. As the temperature rises, the bio-oil yield also increases significantly till 280°C and slightly falls, on further rise in temperature. The same rise in bio-oil yield was observed when the temperature rose to 60min but, tend to fall further doubling the reaction time. The reason for a fall in the bio-oil yield in the above two parameters is the presence of higher volatile matter that gets converted into solid residue at increased temperature and at prolonged periods of reaction time. Thus, the model is significant and found to fit with both temperature and time. On the other hand, concentration has having lesser impact on bio-oil yield when compared to temperature and time. When the concentration is maintained constant on increasing temperature and time, the conversion rate of the biomass increases until the optimal parameter (280°C and 60 min) and starts to decrease on a further rise. Higher temperature enhances biomass cracking and promotes the hydrolysis process resulting in the production of lower-weight compounds (Changi et al. 2015). Further processes such as decarboxylation, dehydration, and repolymerization in the hydrothermal liquefaction process result in the formation of various intermediate and by-products (Wang et al. 2018). Improved biomass conversion was recorded at increased reaction time during the hydrothermal liquefaction process (Dimitriadis et al. 2017, Xu & Etcheverry 2008). According to the choice of biomass, reaction time tends to differ as recorded in the

literature – 10 min for microalgae (Valdez et al. 2012), 15 min for swine manure (Xiu et al. 2010), 30 min for corn stalk (Zhu et al. 2014) and 80 min for bamboo biomass. The water content present during the liquefaction process decides the percentage of the hydrolysis process that enhances the bio-oil yield by promoting maximum conversion. But increasing the biomass concentration the solid residue formation gets elevated and pulls down the bio-oil yield (Qu et al. 2003). Regression coefficients were obtained through the least square technique by Divyabharathi & Subramanian (2021), wherein the effect of linear and quadratic terms was analyzed and proved that there was a significant effect at a 5% level on bio-crude production during the period of study and similar response in terms of aqueous and char yield were 0.822 and 0.937 respectively. The lignin content present in the biomass decides the bio-char production resulting in 0.8 to 6.9% from orange peel by Hydrothermal Liquefaction Process. The lesser the lignin content lesser the bio-char production (Divyabharathi & Subramanian 2021).

Proximate Analysis of Bio-Oil

The proximate analysis of the synthesized bio-oil recorded 31.2 MJ/kg calorific value, holding a density of 937 kg/m³, the viscous nature of 7.05 mm², and TAN as 0.59 KOH/g concentration in Table 3. The bio-oil produced from biomass using the hydrothermal liquefaction process always has a major correlation with the existing biodiesel and diesel standards (Alleman et al. 2016). The properties of biocrude obtained from orange peel were analyzed by Divyabharathi & Subramanian (2021) and registered 32 MJ/kg of heating value, 93°C of flash point, during the period of study, and the properties registered from orange peel had comparable fuel properties with those of biodiesel/diesel and they are potentially used as a marine bunker fuel or furnace oil due to its similar viscosity, flash point and heating value

Table 2: ANOVA response of bio-oil yield.

Source	SOS	DF	MS	F value	P Value	Status
Model	31.517	9	3.501	9.99	0.0010	Significant
A-Concentration	1.333	1	1.333	3.806	0.0820	Not significant
B-Time	2.3105	1	2.310	6.596	0.0302	Significant
C-Temperature	2.482	1	2.482	7.087	0.0259	Significant
AB	3.079	1	3.079	8.792	0.0158	Significant
BC	5.733	1	5.733	16.371	0.0029	Significant
AC	3.308	1	3.308	9.446	0.0132	Significant
A ²	1.776	1	1.776	0.050	0.8268	Not significant
B ²	5.336	1	5.336	15.230	0.0036	Significant
C ²	6.160	1	6.160	17.590	0.0023	Significant

Where, SOS – Sum of Squares, DF –Degree of Freedom, MS – Mean Square

Table 3: Proximate analysis of Bio-oil.

Property	Unit	Bio-oil
Calorific Value	MJ/kg	31.2
Density	kg/m ³	937
Viscosity	mm ²	7.05
TAN Number	KOH/g	0.59

(Hossain et al. 2017). The standard similarity promotes their usage in blending with commercial transportation fuel or replacing its usage. However, proper purification steps like fuel upgradation or downstream techniques, viz. catalytic cracking to perform hydrodeoxygenation, desulphurization, and hydrotreatment must be carried out to remove the excess hydrogen and oxygen from the bio-oil to increase the efficiency (Hossain et al. 2017).

GCMS Analysis of Bio-Oil

GCMS analyzes the organic compounds present in the bio-oil produced. The composition of the bio-oil obtained under optimal operating conditions detected by GCMS was given in Table 4 and Fig. 4. The major compounds eluted out include pentadiene (33.5%) – an organic compound that is volatile and considered a flammable hydrocarbon, tetramethyl 2-hexadecene (24.5%) is an isoprenoid hydrocarbon, a derivative of chlorophyll present in the plant, hexadecane thiol (22.4%) an alkanethiol that forms monolayer by sulfur ions with the atoms present on the surface which is combustible and insoluble in water, trimethoxy methyl dihydroisoquinole (4.3%) used as a flavoring agent and dimethyl octadecanamide (14.7%). All the mentioned compounds were obtained from the decomposition and

Table 4: Major compounds present in bio-oil from citrus peel detected by the GCMS.

Retention time (min)	Area %	Compound name
3.223	0	2-Ethyl-1-pyrrole
4.933	0.1	Tetramethyl-1-dimethyl cyclopentane
5.677	33.5	Pentadiene
7.733	0.2	Trimethyl cyclohexanone
8.190	0.1	Pyridine
17.223	0.1	Hydroquinone
21.170	0.1	Benzene 1,2,3-trimethoxy
27.800	24.5	Tetramethyl 2-hexadecene
28.747	22.4	Hexadecanethiol
31.713	4.3	Trimethoxy-methyl-Dihydroisoquinole
33.087	14.7	Dimethyl octadecanamide
Total	100	

depolymerization of the citrus peel. The presence of hydrocarbon was observed since they are derived from plant-based biomass which is regularly used as a flavoring and fragrant-producing agent in industrial sectors (Sharma et al. 2020). The hydrocarbon derivatives increase with an increase in temperature. However, initially during the temperature raised from 200°C to 250°C, nitrogenized compounds were eluting out, as the temperature reached 275°C, hydrocarbon compounds like phenol, alkanes, and isomeric alkenes were produced. This may be the result of decarboxylation and decomposition processes carried out by acids. The amine derivatives oxygenate and cyclic hydrocarbons which are the result of the repolymerization reaction pull down the stability

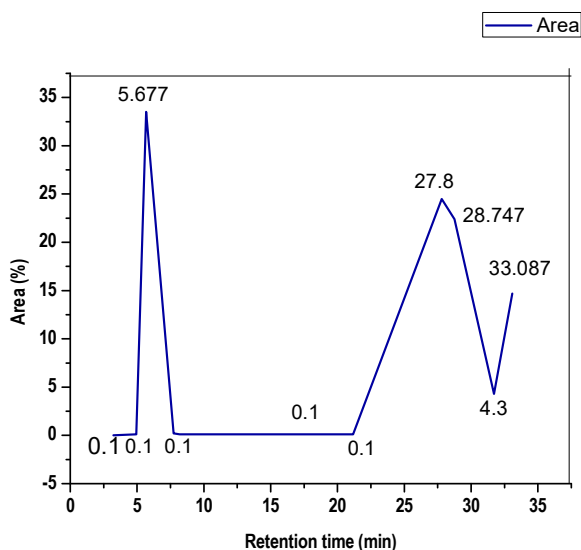


Fig. 4: GCMS of bio-oil from the citrus fruit peel.

of the bio-oil which can be over ruined by undergoing the fuel upgradation process (Palomino et al. 2020, Liu et al. 2013, Chen et al. 2014).

CONCLUSION

Based on the experimental design, the temperature and time influenced the bio-oil yield compared with the concentration of citrus fruit peel as biomass which was confirmed by ANOVA *f* and *p* test. The optimized parameters for the bio-oil were recorded at 280°C at 60 min reaction time and 80 g/200mL concentration by undergoing a hydrothermal liquefaction process. The maximum bio-oil yield was recorded as 29.4%. The fuel property of the bio-oil was obtained as 31.2 MJ/kg calorific value, 937 kg/m³ density, 7.05 mm² viscosity, and 0.59 KOH/g TAN number. The GCMS analysis reveals the presence of hydrocarbons and alkanethiol which are flammable and hold the standards of commercial transportation fuel. Thus, the produced bio-oil can be blended along with the transportation fuel after carrying out the fuel upgradation process that removes excess oxygen, sulfur, and nitrogen content or it can be used directly as marine bunker fuel or for furnace purposes.

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