



Solar Thermal Pyrolysis of Karanja Seeds for a Sustainable Approach for Liquid Biofuel Utilization

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ABSTRACT

The present study is based on the conversion from biomass to biofuels of karanja (*Pongamia glabra*) seeds via solar thermochemical pyrolysis process. Karanja seeds were pyrolysed at a cavity type reactor temperature of 280-340°C. The pyrolysis process was occurring in the range of 210-550°C. The ultimate and proximate analysis of the pyrolysed bio-oil was performed based on ASTM standards. The FTIR (Fourier transform infrared spectroscopy) analysis of the liquid product indicated the presence of alkenes, alkanes, ketones, carboxylic acids and aromatic rings. GC-MS (gas chromatography-mass spectrometry) demonstrated the presence of hydrocarbons having between 15 and 34 carbon atoms in a chain.

INTRODUCTION

Biomass is another substitution of energy dependence on non-renewable sources. Probably it has a capacity to overcome the present requirement of energy. Bio-fuel from non-conventional oil containing seeds is one of the major search areas of biomass energy (Mondal et al. 2018). Extraction of oil from these seeds is possible via thermochemical and biochemical ways. One of the major advantages of biomass is negligible amount of sulphur content in the fuel or oil. These fuels are CO₂ neutral fuels as the generated CO₂ after burning is used by plants for their growth. Hence, these are better and with very less amount of carbon footprint on the environment. The projected capacity of biomass is about 1.08 × 10¹¹ toe (tons of oil equivalent), which is far more than the present need of energy (De Wit & Faaij 2010). Combustion, pyrolysis, gasification and high pressure liquefaction are the main processes for obtaining the oil from biomass (Elliott Beckman et al. 1991).

Here are some advantages of using solar energy for biomass production according to Z'Graggen & Steinfeld (2009):

- Upgradation of calorific value of the feedstocks.
- Decontamination of the gaseous product by the by-product of combustion.
- Environment seems to be more cleaner due to the reduced discharge of pollutants in the air.

- Consumption of pure oxygen from the air for the energy intensive process has been eliminated.

The above four points add more advantages to solar thermal pyrolysis for bio-oil generation. There are limited number of studies that have been done using this technology, which are discussed below.

Gasification of biomass is a well known renewable source for transport fuels; when heat supply to this process is done by concentrating solar system it becomes even more efficient process (Gregg et al. 1980, Mondal et al. 2014). Mainly biomass contains carbon, hydrogen and oxygen as CH_{1.4}O_{0.6}, that is carbon 52.2%, hydrogen 6.1%, oxygen 41.8% as per weight. Burning, which takes place in presence of oxygen, generates carbon dioxide and water as products. These products are then converted to synthetic fuels. Nzihou et al. (2012) have described very well about engineering a commercial solar gasification system.

Concentrated visible light radiation was used by Hopkings et al. (1984) for flash pyrolysis of cellulosic material. Clear quartz spouted bed reactor was used for study and flux was supplied by an arc image furnace which uses xenon bulb as light source. Flechsenhar & Sasse (1995) used fixed bed reactor containing 0.05 g of biomass under argon atmosphere. The irradiation power was derived by means of sun simulator consisting basically of a xenon lamp placed in the focus of a parabolic reflector. Image furnace technology also has been used for flash pyrolysis of cellulose

(Boutin et al. 2002). Packed-bed solar reactor for the steam gasification for carbonaceous matter was studied by Piatkowski et al. (2009, 2011). Reactor was subjective to radiation flux concentration up to 2953 sun, creating temperature up to 1490 K, yielding high quality of syngas. Solar-to-chemical conversion efficiency varied between 17.3% and 29%.

Li et al. (2016) studied the product distribution from solar pyrolysis of agriculture and forestry biomass residue using solar furnace setup. Morales et al. (2014) investigated orange-peel pyrolysis using parabolic-trough concentrator. The average irradiance of pyrolytic reactor surface was 15.65 suns. Reflectivity by biomass was one of the reasons for heat loss in the process, which was around 38%, and around 36% was due to the temperature difference between reactor and environment. The maximum temperature of the reactor at the middle of the focal line was 465°C. Reported weight loss of orange peel was 79 wt% at an irradiance of 12.55 kW/m². Yield of bio-oil was 77.64% and 1.43% was non-condensable gases with 20.93% of char. This proves better efficiency of solar thermal pyrolysis with 465°C of temperature achieved, which is in the range for pyrolysis of biomass.

Use of solar furnace for the pyrolysis process is also one of the methodologies other than CSP. Zeng et al. (2015) reported solar pyrolysis of wood on a laboratory scale by solar assisted pyrolysis reactor setup. In this study, they used beech wood and studied effect of temperature and sweep gas flow rate on the product yields. In this study, they had control on heating range and temperature under solar concentration. Around 51% yield of gas was obtained by pyrolysis at 2000°C with heating rate of 50°C/min and argon flow rate of 6 NL/min. At higher temperature gas yields were higher (Zeng et al. 2017a). This group is continuously working on wood pyrolysis using this technology with computational studies and modelling (Zeng et al. 2016). When water content increases, it reduces the tar decomposition, carbon monoxide and hydrogen decreases. Char decomposition plays a major role in yield of product bio-gas (Zeng et al. 2017b).

Solar tracking helps to remain focused with high temperature needed for the process. In whole, the sun changes its position using solar tracker Fresnel, continuously adjusted with the position of the sun (Abdul-lateef 2012). This Fresnel system has been built in-house by Zeaiter et al. (2015). They studied scrap rubber catalytic pyrolysis using this system.

Chintala et al. (2017) designed a Scheffler parabolic dish with 16 m² of reflected area. The dish was used to concentrate the sunlight radiation onto pyrolysis reactor and bio-oil is produced. An automatic tracker system was also attached

with it. System was designed for various types of biomass feedstock, however, in this study they used *Jatropha* biomass for oil production. The average range of temperature was around 250-320°C and exploring maximum 20% of bio-oil yield. The temperature was pretty much in the range of required temperature for pyrolysis of biomass. Further studies and characteristic studies for char, liquid oil and gas were done. Oil was utilized in compression ignition engine after upgradation and blending with diesel at 20%, 40% and 60% shares.

MATERIALS AND METHODS

Raw materials: Karanja seeds were procured from the local market and directly used for pyrolysis. The procured seeds were washed several times to remove the dust and then kept in open for drying and then kept into the oven at 110°C for removing moisture. The seeds were then crushed by the seed crusher and placed into the pyrolysis reactor through hopper. To assess the volatile and non-volatile matter in the material, moisture content and ash content, proximate analysis was carried out by following ASTM D3172-07a guidelines.

Carbon, hydrogen, nitrogen, sulphur and oxygen elemental analyser (Thermo Flash 2000 CHNS/O analyser) was used to assess the contents of carbon, hydrogen, nitrogen, sulphur and oxygen.

A capillary column coated with a 0.25 mm film with DB-5 was used. The capillary tube's length and diameter were 30 m and 0.25 mm respectively. Helium gas of 99.999% purity was used as a carrier gas. The oven initial temperature was set 60°C for 2 min and then 300°C at a rate of 10°C/min and maintained for 10 minutes. The compounds were identified by the NIST library.

Experimental set-up and procedure: A circular type conventional fixed bed reactor for lab scale pyrolysis process was developed, having a total biomass capacity of 18 kg/batch. The distributed reflection of sun rays onto the pyrolysis reactor was concentrated by using a parabolic solar concentrator of 16 m² as shown in Fig. 2d. The schematic diagram (Fig. 1) and detailed technical description (Table 1 and Table 2) of scheffler dish and the reactor have been defined respectively. A timer was set with the wheel of the scheffler dish for uninterrupted focusing on the reactor. As the solar concentrator was concentrating on the surface reactor, temperature increased significantly by captivating heat. The involved process here is heat transfer mechanism along with simultaneous increase in biomass temperature. When the inner reactor temperature reaches to 210°C, the reaction took place inside the reactor and the vapour forms. Then, formed fuel vapours were condensed and stored in a

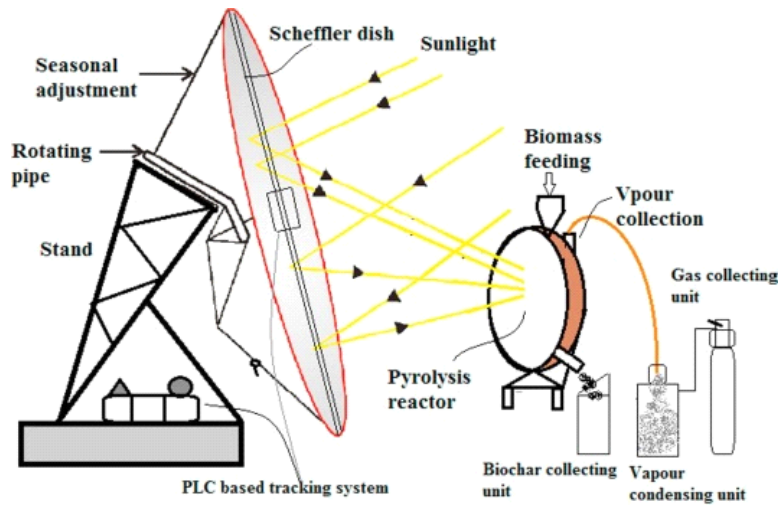


Fig. 1: Concept diagram (Chintala et al. 2017) of solar thermal parabolic dish with reflection technology.

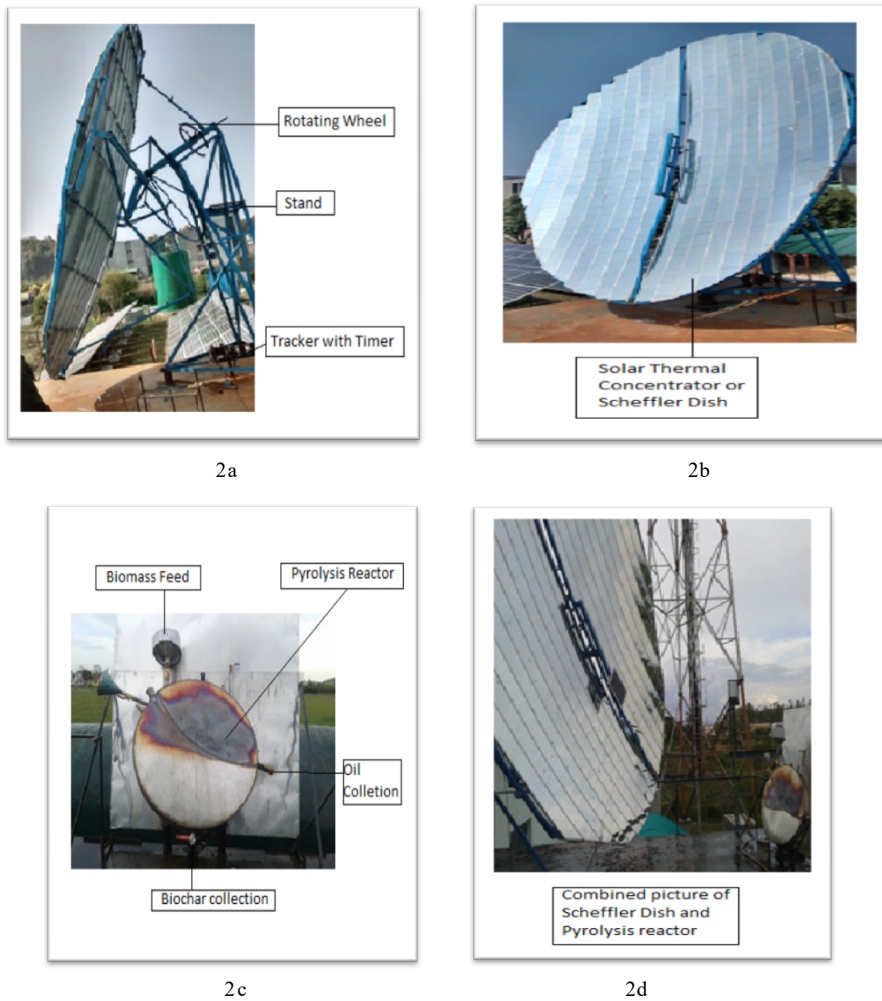


Fig. 2: (a) Side view of scheffler dish; (b) Front view of scheffler dish; (c) Solar thermal pyrolysis reactor; (d) Overall setup of solar thermal pyrolysis

Table 1: Technical specifications of the solar reflector.

Depiction	Values
Solar Thermal Reflector	
Category	Scheffler Dish
Profile	Parabolic
Surface area of the collector (m ²)	16
Number of dishes	1
Reflectivity (%)	92
Concentration	Focus
Concentration ratio(C.R.)	16:1
Reflecting surface	Mirrors with protective coating at black
Mounting location	Roof top

Table 2: Technical description of the pyrolysis reactor.

Description	Values
Pyrolysis Reactor	
Type	Conventional fixed bed reactor
Reactor material	SS 316
Reactor shape	Circular
Dia. (cm)	110
Width (cm)	12
Max. temperature (°C)	850
Max. pressure (bar)	5
Reactor feed capacity (kg)	18
Insulating material	Glass Wool

glass flask. As shown in Fig. 2c, the pyrolysed oil was collected from the reactor. This process is continuous for every lab scale operation. The entire time duration is approximately 4-5 hours for every batch reliant on the solar energy intensity. The range of 0.05-0.08 MW/m² for incident solar flux was observed at an average reactor temperature of 260-380°C. In case of normal direct irradiance (DNI) the range of 2.5-3.2 kW h/m²/day was observed.

As shown in Fig. 1, the schematic diagram of solar thermal parabolic dish can give an idea of the solar thermal reflection technology. The technical specifications of the scheffler dish, which has been installed in the project site are described in Table 1. An abrupt idea about the solar reflection and concentration can be developed from Table 1.

The conventional fixed bed reactor made of stainless steel has been installed in front of the solar reflector for the pyrolysis process. The full specification of the reactor are given in Table 2.

CHARACTERIZATION OF PYROLYSED PRODUCTS

Solar thermal pyrolysis is basically a way to generate energy from the sun for pyrolysis of biomass production. Proc-

ess which involves burning of substances at high temperature in absence of oxygen or oxidation environment. In short, it is thermochemical processing of biomass under inert atmosphere. Pyrolysis reactions are endothermic. Heating rate and residence time divide pyrolysis into three types: flash, fast and slow. Main challenges in biomass are its composition and related decomposition mechanisms. Different types of reactors, heating rate, residence time and present environment also have significant effects on the pyrolysis process. Mechanism of conversion of biomass constituents by pyrolysis involves breakage of bonds between the polymers and leads to release of volatile compounds and rearrangement reactions. This production is known as primary conversion and some unstable compounds can undergo further conversion called secondary conversion. Primary conversion includes char formation, depolymerization and fragmentation. Compounds formed in primary mechanism, unstable under reactor temperature can undergo further cracking or recombination. Volatile compounds break into lower molecular weight stable molecules by cracking.

Ultimate and proximate analysis: The karanja seeds have a very high volatile content of 72.25%, which was reduced to 31.5% drastically after the solar thermal pyrolysis. These parameters are based on Table 5. As the volatile content decreased after the pyrolysis, the fixed carbon increased significantly. It also shows the other elemental composition after the ultimate analysis and proximate analysis of biochar from different feedstocks.

Thermo-gravimetric (TG) analysis: Getting the knowledge about the pyrolytic zone of producing oil of karanja seeds, karanja biomass thermo-gravimetric analysis was carried out by using thermo-gravimetric (TG) analyser (EXSTAR TG/DTA 6300). To perform TG analysis the air flow rate of 200 mL/min was maintained. From the Fig. 3, the TGA and DTG values can be understood in a graphical representation. For this experiment, 10 mg alumina (Al₂O₃) and seeds powder was taken with the heating rate of 10°C/min from room temperature to 980°C.

Fourier transform infrared (FTIR) analysis: For the best possible outcomes and to monitor the key reaction type formations during the karanja seeds pyrolysis method, Fourier transform infrared (FTIR) spectroscopy was used. FTIR used was of the make of Perkin Elmer (Frontier FT-IR/FIR) with KBR as reference (Mawhinney & Yates Jr. 2001). From Fig. 4, it can be observed that the peak range is between 400 cm⁻¹ and 4000 cm⁻¹ with the resolution of 8 cm⁻¹.

GC-MS analysis: Gas chromatography-mass spectrometry (GC-MS) is used to categorize the variable components in the substance. The chemical compounds present in the pyrolysed oil can be determined by the GC-MS analysis

Table 3: FTIR spectra peaks obtained from karanja oil pyrolysis.

Peak range (cm ⁻¹)	Wavelength (cm ⁻¹)	Functional Group	Vibration type
3500-3200	3212.08, 3354.37	alcohol	O-H
3000-2800	2852.08, 2920.28, 3004.17	Amine salt	N-H
1712-1665	1711.80	α,β -unsaturated aldehydes, ketones	C=O
1470-1350	1464.02, 1403.85, 1378.44	Alkyne	Ca•C deformation
1300-950	1275.37, 1116.11, 966.69	C=O stretching and deformation	Ether and esters
900-650	744.13, 619.24	Mono and polyclinic substituted aromatics group	O-H

Table 4: Chemical compounds in karanja seed pyrolytic oil as determined by GC-MS.

Compound Name	Molecular Weight	Molecular Formula
4H-1,3-Benzodioxin	136	C ₈ H ₈ O ₂
3-(3-Methyl-3-Oxaziridinyl)Pyridine	136	C ₇ H ₈ ON ₂
1H-Pyrimido[5,4-E][1,2,4]Triazine-5,7-DI	270	C ₁₂ H ₁₀ O ₂ N ₆
Cyclooctylidene-(2-Phenylaziridin-1-YL)	242	C ₁₆ H ₂₂ N ₂
2-[3-[2-OXO-3-Oxazolidinyl]Propyl]-1,2-B	310	C ₁₃ H ₁₄ O ₅ N ₂ S
Spiro[Isobenzofuran-1(3H)-One-3,2'-Tetra	204	C ₁₁ H ₈ O ₄
3-Cyano-1,4,5-Trimethyl-2-Phthalimido-PY	279	C ₁₆ H ₁₃ O ₂ N ₃
Sydnone, 3-Phenyl	162	C ₈ H ₆ O ₂ N ₂
4H-Thiopyran-4-One, Tetrahydro-, 1-Oxide	132	C ₅ H ₆ O ₂ S
Tetrahydro-2h-Thiopyran-4-yLidenemalonon	196	C ₈ H ₈ O ₂ N ₂ S
Cyclohexanamine, N-(Benzoyloxy)	219	C ₁₃ H ₁₇ O ₂ N
(2E,4Z)-3-Methoxy-2,4-Hexadienedinitrile	134	C ₇ H ₆ ON ₂
Cyclooctylidene-(2-Phenylaziridin-1-YL)A	242	C ₁₆ H ₂₂ N ₂
4H-Thiopyran-4-One, Tetrahydro-, 1-Oxide	132	C ₅ H ₆ O ₂ S
10-Chloro-1-Decanol	192	C ₁₀ H ₂₁ Cl
Cyclodecane	140	C ₁₀ H ₂₀
1H-Imidazole, 2-Ethyl-4,5-Dihydro-4-Meth	112	C ₆ H ₁₂ N ₂
E-11-Tetradecen-1-OL Trifluoroacetate	308	C ₁₆ H ₂₇ O ₂ F ₃

only. From the Table 4, we can see the compound names, their formula and molecular weight. The GC-MS was of the Perkin Elmer make.

RESULTS AND DISCUSSION

The main source of energy for pyrolysis process is the thermal energy, which comes from either electrical heating or from coal burning, which are mainly non-renewable sources. Development of other technologies for energy, to use in biomass pyrolysis, is one area of research opportunity. There are several studies going on in this field for new as well as betterment of technology. In this paper, we have mainly looked at the status of functioning operation for pyrolysis using solar thermal energy. We have also looked at the trends and challenges associated with these for being better yielding processes. The overall performance analysis of the pyrolysed karanja oil has been described below.

Biomass Characterization

Some of the following methods can classify the characteri-

zation of biomass. After the characterization of the biomass and bio-oil, a clear knowledge can be derived, such as temperature zone of the oil production, functional groups present in the oil, presence of carbon content, hydrogen content, etc.

DTG and TGA of karanja seeds biomass: Fig. 3 shows that the DTG of karanja biomass lies between the temperature of 300°C to 680°C. Whereas, the TGA of the seeds lie between the temperature of 320°C and 790°C. By these data, it is clear that the pyrolytic temperature zone is from 280°C to 703°C for the karanja seeds biomass. It has been observed that the mass degradation is about 92% in the existence of air at 703°C, after which the thermal deprivation-turned out to be stable.

FTIR analysis of karanja seeds biomass: Fourier transform-infrared spectroscopy (FTIR) is used to identify organic as well as inorganic materials in pyrolysed karanja oil. This technique is used to measure the absorption of infrared radiation by the sample material versus wavelength. The infrared absorption bands identify the molecular components

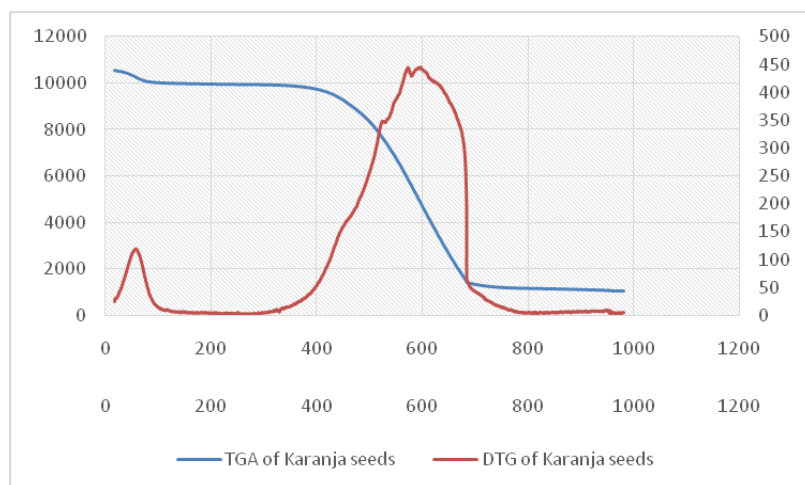


Fig. 3: DTG and TGA of karanja seeds.

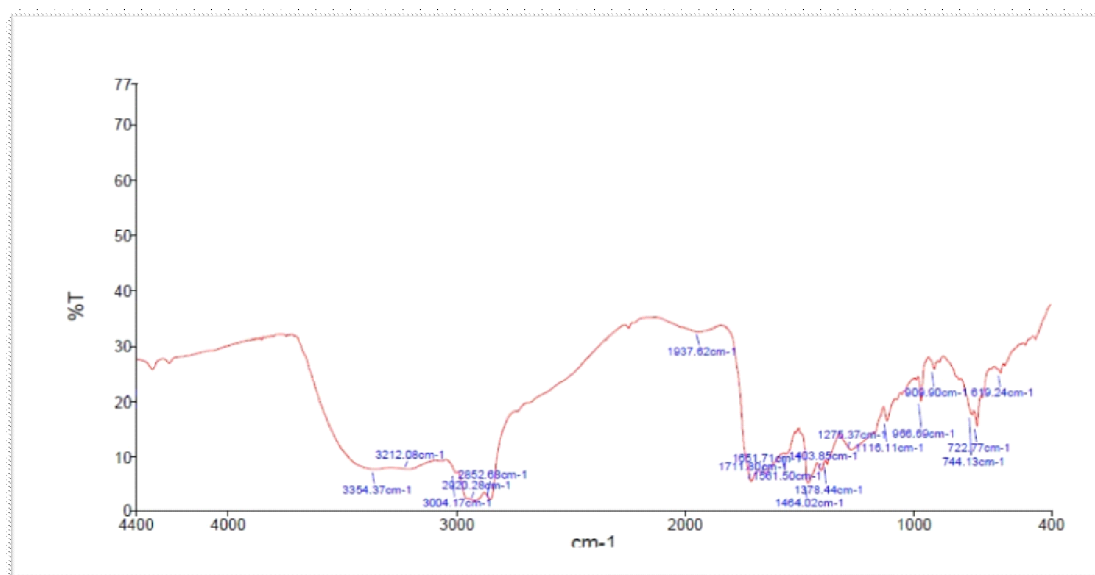


Fig. 4: FTIR analysis of karanja seeds oil.

and structures present in the oil.

Various FTIR spectra given in Table 3, confirm the presence of alcohol, alkane, aldehyde, ketones and alkene functional groups (which are crucial components of fuel to run the automotive engine) in the biomass and bio-oil.

Gas chromatography-mass spectrometer analysis of pyrolysed oil: Gas chromatography-mass spectrometry (GC-MS) is an analytical method that combines the features of gas-chromatography and mass spectrometry to identify different substances within the karanja oil.

CONCLUSION

Solar thermal pyrolysis of karanja seeds biomass was carried out in a pyrolysis reactor made of stainless steel, which produced pyrolysed products such as (i) bio-oil and (ii) biochar. Overall bio-oil yield was about 22% along with 55% biochar and 23% pyrolytic gas (though, the pyrolytic gas was collected as pyrolytic oil after condensing) at maximum. Though, the average oil content of biomass was about 14% due to substantial heat losses from the reactor (although glass wool used as an insulator), there was deficiency of

Table 5: Ultimate and proximate analysis of biochar from different feedstocks.

Features		Karanja seed (Present study)	Karanja biochar (Present study)	Jatropha biochar (Chintala et al. 2017)	Mohua biochar (Pradhan et al. 2016)	Castor biochar (Singh et al. 2014)
Proximate) analysis (wt. %	Moisture Content	15.1	0.0	2.3	5.14	-
	Volatile Content	72.25	31.5	36.4	36.17	-
	Ash Content	3.45	8.5	12.8	8.66	-
	Fixed Carbon	9.20	59.4	48.5	50.03	-
	Carbon (C)	51.69	61.57	71.14	78.12	76.82
Ultimate Analysis (wt.%)	Hydrogen (H)	3.24	6.83	3.89	4.12	-
	Nitrogen (N)	6.44	3.26	4.55	2.81	-
	Sulphur (S)	0.04	0.06	0.17	0.16	-
	Oxygen (O)	38.59	28.28	20.07	14.84	14.28
Gross Heating Value (kJ/kg)	-	53.50	60.50	16.15	26.05	28.93

catalysts and inappropriate solar reflection. The classification of these pyrolysed by-products was carried out as per the standards.

The following conclusions can be drawn based on the outcomes:

- The pyrolytic temperature zone is 280°C to 703°C for the karanja seeds biomass. It is observed that mass degradation is about 92% in the existence of air at 703°C after which the thermal deprivation became constant.
- FTIR analysis established the existence of alcohol, alkane, aldehyde, ketones and alkene functional groups (which are crucial components of fuel) in the biomass and bio-oil.
- The karanja seeds have a very high volatile content of approximately 72.25%, which was reduced to 31.5% drastically after the solar thermal pyrolysis. As the volatile content decreased after the pyrolysis, the fixed carbon increased significantly.
- As per the analysis performed in GC-MS and FTIR, it is understood that the main components like alkanes, alkenes and ketones are present in the pyrolysed bio-oil. Therefore, these pyrolysed oils have good possibility as a fuel for testing in engines. As per the ASTM standards, the fuel properties were found in order.

This experimental investigation shows that the application of solar thermal technology for the production of biofuels has the potential for future study. The non-uniform and less distribution of solar radiation into the pyrolysis reactor is the major drawback for the production of low oil yield. The quality of oil yield will be improved based on quantity and quality if the operating temperature does not

fluctuate frequently, proper insulation outside the reactor and proper usage of catalysts inside reactor. A hardcore research is needed on the reactor (cavity type) to make this technology successful.

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