

Bio-Oil Extraction from the Shells of Cocos Nucifera – A Source of Generating Renewable Energy and Its Analysis



M. Shireesha, Yasser Mirza Baig, C. Sarita, Syed Rashid Iqbal, Caroline Wesley, N. Vaishnavi

Abstract: Biomass is an important source of energy and fuel worldwide after coal, oil and natural gas. These fossil fuels do substantially more harm than renewable energy sources like biomass energy. Oil extracted from biomass is considered as an attractive option. In our project, we have specifically selected coconut shells as our feed as they are carbon-neutral, easy to store and abundantly available. Coconut shell also known as Cocos Nucifera shell in biological terms, once a discarded outer hardcover is now a product of great demand. Coconut shell charcoal is used as domestic and industrial fuel. This is obtained by various techniques. Initially, the shells are burned at high temperature and condensed to extract bio-oil using a series of unit operations and processes such as distillation, gas chromatography. These samples are then sent for analysis to compare them with the conventional fuel sources and then antimicrobial activity is examined. The medium-chain fatty acids in coconut oil have antimicrobial properties that can help protect against harmful microorganisms. Lauric acid and capric acid are known to have potent antimicrobial properties. Different bacterial cultures have been introduced later to test the ability of the oil to resist the harmful microorganisms and fungal cultures. Various analysis such as Infrared Spectroscopy, Gas-Mass Spectroscopy and Ultimate analysis are performed on the retrieved samples of oil extracted from the coconut shells. It is to be observed that the carbon content in the Cocos nucifera derived oil is less than the conventional diesel oil which makes it best for environmental uses.

Keywords: Biomass; Cocos Nucifera; Coconut Shell; Distillation.

I. INTRODUCTION

Petroleum products like diesel, naphtha, gasoline or valuable chemicals are employed in every aspect of life. Today, with rapid rise in world population, the demand for petroleum products is escalating day by day.

But the world's oil supply is fixed since petroleum is formed far too slowly respective to speed at which it is being extracted. As countries develop, advancement towards industrial and better living standards are approaching sustainable energy utilization.

Keeping in mind the growing environmental issues with the use of unsustainable resources, there is a widening interest in renewable energy sources such as hydro, wind, solar, biomass and geothermal energy.

A renewable resource, known as a stream asset, is a natural resource which will replenish to supplant the portion exhausted by usage and consumption, either through natural propagation or other recurring processes in a specific amount of time in a human time scale.

Renewable energy often provides energy in four alternative ways: electricity generation, air or water heating or cooling, transportation and grid.

On a global level, at least 30 nations have renewable energy resources contributing to 20% of total energy supply.

National renewable energy markets are projected to grow strongly with the coming decade and beyond. Countries such as Iceland and Norway, generate all their electricity using renewable energy already, and other countries have set a goal to succeed in employing 100% renewable energy in near future. While many energy projects are large scale, sustainable technologies are also suited to rural, remote areas and developing countries.

As most of the inexhaustible energy technologies provide electricity, renewable energy deployment is usually applied in conjugation with further electrification, which has several benefits. Additionally, electrification with these resources is more efficient and thus results in significant reduction in primary energy requirements. Biomass may be a biological material which springs from both plant and animal origin. It is the organic material which is not used for food or feed and in most cases used for energy and warmth production. Biomass is categorized by the source from which it is obtained. The three main categories of biomass are: forestry and wood processing residues, crop residues and animal wastes. Biomass is often converted with the aid of principle methods which include biological processing, thermo-chemical processing, combustion, gasification, liquefaction, alcohol fermentation and pyrolysis. Our research team has used pyrolysis process for extraction of bio-oil from biomass which are coconut shells. We have also analyzed the anti-microbial activity by introducing varied bacterial cultures.

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II. PRODUCTION OF BIO-OIL

A. Introduction

Biomass is defined as the material which can't be used as either food or feed for cattle. This material is employed in multiple ways like fertilizer, chemical manufacture and also energy production. These materials are often found mostly in food industries and waste from residential areas. In our project, we've selected coconut shells as our feed thanks to their wide availability from temples and households. [4]

B. Materials and Methods:

- Raw materials:** The source of collection can be from the most inexpensive places like temples, coconut water stalls, and home used coconuts. They were cleaned and sun dried. Then the cleaned shells were polished with sandpaper to make the surface of the shells clean.
- Chemicals:** Analytical grade-petroleum ether, chloroform and methanol were used as solvents in the chromatographic extraction techniques. Nutrient agar media was procured.
- Bacterial cultures:** Two bacterial cultures, *Escherichia coli* and *Staphylococcus aureus* were procured from microbiology. They were sub-cultured using growth medium that is the nutrient agar medium.

C. Feed Preparation

Huge amount of coconut is collected. The raw materials don't always have to be consumable but the shell should be used in any condition. Further the shells were ground manually to obtain small pieces which were used in the extraction of oil.



Fig 2.1 Raw Coconut Shell

D. Pre-Treatment of Coconut Shell

The collected coconuts are further dried for 2 days in the sun. This helps it to lose moisture, and the loss of moisture results in good yield of oil. [5] The pulp is scooped out using a scraper and even the coconut fibers are removed by filing from the coconut shell. We obtain a smooth surface on the exterior and the interior of the shell which gives a slight sheen to it.

E. Size Reduction of Feed

Size reduction is important as using a powdered form of shell yields a substantial amount of oil compared to using solid shells. The coconut shells are then grounded by jaw

crusher to obtain smaller pieces then, the particles go through a roll crusher to further reduce the size. Now the small pieces with larger surface area are sent into the ball mill for around 15 minutes to get fine particles and powdered shell. [6] Then it is sieved in accordance with particle size. The particles of size 2.8 mm - 4.0 mm are collected to be used as feed. A mixture of powdered shells and small particles is obtained as the final feed. [7]



Fig 2.2 Crushed Coconut Shells



Fig 2.3 Coconut Shell Powder

F. Proximate Analysis of Raw Material

A typical proximate analysis of a feed consists of moisture, ash, volatile matter, and fixed carbon contents. This fixed carbon is the material, apart from ash, that doesn't vaporize when heated in the absence of air. It is usually determined by subtracting the sum of three values: moisture, ash, and volatile matter in weight percent from 100 percent. [8]

For economic considerations it is important to remember moisture and ash contents of coal or other solid fuel source because these values don't actually contribute to the heating value of the fuel.

G. Ultimate Analysis of Raw Material

Ultimate analysis is used to determine the carbon, hydrogen, sulfur, nitrogen, ash, oxygen, and moisture contents of solid fuels. For specific applications, other chemical analysis may be used. These may involve identifying different forms of sulfur present.

Table 3.1: Elemental Analysis

ELEMENTS	AMOUNT
Carbon	53.7
Hydrogen	6.18
Oxygen	38.45
Nitrogen	0.88
Sulfur	0.04
Calorific value	19
Moisture content	6.98

Table 3.2: Component Analysis

COMPONENTS	AMOUNT
Volatiles	78.22
Fixed carbon	19.48
Ash	0.32

III. METHODOLOGY (TRIAL RUN)

This is a small procedure that is performed to confirm production of bio oil from the coconut shell.

- For the trial run the furnace is heated to a temperature of 800 Kelvin with a blower of capacity 0.5 hp. To the open furnace coal is added and is heated up to red hot.
- The temperature is maintained in between 600-650-degree Celsius and is kept under check with aid of IR thermometer.



Fig 3.1 Open Hearth Furnace

- Once desired temperature range was reached, the coconut shell powder was put into a container made of molding material.
- After introducing the feed into a container, we closed the top using a steel plate as the lid. The furnace temperature was noted to be around 610 degrees Celsius and this container was placed in the center of all the red-hot coal.
- After being in the furnace for around 3 minutes the fumes from the roasted coconut shell are observed. The lid is adjusted in such a way that a very little amount of vapor can escape and the maximum vapor is condensed after reaching the lid.
- It is evident that the oil is not obtained in liquid form but is scraped out from the lid covered. This is the evidence that shows the coconut shell had oil and can be extracted.
- The roasted coconut shell is further stored for experimental purpose and to find out if could be used for any other purpose. The roasted coconut shell is

further stored for experimental purpose and to find out if could be used for any other purpose.



Fig 3.2 Crucible in Furnace



Fig 3.3 Vapor Formation

IV. EXPERIMENTAL SET-UP

It is a simple experiment conducted in three different equipment: reactor, condenser and a beaker as shown below.

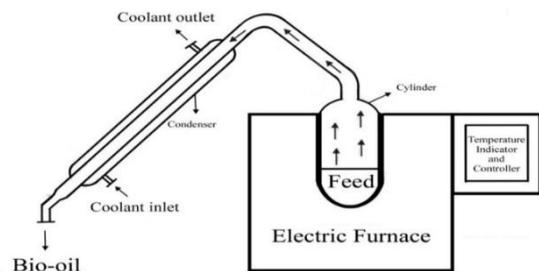


Fig 4.1 Experimental Setup

The reactor is made up of stain steel material of dimensions 15cm*7cm*10cm which can withstand a pressure of 300 psi. This specific reactor is used as it is ideal to handle the pressure and temperature needed for the experiment. [9] Addition of coal and wooden pieces according to the requirement is done and temperature is maintained with the aid of IR thermometer.

A. Experimental Procedure

- The 50% of the mixed feed was added to the reactor of the total volume (that is about 300 gm).
- The reactor was added to the condenser so that the vapor produced by the roasted shell is collected and condensed.
- The condenser and the reactor are both made up of stain steel and an isolating material is wrapped around it for efficient heat transfer.
- The reactor was is placed into the furnace and the condenser outlet outside the furnace towards the beaker.
- Ice water is used as the coolant in the experiment. Using water pump the water was sent into the condenser.

The set up was done as shown below:



Fig 4.2 Experimental Set-Up



Fig 4.3 Reactor in the Furnace

B. Experimental Conditions

The temperature was measured to be around 650 degrees Celsius and after 5 mins the first vapors started emitting from the condenser. The oil was dropping in a stream of steady droplets and some of the vapor was emitted as well. The droplets were collected below using a beaker.^[10] Cooling of the vapors was continuously done using ice water. After about 10 mins, the steady stream of droplets reduced to occasional droplet before seizing altogether.

This signified that our internal feed had been exhausted of the oil. After this point was reached, we took the entire apparatus out of the furnace and cooled it by placing it within a bucket of cool water. After the reactor had reduced to room temperature we detached the condenser from our reactor.^[11] Upon emptying the internal left-over feed, we obtained charred pieces of coconut shells which were very brittle. A sample of them is shown below:-



Fig 4.4 Residual Char

The charred pieces were completely emptied from the reactor. When introducing a feed of around 300 grams, we received charred material weighing around 220 grams.^[12] These charred pieces were sent to a dye removing plant. The plant used these charred pieces in their dye removal process.

C. Experimental Yield

From our first trial, using 300 grams of feed, we obtained around 60 ml of oil. Then we introduced more coal into the furnace and allowed it to reach sufficient temperature. The experiment was then repeated in the same manner as the first trial. We introduced an average of 300 grams of mixed feed into the reactor and obtained 55 to 60 ml of oil per trial. This whole procedure was repeated 3 times, so at the end, we had obtained around 165 ml of bio-oil and around 700 grams of charred coconut pieces.

Considering the formula to calculate yield as:

$$(\text{Weight of oil obtained} / \text{weight of feed used}) \times 100$$

Then the yield we would calculate would be:

$$(60/300) \times 100 = 20\% \text{ yield}$$

D. Bio Sample

As per trial of the experiment we extracted around 60 ml of oil from 300 grams of feed. So, in this case, the yield of the experiment can be calculated to give 20% yield for the process. The yield obtained is relatively low to medium amount of yield. We then plot a graph to show the amount of oil which we obtained versus the temperature at which the furnace was at. At each temperature, we tried to record an accurate measure of the oil obtained.

The graph was plotted using the following table values which we recorded during the oil formation process.

Table 4.1: Oil vs Temperature

Oil Obtained (in ml)	Temperature (in degrees Celsius)
0	0
1	100
3	200
5	230
7	250
10	300
16	400
21	450
30	500
40	575
40	575

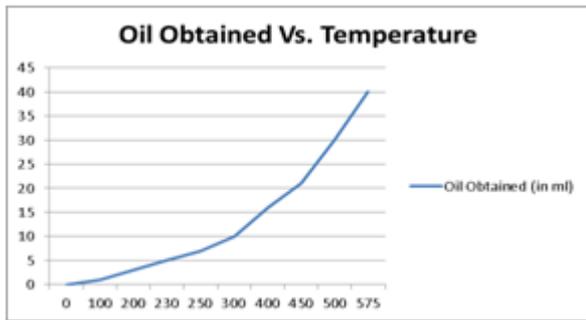


Fig 4.5 Oil vs. Temperature Graph

The oil sample which we obtained is shown below:



Fig 4.6 Bio-Oil Sample

The oil we obtained was very dark in color and it had a very pungent smell. As the oil was not a refined product, so our next step in the process was to purify the oil sample. The purification of the oil was done using distillation. Around 20 ml of the feed is taken separately and it goes through GC analysis so that we could identify what are all the various components that our oil comprised of and which components we desire to reduce during further purification process.

The extraction of the oil sample was the first half of the experiment and the second half comprised of purification of this obtained oil sample.

V. PURIFICATION OF BIO-OIL

A. Need for Purification

After we produced the bio-oil, aside from just the physical aspects we went for an elemental analysis of the oil and compared it to standard diesel to ascertain if our bio-oil really might be used as an alternate fuel. The comparison between the two is shown below:

Table 5.1:- Comparison of various oils

S.No	Element	Coconut shell Pyrolytic Oil	Diesel
1	Carbon	59.14	85.72
2	Hydrogen	3.47	13.2
3	Nitrogen	4.21	0.18
4	Sulphur	2.34	0.3
5	Oxygen	30.84	0.6
6	Empirical Formula	C4.93H3.44N0.3S0.07O1.93	C7.14H13.09N0.01S0.01O0.04

As you can see from the above table, our bio-oil has a far less carbon content than conventional diesel. The other components were also on par but the reason we decided to go for purification was due to the larger percentage of Sulphur in our bio-oil.

When we burn fuel, both carbon oxides and Sulphur oxides are given off as well as some other compounds but these two are found to be the foremost harmful to the environment. With the lower percentage of carbon, the oxides of carbon which would be released would also be less so that aspect is taken care of. Purification was required because we aspired to reduce the Sulphur content considerably as well so that Sulphur oxides would also be decreased. [14]

B. Distillation Set-Up Design

We proceeded to design our distillation set-up considering essential necessities to be examined. We concluded that our refining set-up need not be huge since we had a feed of 160 ml and we chose to distil just 80 ml feed for each refining. Remembering this, we chose to go for a more modest, versatile purifying set-up. This was discovered to be adequate to meet every one of our prerequisites and furthermore would help us from pointlessly having too enormous a mechanical assembly for a little feed. [15]

We planned the refining contraption to such an extent that we would have a feed jar of 250 ml. Heat would be provided to flask through methods for a warming mantle and because of low temperature prerequisites, we required a mantle that had a greatest temperature setting of 150 degrees Celsius. The flask had 3 openings with 2 of them being thermal wells utilized for temperature recording of the feed and fumes. The third opening was a valve framework utilized for introduction of the feed to the flask. On the vapor side, thermal well an additional opening was given for the joint to the condenser. The condenser was intended to be long and had a nozzle towards the end with the goal that the distillate could be gathered in a receptacle. This whole set-up was to be made of glass. [16]

After we obtained the set-up, we used vacuum grease at all the connections for a smooth fit. 2 pipes were attached to the inlet and outlet of the distillation column and all the connections were then covered with Teflon tape so that none of the vapors escaped and all the connections were secured.

The distillation apparatus after all the connections were done properly is depicted below:



Fig 5.1 Distillation Set-Up

C. Distillation Procedure

- The condenser inlet and outlet were then placed inside an ice bucket with a motor such that the water circulated exterior to the condenser.

- Once this was accurately working we introduced 80 ml of feed bio-oil into the flask.
- For the first distillation trial, we set the heating mantle at 80 degrees and allowed the distillation to take place.
- The bio-oil started bubbling at around 85 degrees Celsius and slight vapors were forming inside the flask. After around 4-5 minutes, the primary drop of distillate was obtained at a feed temperature of 94 degrees Celsius.



Fig 5.2 Distillation Feed Inlet Set-up

- With the internal oil having such high temperatures, if we had introduced a new sample of oil which was at room temperature to this, we would run the risk of cracking our feed flask or an explosion due to a sudden temperature change in which our feeds would be at different temperatures and would release uncontrolled energy.
- Due to this, during our distillation, we did not introduce more feed into our flask but rather we kept distilling the same feed sample. The distilled samples taken at different temperatures are shown below:



Fig 5.4 Sample at 80 degrees celsius

- After this first drop, the distillate slowly started dropping into our collection flask. After a while these drops seized and we obtained around 25 ml of distillate.
- The feed flask still contained quite amount of feed mixture. Now without adding additional feed we increased the mantle temperature to 90 degrees and continued with the distillation.
- With this increase in temperature, the oil was then heated further and a special distillate was obtained. This second distillate first drop was obtained at 102 degrees Celsius and around 10 ml of this distillate was distilled out.
- After this, in our last trial we set the mantle to 100 degrees Celsius and extracted last distillate sample. At this temperature, the oil was boiling intensely. No new feed was added to the present distillation trial. We got around 5 ml of distillate during this trial.

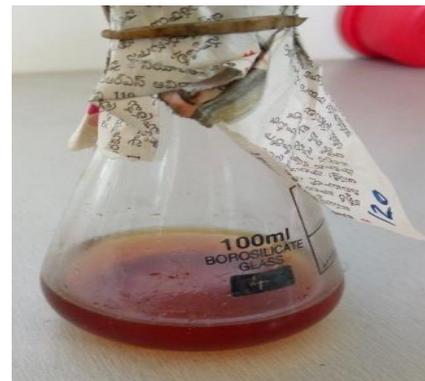


Fig 5.5 Sample at 120 degrees



Fig 5.3 Heating of Feed



Fig 5.6 Final Sample

- The mantle was then turned off and the oil was allowed to cool within the flask. When measuring the temperature of the oil using an IR gun we recorded oil temperatures of around 198 degrees Celsius. This was also one of the main reasons why we didn't introduce new bio-oil feed into our distillation feed flask.

D. Extraction of Oil

About 250 gms of ground shells were heated in the earthen pot for the span of three hrs giving a yield of 25cc of oil.

E. Fractionation of Oil

5 gms of crude oil was taken into the separating funnel with 20 ml of petroleum ether and was shaken vigorously for 5-10 mins. Yellow color development within the petroleum ether indicates extraction, which is then separated during a dish and therefore the solvent is then evaporated. This procedure is dispensed for a number of times till the batch of petroleum ether becomes colorless after shaking.

The residual oil in the separating funnel is then subjected to extraction procedure with chloroform. The remaining oil in separating funnel is dissolved in methanol since it is seen that the crude oil in methanol is immiscible.

All the three plates are kept for the evaporation of the solvent. We obtained yellow color extract from petroleum ether, black extract from chloroform and methanol and were named after petroleum ether.

VI. VI. ANALYSIS OF BIO-OIL

The analysis of bio-oil which is extracted by coconut shell is done in many methods which signifies the quality of bio-oil and allows us to further study about the properties of it. [17]

In this experiment we are applying as many as 3 methods to study the properties of Bio-oil.

1. Thermo-Gravimetric Analysis

Thermogravimetric analysis is a method of thermal analysis during which changes in physical and chemical properties of materials are measured as a function of accelerating temperature rate and as a function of time. The thermogravimetric temperature is plotted on x-axis and mass is plotted on the y-axis. Change in mass of our analyte is studied because of physical and chemical transition.

From the oil obtained after pyrolysis, the thermogravimetric analysis is performed for the temperatures 450,500,550 and 600°C. The yields obtained at these temperatures increases up to 575°C. A further increase in temperature to 600°C causes a steep decrease in the yield due to the temperature-pore distribution relation in the graph mentioned below:

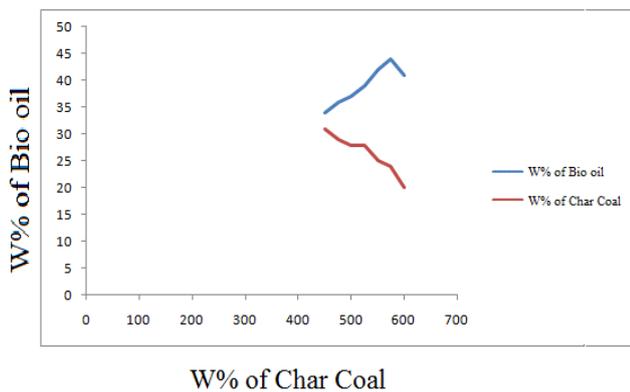


Fig 6.1 Temperature-Pore Distribution Relation

2. Gas Chromatography- Mass Spectroscopy

Gas chromatography (GC) is a common form of chromatographic technique utilized in analytical chemistry for separating and analyzing compounds which will be vaporized without decomposition

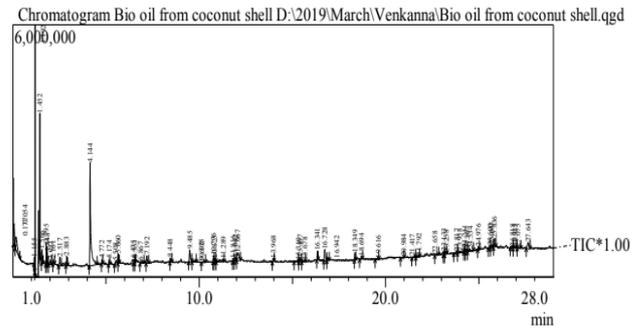


Fig 6.2 Retention times for feed

Above is actual retention time plot of the bio-oil. As you can see, numerous peaks were obtained for various different components. A table which shows all these various components is shown below.

Peak	R Time	Area	Area%	Height	Base m/z	Name
1	0.054	1791602	3.81	504098	44.00	1,3,5-Triazine-2,4-diamine, 6-chloro-
2	0.177	2282326	4.86	323333	44.05	Cyclononane (CAS) NONADECAN
3	1.155	642319	1.37	278376	44.00	2-Octanamine (CAS) 2-Octylamine SS
4	1.192	8313214	17.69	15748609	45.05	Ethanol
5	1.452	3329610	7.08	3410922	43.10	1-Propanol, 2-methyl- (CAS) Isobutyl
6	1.566	528530	1.12	321158	43.05	2-Propanone, 1-hydroxy- (CAS) Aceto
7	1.795	572669	1.22	524916	45.10	Ethane, 1,1-diethoxy- SS Acetaldehyd
8	1.848	792585	1.69	289946	41.05	1-Butanol, 2-methyl- (CAS) 2-Methyl
9	2.032	247927	0.53	116374	43.05	2-Butanone, 1-(acetyloxy)- SS 2-Butan
10	2.161	361944	0.77	106284	44.05	Formamide, N,N-dimethyl- (CAS) N,
11	2.517	552023	1.17	207851	96.00	1H-Pyrazole, 3,5-dimethyl- (CAS) 3,5
12	2.883	317438	0.68	225905	43.10	1-Butanol, 3-methyl- (CAS) 3-Methyl
13	4.144	8856970	18.85	2457853	94.05	Phenol (CAS) Lral SS ENT 1814 SS P
14	4.772	276431	0.59	106548	41.10	1-Heptanol, 6-methyl- (CAS) 6-Methyl
15	5.174	386818	0.82	120176	108.10	Phenol, 2-methyl- SS o-Cresol SS o-H
16	5.508	450490	0.96	75340	108.10	2-METHYLPHENOL SS
17	5.660	484238	1.03	230128	81.05	Mequinol
18	6.475	368731	0.78	93689	73.05	4-HYDROXY-2,5-DIMETHYL-3-HE

Peak	R Time	Area	Area%	Height	Base m/z	Name
19	6.553	305353	0.65	106545	44.05	Phenol, 2-((dimethylamino)methyl)-4-
20	6.867	444184	0.95	59280	44.00	3,4-XYLENOL SS
21	7.192	612136	1.30	183903	123.10	Phenol, 2-methoxy-4-methyl-
22	8.448	365985	0.78	109587	137.10	Phenol, 4-ethyl-2-methoxy- SS p-Ethyl
23	9.485	1159680	2.47	287555	154.05	Phenol, 2,6-dimethoxy- SS Pyrogallol
24	9.690	215522	0.46	32040	137.15	2(3H)-Furanone, dihydro-3,4-bis(4-H
25	10.125	292707	0.62	17193	44.05	Tricyclo[4.3.1.1(3.8)]undecan-1-amin
26	10.776	313144	0.67	147913	168.10	1,2,4-Trimethoxybenzene SS Benzene
27	10.825	329772	0.70	90172	164.10	EUGENOL SS
28	11.289	656126	1.40	60145	71.05	Propanoic acid, 2-methyl-, 2-ethyl-3-H
29	11.816	210186	0.45	80463	167.10	Benzene, 1,2,3-trimethoxy-5-methyl-
30	11.942	321424	0.68	84433	70.05	1,2-Cyclohexanediol
31	12.067	1411526	3.00	182789	55.10	5-Tridecene, (Z)- SS
32	13.968	204660	0.44	80235	194.00	Phenol, 2,6-dimethoxy-4-(2-propenyl)
33	15.310	288183	0.61	35798	73.05	Ethanedioic acid, bis(trimethylsilyl) e
34	15.407	336756	0.72	108080	43.05	3,7,11,15-Tetramethyl-2-hexadecen-1-
35	15.678	250079	0.53	41543	82.05	Ethanol, 2-(9-octadecenyloxy)-, (Z)- (
36	16.341	413974	0.88	216052	74.10	Tridecanoic acid, methyl ester
37	16.728	1228139	2.61	268595	43.10	n-Hexadecanoic acid
38	16.942	225660	0.48	47020	73.10	D-Glucuronic acid, gamma-lactone (f
39	18.349	372253	0.79	185493	74.05	Eicosanoic acid, methyl ester (CAS) A
40	18.694	197822	0.42	67264	41.10	Hexadecanoic acid (CAS) Palmitic ac
41	19.616	233135	0.50	33166	44.05	Heptanal SS n-Heptaldehyde SS n-He
42	20.984	211043	0.45	32943	44.05	Aminocarb SS Matalcil SS
43	21.417	207316	0.44	23809	40.00	HYDANTOIN, 1-N-FORMYL-5-HYD
44	21.792	232237	0.49	30120	44.05	3-Pyridinecarboxaldehyde, O-acetylfox
45	22.658	253621	0.54	32165	44.05	2-Amino-benzoyl fluoride SS
46	23.133	273749	0.58	83615	44.00	Stigmasta-3,5-dien-7-one (CAS) DEL
47	23.258	308285	0.66	45599	55.10	Cyclohexanecarboxylic acid, 1,3-dime
48	23.817	223091	0.47	38116	43.05	D-Streptamine, O-6-amino-6-deoxy-a
49	23.958	245615	0.52	33605	44.05	Heptanal SS n-Heptaldehyde SS n-He
50	24.241	213772	0.45	97160	69.10	2,6,10,14,18-Pentamethyl-2,6,10,14,1
51	24.367	204852	0.44	53112	73.05	3-Octanol, 3-methyl- SS Amylethylme
52	24.534	466386	0.99	46388	44.05	Androst-5-en-17-one, 3-methoxy-16,1
53	24.976	275460	0.59	78650	55.10	OLEIC ACID, PROPYL ESTER SS
54	25.600	214579	0.46	46089	41.10	5-Hepten-2-ol, 6-methyl- SS 6-Methyl
55	25.667	311726	0.66	67019	43.05	2-Propen-1-one, 1-(5,8-dihydroxy-2,2-
56	25.836	754047	1.60	196386	43.05	gamma-Sitosterol SS Stigmast-5-en-2-
57	26.818	202054	0.43	36218	43.05	3-N-HEXYL-DELTA-9-TETRAHYD
58	26.925	255860	0.54	34532	44.05	Heptanal SS n-Heptaldehyde SS n-He
59	27.075	218428	0.46	29361	55.10	Benzoic acid, 2-amino-, 4-methylcyclo
60	27.643	980756	2.09	151061	253.10	1-Octanamine, N-nitroso-N-octyl- (CA

Fig 6.3 Components in Feed Bio-Oil



As you can see, various components are present in the bio-oil, most of them being organic in nature. The main component which had the largest area percentage in our feed was ethanol. [18] Other components which have large area percentages are organic components which have various structures and no common name only a chemical formula name.

After seeing this retention time plot and components we then sent the distilled sample for comparison. The results of the distilled sample are shown below.

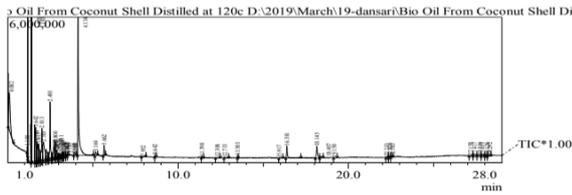


Fig 6.4 Retention Times for Distilled Sample

This retention time plot was for the distilled sample at 120 degrees Celsius. This sample was chosen because it had the best physical properties of all the distilled samples.

As you can see, there is a clear change in how the retention time plots look for the distilled sample and for the feed. The components of the distilled sample are listed below.

Peak	R. Time	Area	Area%	Height	Base m/z	Name
1	0.082	9607327	7.02	1516722	43.10	2,2-DIDEUTERO OCTADECANAL
2	1.150	876722	0.64	419450	44.00	Piperazine (CAS) R22 SS Uvilon SS A
3	1.183	7892295	5.77	14529508	45.05	Ethanol (CAS) Ethyl alcohol SS EtOH
4	1.405	40227724	29.41	21247859	43.05	Acetic acid (CAS) Ethylic acid SS Vin
5	1.642	5212793	3.81	1464373	45.00	Propanoic acid (CAS) Propionic acid
6	1.716	3113851	2.28	864779	45.00	3-hydroxy-2-butanone SS ACETONIN
7	1.817	3591362	2.63	764825	55.05	1-Butanol, 3-methyl- (impure) (CAS)
8	1.891	1610046	1.18	509102	43.05	Propanoic acid, 2-methyl-
9	2.013	5126347	3.75	1370930	57.00	1-Hydroxy-2-butanone SS 2-Butanone
10	2.103	5530640	4.04	794228	58.00	Heptanal, 2-methyl- SS
11	2.260	2513850	1.84	460700	43.05	Diethoxymethyl acetate SS Methanol
12	2.378	1523476	1.11	331076	43.05	Butanoic acid, 2-methyl-5-oxo-1-cyclo
13	2.493	6267264	4.58	2388728	96.00	2-Furancarboxaldehyde (CAS) Furfur
14	2.600	911572	0.67	272762	44.05	2-Propenamide, N-(1-cyclohexylethyl
15	2.711	2294237	1.68	536000	43.05	2-ETHYLBUTANAL SS
16	2.800	1927240	1.41	657886	43.05	2-Propanone, 1-(acetoxyl)- (CAS) Ac
17	2.872	1152920	0.84	329684	43.05	ISO-AMYL ACETATE SS
18	2.967	747228	0.55	219502	42.05	2H-Pyran-3(4H)-one, dihydro- (CAS)

Peak	R. Time	Area	Area%	Height	Base m/z	Name
19	3.042	504674	0.37	149269	43.05	Cyclohexane, 1R-acetamido-2,3Z-epo
20	3.108	492089	0.36	122536	43.05	Heptane, 2-(hexyloxy)- (CAS) ETHER
21	3.183	571033	0.42	146754	47.00	1-Tert-butoxy-6-trimethylsilyloxyhex
22	3.218	563670	0.41	239185	67.05	2-Cyclopenten-1-one, 2-methyl- SS 2-
23	3.276	251801	0.18	135532	95.05	Ethanone, 1-(2-furanyl)-
24	3.311	788049	0.58	183424	42.10	Butyrolactone
25	3.467	421446	0.31	90718	43.05	2,4-Pentanedione, 3-methyl- SS Meth
26	3.517	233028	0.17	59623	68.00	8-Azabicyclo[3.2.1]octane-2-carboxyl
27	3.931	520983	0.38	109156	53.05	Furo[3,4-b]furan-2,6(3H,4H)-dione, 4
28	4.134	22192176	16.23	7756036	94.05	Phenol (CAS) lzal SS ENT 1814 SS P
29	5.164	776723	0.57	179127	108.10	Phenol, 2-methyl- SS o-Cresol SS o-H
30	5.662	1141039	0.83	425642	109.05	Phenol, 2-methoxy-
31	7.952	162520	0.12	18893	44.00	Propanoic acid (CAS) Propionic acid
32	8.642	224931	0.16	95364	73.10	Cyclohexasiloxane, dodecamethyl- (C
33	11.398	257894	0.19	73675	73.10	3-Ethoxy-1,1,1,7,7-hexamethyl-3,5,7
34	12.308	162374	0.12	26659	44.00	2-(4,5-Dihydro-3-methyl-5-oxo-1-phen
35	12.733	318910	0.23	36696	71.05	Pentanoic acid, 2,2,4-trimethyl-3-carb
36	13.503	208993	0.15	109707	73.05	Benzeneacetic acid, alpha-,3,4-tris(t
37	15.917	158919	0.12	18817	44.00	2-Aminononadecane SS
38	16.381	1740628	1.27	505798	74.05	Pentadecanoic acid, 14-methyl-, meth
39	18.145	2316365	1.69	491010	55.10	9-Octadecenoic acid (Z)-, methyl ester
40	18.407	329977	0.24	54398	74.00	Eicosanoic acid, methyl ester (CAS) A
41	19.150	161240	0.12	15543	40.00	1-CYCLOHEXYL-ETHANOL SS
42	22.233	278064	0.20	49664	44.00	Formamide, N,N-dimethyl- (CAS) N,
43	22.426	272857	0.20	44106	44.00	Cyclohexanone, 3-hydroxy- SS 3-Hyd
44	22.583	202841	0.15	29754	44.00	Imidazole, 2-amino-5-[(2-carboxy)vin
45	27.158	253596	0.19	30383	40.00	6-Dimethyl(trimethylsilyl)silyloxytetra
46	27.417	252680	0.18	31979	40.00	1,2,4-Trioxolane-2-octanoic acid, 5-oc
47	27.739	259127	0.19	28746	40.00	Spiro[2.3]hexan-4-one, 5-chloro-6-me
48	27.833	201023	0.15	24759	40.00	Pyrazine, ethoxy- SS 2-Ethoxypyrazin
49	28.126	198852	0.15	31556	44.05	L-Asparagine SS Asparagine, L- SS (-
50	28.292	214002	0.16	23280	44.00	Tricyclo[4.3.1.1.3,8]undecane-3-carbo
		136759398	100.00	60015903		

Fig 6.5 Components in Distilled Sample

The component with the largest area percentage for the distilled sample is acetic acid/ethylic acid. Subsequent largest component is phenol.

Apart from this, once we went through the list of varied components of both the feed and therefore the distilled samples, many various components which were found within the feed sample were not found within the distilled sample. This occurred because of the distillation temperatures removing a number of the components as residue.

A. Chromatographic Separation

A 0.74 m long silica column was used for the separation of extracts obtained by solvent extraction. 5.5 gms of silica gel was taken and activated in hot air oven. 5 gms activated silica was soaked within the solvent that's the petroleum ether and 0.5 gms was mixed with the petroleum ether extract. The sample was run within the column by using the solvent. Petroleum ether and chloroform were initially washed then with decreasing concentration of chloroform and eventually with chloroform. The eluted samples were collected within the stopper tubes and named TT1, TT2 then on. The elution was simultaneously monitored by thin-layer chromatography and spots were observed and reported. TT2 showed one spot under UV rays and an I2 vapor chamber. TT3 was further purified by the flash column chromatographic method. Once the solvent is evaporated yellowish-orange emulsion is obtained.

B. Spectrometric analysis of the sample:

The samples were further subjected to IR spectroscopy and gas chromatography mass spectroscopy which has indicated the probable function group and molecular weight.

3. Ultimate Analysis of Bio-oil

In the table below, elemental composition of coconut shell pyrolytic oil is compared with conventional diesel.

Table 6.1:- Ultimate Analysis

S.no	Element	Coconut shell Pyrolytic Oil	Diesel
1	Carbon	59.14	85.72
2	Hydrogen	3.47	13.2
3	Nitrogen	4.21	0.18
4	Sulfur	2.341	0.3
5	Oxygen	30.84	0.6

From the analysis it can be observed that the carbon content in bio-oil is much less than the carbon content in diesel. The bio-oil however has higher Sulphur and oxygen content than what is desired. [19]

A. Anti-Microbial Activity

For checking the anti-microbial activity ditch plate method was administered since it is applicable for water-soluble and insoluble compounds.



In the ditch plate method, a ditch 1 cm *9 cm is cut from a nutrient agar plate then it's stuffed with 4ml molten nutrient agar mixed with 0.5 ml of sample oil. Culture suspensions of the bacterial cultures were streaked across the ditch. The plates were incubated at 37 degrees centigrade for 24 hrs. We observe the zone of clearance on and near the ditch for positive results.

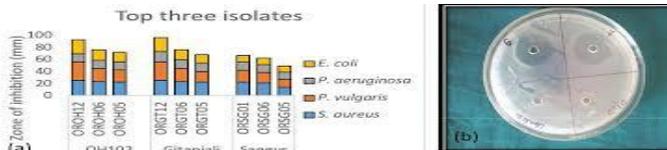


Fig 6.6 Anti-Microbial Study

In agar cup diffusion method, suspension is added to molten nutrient agar and is mixed well, then poured into a sterile Petri plate. After cooling, wells were made and 50 microlitres of petroleum ether extract of the oil was added with appropriate controls. [20]

VII. RESULTS AND DISCUSSIONS

A. Infrared Spectroscopy Analysis

The IR study indicates the presence of alkanes, alkenes, alkynes and acid groups for petroleum ether. The absorbance of bio-oil is greater than the diesel.

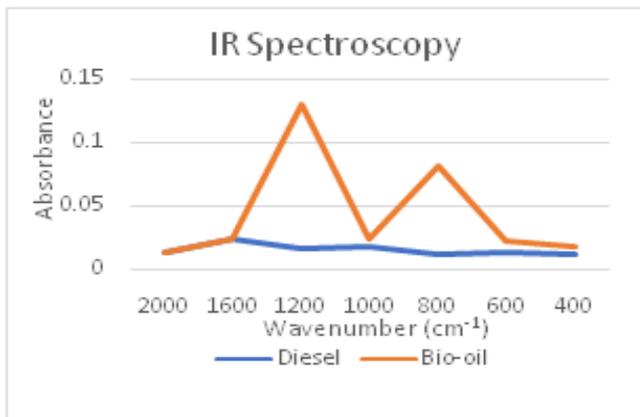


Fig 7.1 IR Spectroscopy Graph

B. GC-MS analysis

The samples are collected and subjected to gas chromatography-mass spectroscopy. It was found that the samples are mixture of molecules having close structural resemblance and molecular weight.

Table 7.1 Chemical Elements found in Bio-oil

S.No	Retention time	Area%	Name
1	1.183	5.77	Ethanol
2	1.642	3.81	Propanoic acid
3	1.716	2.28	3-hydroxy-2-butanone(Acetoin)
4	2.013	3.75	1-Hydroxy-2-butanone(2-Butanone)
5	2.103	4.04	Heptanal
6	3.042	0.37	Cyclohexane
7	3.108	0.36	Heptane
8	3.276	0.18	Ethanone
9	3.311	0.58	Butyrolactone
10	3.467	0.31	2,4-Pentanedione (Acetylacetone)

The various chemicals found in distilled bio-oil sample had got these retention times and the area space. The GC-MS provides us ample opportunity to study about the components present in the bio-oil which will be eventually responsible for the combustion or ignition.

VIII. CONCLUSIONS

The samples were tested for their anti-microbial activity. Petroleum ether is effective on Staphylococcus aureus which is responsible for skin infections. It also gave a satisfactory result for Escherichia coli (part of intestinal flora) though the shell oil gave lesser inhibition. [21]

We can use petroleum ether extract as an alternative for the further tests that needs to be done and purify the component that is accountable for inhibitory action. The usage of fuels and crude oils has reached its apex and the demand for more oil only increases every day but on the same scale, the levels of pollution due to usage of these fuels has also increased tremendously. [22] Not only do these fuels pollute our environment, they are also in limited supply and regenerate at an extremely slow rate. With the current usage of these fuels, their supply diminishes and these fuels will not sustain us in the future.

An alternative fuel needs to be manufactured which not only causes less pollution than conventional fuel but also needs to be formed from a source which will not be depleted easily but can be found virtually anywhere. Such an alternative is bio-oil which is produced from coconut shells. This bio-oil is made from organic biomass and has favorable properties when it comes to the pollution which is given off when it is burned. [23] Apart from this, biomass is easily available and waste issues are taken care of.

The most probable application of bio-oil includes the induction of fuels in boilers, engines, turbines for heat and power generation. Bio-oil can be further converted to transportation fuel by introducing significant reforming processes. Recent studies have shown that bio-oil is used vitally for soil conditioning, additives in fertilizer and pharmaceutical industries. Bio-Char filters are being used in a small scale to provide certain communities with clean and fresh water. Apart from the above mentioned fundamental applications there is an immediate need to manufacture bio-fuels on a commercial scale.

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