KERONE
Kerone Engineering Solutions Limited

EXTRACTION OF ESSENTIAL OIL FROM LEAF & BIOCHAR PRODUCTION FROM WOOD OF *EUCALYPTUS SPP.*

In Association with SVCH-Technologii, Moscow (Russia)

ISO 9001:2015 | ISO 14001:2015 | ISO 45001:2018

ABOUT US

KERONE is now renowned for serving the specialized needs of customers with the best quality and economical process of Heating /cooling and drying products, manufactured in a high-quality environment by a trained and qualified workforce (special purpose machinery)

KERONE is a pioneer in application and implementation engineering with its vast experience and team of professionals.

KERONE is devoteded to serve the industry to optimize its operations both economically and environmentally with its specialized heating and drying solutions.

KERONE is having immense expertise in manufacturing and implementing various types of engineering solutions.

KERONE is possessing employee strength of more than 280+ experts continuously putting efforts for happy industrial engineering solutions.

WHY CHOOSE US

> Enhance the value of customer operation through our customer need centric engineering solution.

With decades of expertise, cutting-edge technology, and a customer-centric approach, Kerone Engineering offers tailor-made heating solutions that prioritize quality, flexibility, and cost-effectiveness. Benefit from our commitment to excellence, post-sales support, and innovative solutions for your unique heating needs. Choose Kerone Engineering for reliability, performance, and unmatched value.

MISSION

- To enhance the value of customer operation throughi our customer need centricengineefing solution
- We are cominitted to provide our customers, unique arid best in class products in Industrial heating drying and cooling segment with strategic tie-up for the technical know-how with renowned leader in the industry specific segment

VISION

- Turn into a world leader in providing specialized, top-notch quality and ecological industrial heating, cooling, and drying solutions across the globe.
- To attain global recognition as the best of quality and environment-friendly engineering solution company.

Extraction of essential oil from leaf and biochar production from wood of Eucalyptus spp.

Objectives

Optimization of extraction of essential oil from Eucalyptus using Hydro distillation

Optimization of biochar production from spent leaves and wood of Eucalyptus

Hydro distillation of Eucalyptus spp. leaves

Table.1.1 – Total number of runs carried out for the hydro distillation process

Runs	Moisture (%)	Particle Size	Ratio (Sample: Water)	Wt. of sample(g)	Volume of solvent (ml)	Temp(°C)
1	MCs	1 x 1	1 : 5	20	100	100
2	MCs	1 x 1	1 : 3	33.3	100	100
3	MCs	powder	1 : 5	20	100	100
4	MCs	powder	1 : 3	33.3	100	100
5	MCas	1 x 1	1 : 5	20	100	100
6	MCas	1 x 1	1 : 3	33.3	100	100
7	MCas	powder	1 : 5	20	100	100
8	MCas	powder	1 : 3	33.3	100	100

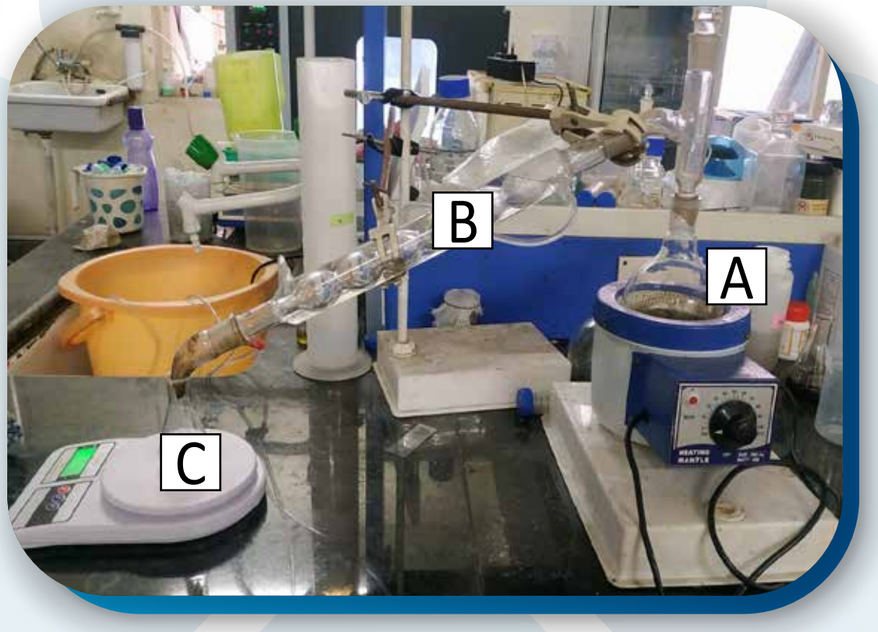


Figure.1.1 – Simple water distillation setup

*MCas and MCs refers to moisture content of as received (44.24%) and solar dried (11.1%) samples respectively Particle size 1 x 1 and powder refers to 1 cm x 1 cm and 300-600 micron respectively

Simple distillation of Eucalytpus spp. leaves has been carried out. Flask “A” – sample + water. The flask A is heated at 100 °C and the vapor is generated and it is sent to the three way tube. Then it reaches the condenser “B” where the vapors condense to water and it is collected in conical flask “C”

Results and Discussion

Table.1.2 – The volume and yield of distillate collected from Eucalyptus leaves

Runs	Volume of distillate (ml)	Mass of distillate (g)	Density (g/ml)	Yield (%)	Useful heat (kcal)
1	67.50	67.34	0.99	6.04	0.42
2	67	65.38	0.97	3.51	0.69
3	62.50	60.86	0.97	5.4	0.42
4	41	40.02	0.97	2.15	0.69
5	85	85.50	1.00	7.60	0.43
6	88	87.93	0.99	4.73	0.69
7	71	69.5	0.97	6.23	0.43
8	52	52.60	1.01	2.83	0.69

- Hydro distillation was carried out until evaporation of solvent is complete to preserve the fragrance of oil
- Powder sample had less distillation time compared to the 1x1 sample. Owing to space occupancy by the 1x1 sample is high compared to powder sample, the rate of evaporation of the liquid/ solvent phase is affected leading to difference in the volume of distillate collected and the yield.
- The distillate collected gives the good fragrance but it is not equal to the commercially available oil.
- Separation of oil-water mixture enhances the oil concentration and its quality

Hydro distillation of Eucalyptus spp. leaves

To understand the effect of moisture content, ratio and particle size, volume of distillate at constant time of 40 min has been collected

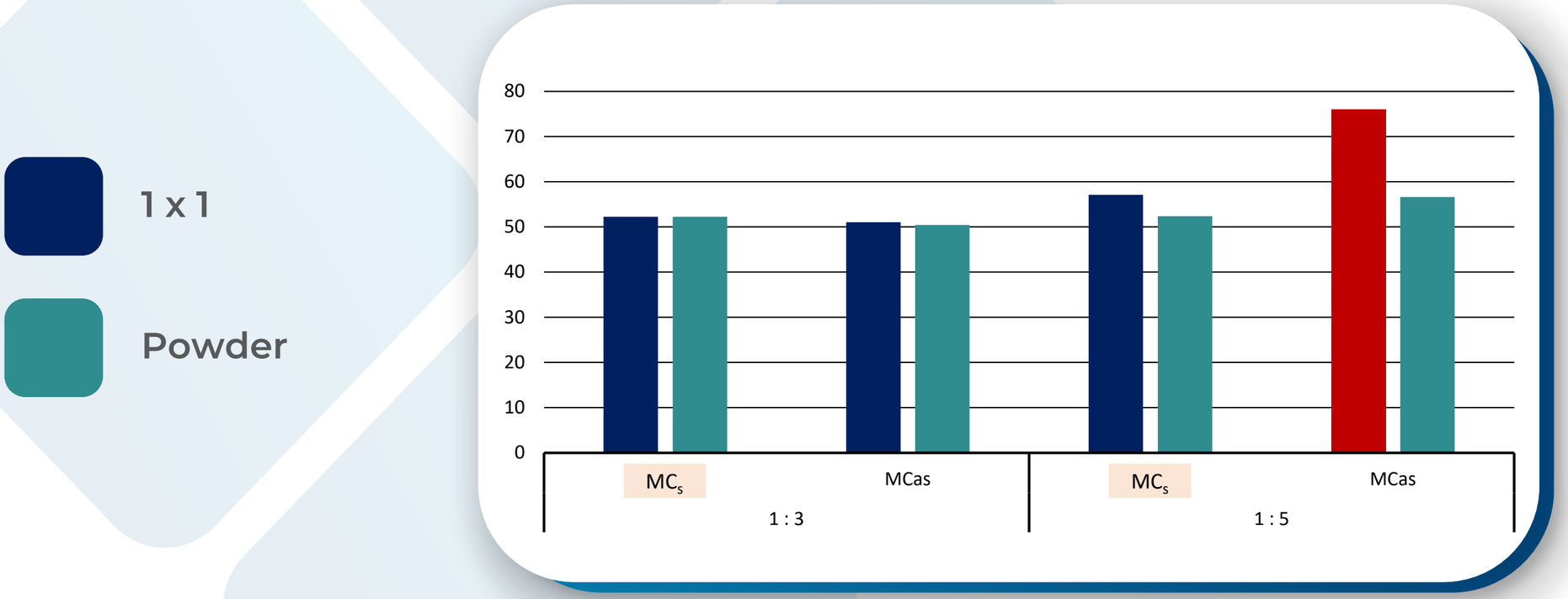
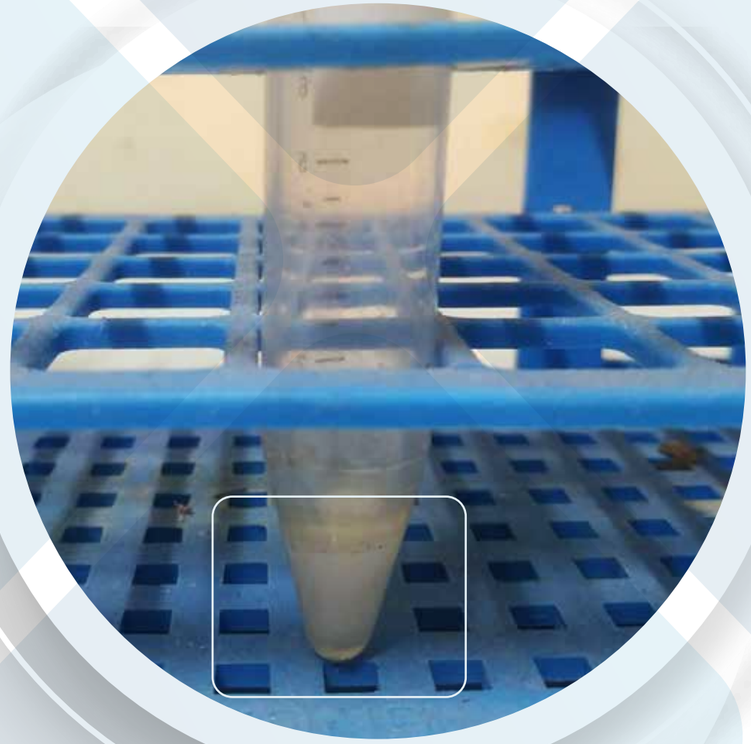


Figure 1.2 – Effect of moisture content and ratio on distillate volume at 40 min.

Optimal parameters for higher essential oil yield: MCas (44.24%) Particle size: 1x1, Sample: water ratio: 1:5. This gives the volume of distillate as 76.05 ml per 20 g sample.

Hydro distillation of Eucalyptus spp. leaves

Sodium sulphate precipitation procedure is being followed to dehydrate the distillate for oil separation



- Sodium sulphate acts as the dehydrating agent to separate oil from distillate.
- Anhydrous sodium sulphate salt is added and this lead to the solidification of sodium sulphate by absorbing the water.
- However, negative response has been recorded.

Figure.1.2 – Separation of oil using Sodium Sulphate Dehydration

Hydro distillation of Eucalyptus spp. leaves

Phase transfer method using chloroform has been carried out to separate oil into organic phase

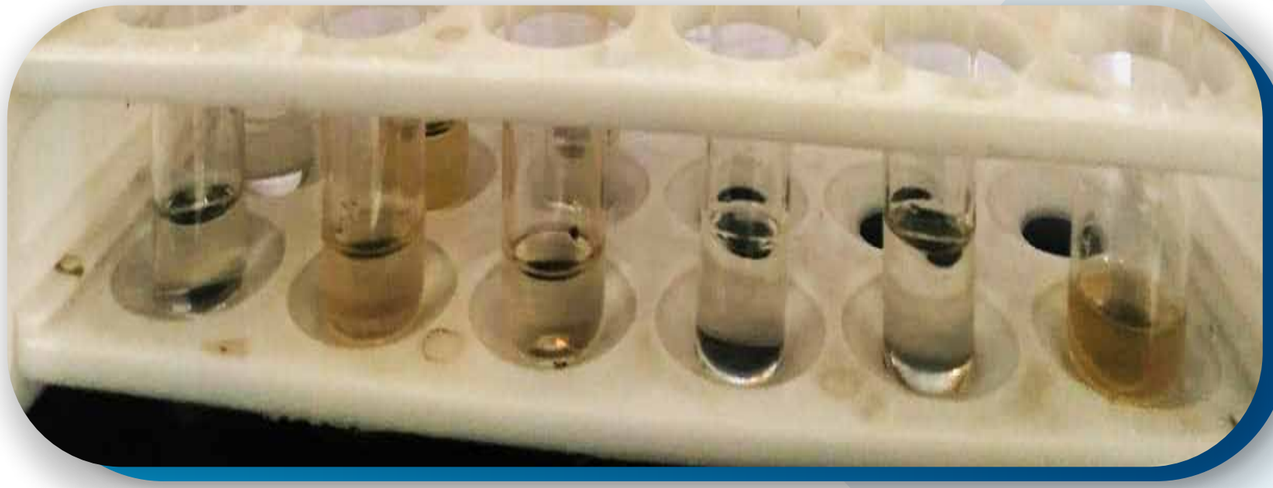


Fig. Separation of oil through chloroform phase transfer

- The Phase transfer method is the transfer of particles between two immiscible phases.
- Chloroform has been added to extract oil from distillate in the ratio of 1:1.
- Two distinct separate layer has been observed however there is the loss of fragrance of separated oil.

Table – Yield of the oil separated by Phase Transfer Method

Run Number	Yield of oil obtained (%)
1	40
2	41
3	40
4	44
5	45
6	36
7	40
8	44

Hydro distillation of Eucalyptus spp. leaves

Estimation of saponification number to determine fatty acid content in essential oil

- Saponification number is the amount of KOH consumed by 1 g of fat.
- The greater the number the more shorter and medium chain fatty acids are the oil contains.
- Here the results obtained are very low and which indicates the presence of longer fatty acid chain and higher molecular weight.
- This value doesn't have any range it is based on the oil/fat we use.

Table.1.5– Saponification value of Eucalyptus oil

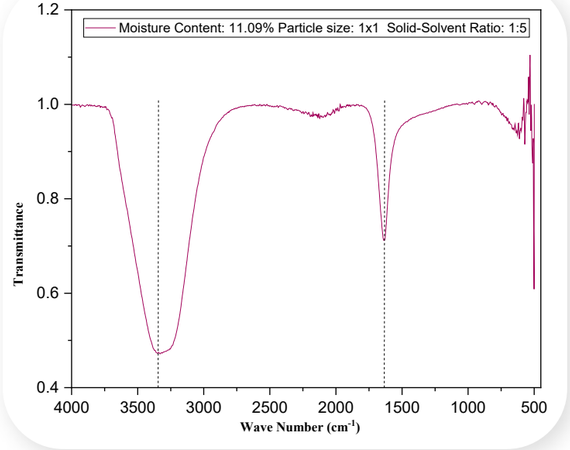
Runs	Saponification number
1	0.1736
2	0.168
3	0.224
4	0.5656
5	0.756
6	1.2208
7	0.1568
8	0.1344

Hydro distillation of Eucalyptus spp. leaves

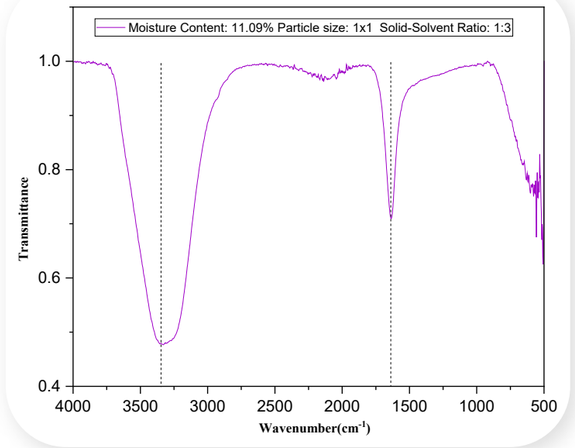
Fourier transform infrared spectroscopic analysis to observe functional groups in essential oil

Peaks at	Functional groups	Compound
1641 cm^{-1}	C=C stretching	β-pinene

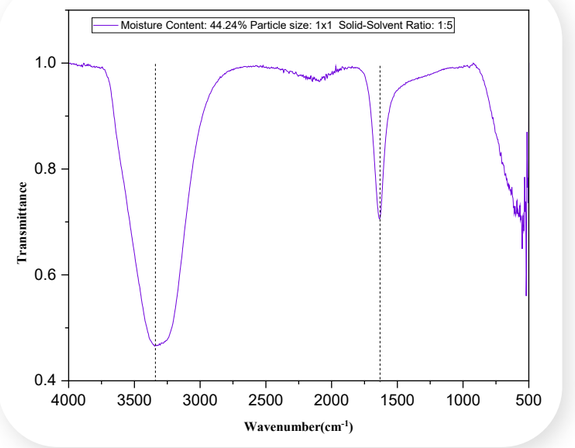
Run 1



Run 2



Run 5



Run 6

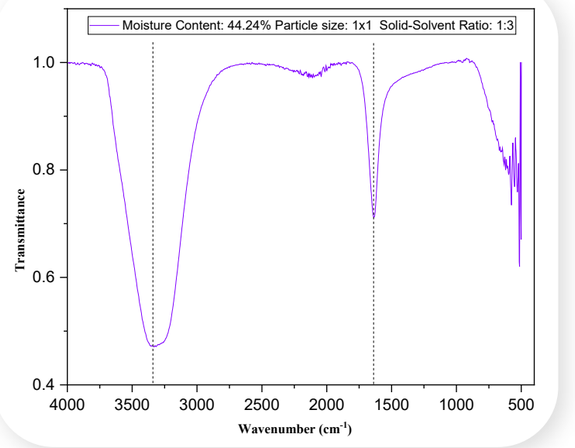
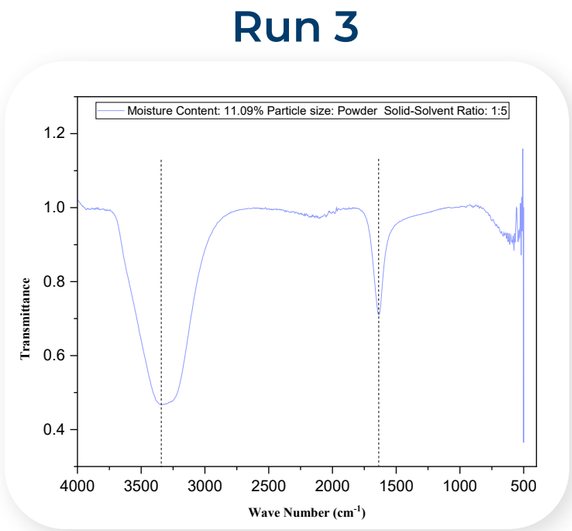
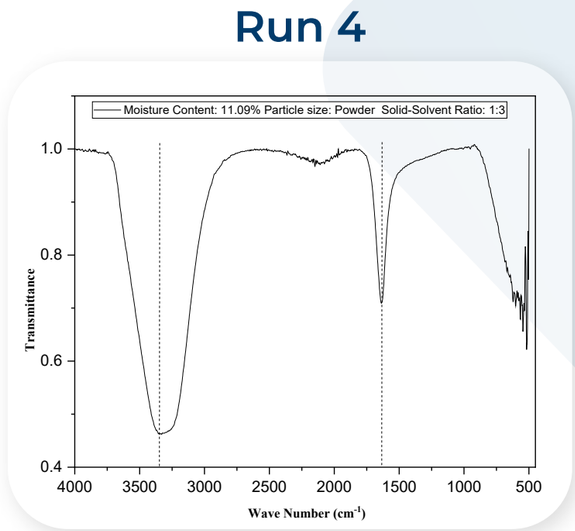


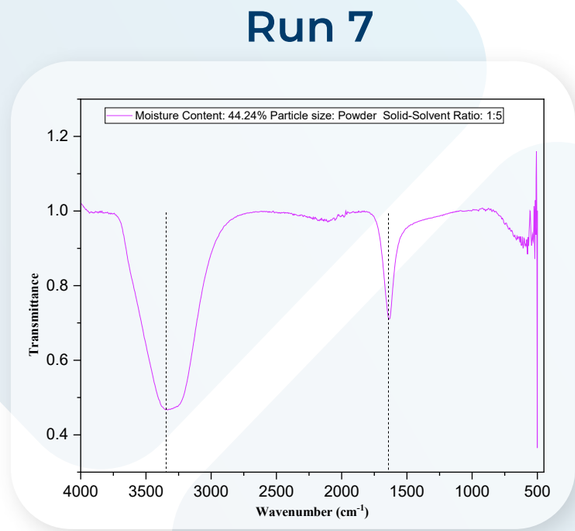
Figure.1.5 – FTIR results of 1x1 sample

Hydro distillation of Eucalyptus spp. leaves

Peaks at	Functional groups	Compound
1641 cm^{-1}	C=C stretching	β-pinene







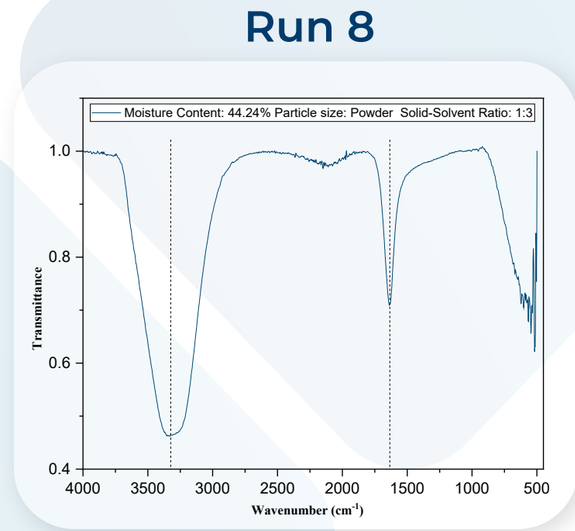


Figure.1.6 – FTIR results of Powder sample

Hydro distillation of Eucalyptus spp. leaves

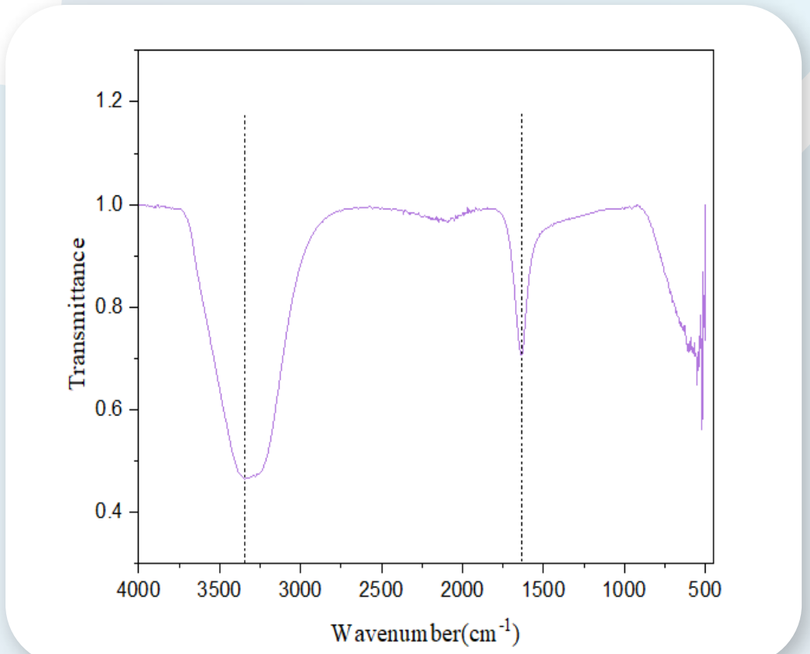


Figure.1.7 – FTIR of Run 5

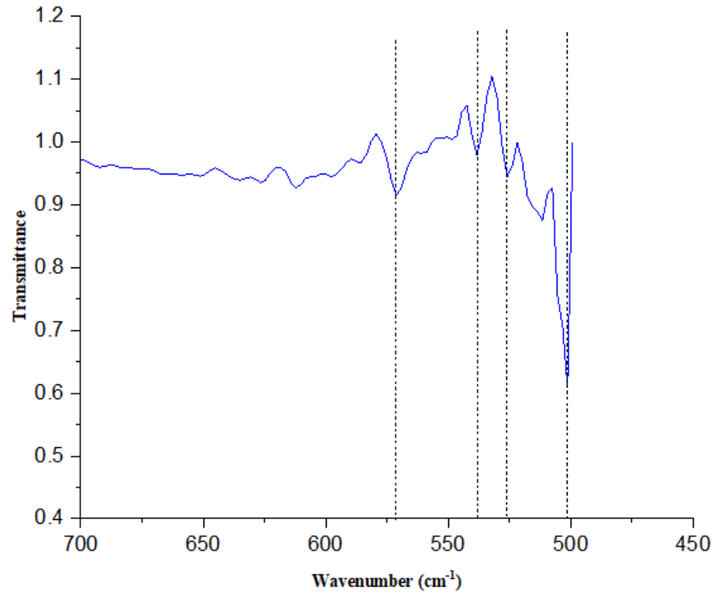
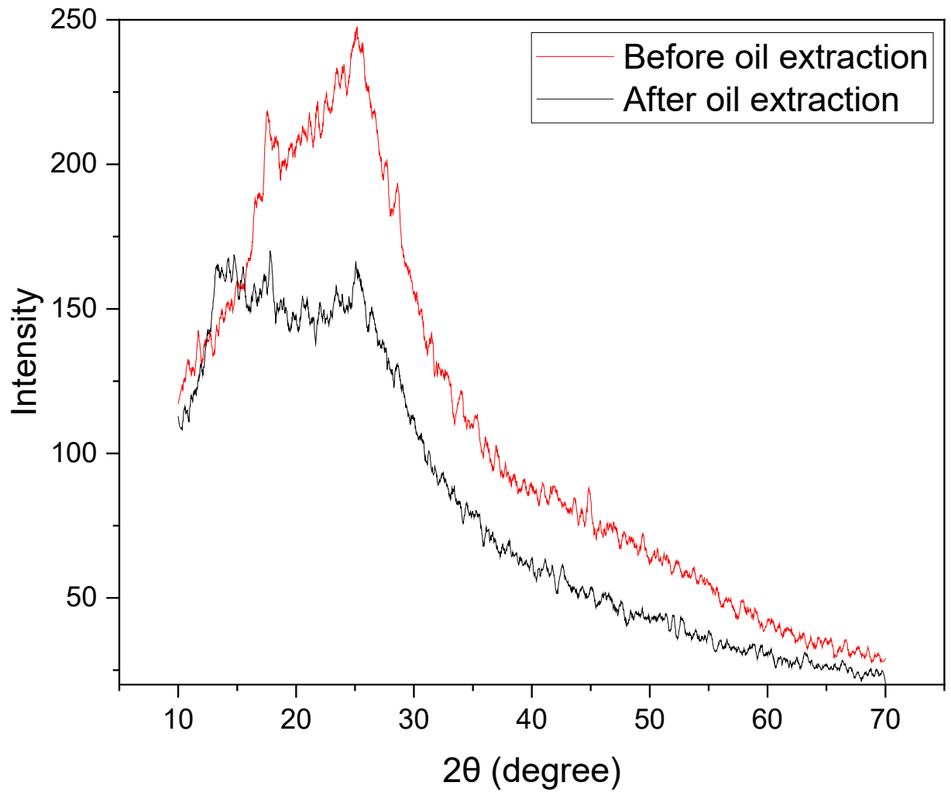


Figure.1.8 - Finger print region of Run 5

- Higher presence of β-pinene compounds however its intensity is similar. Variation in intensity 1,8 cineole compounds are observed .
- Peaks at 1641 cm-1, 3352 cm-1 corresponds to C=C stretching of β-pinene, O-H stretching corresponding to citronellol, isopulegol, and neo-isopulegol.
- Peaks at 501 cm-1 – 570 cm-1 corresponds to 1, 8 cineole compounds

Hydro distillation of Eucalyptus spp. leaves

X-ray diffraction analysis to observe changes in structures of spent leaves



- After the extraction of oil, change in crystallinity of leaf has been observed.

Figure.1.14 :
XRD data of the before extraction and after extraction sample

Soxhlet extraction for oil from Eucalyptus spp. leaves

Methodology

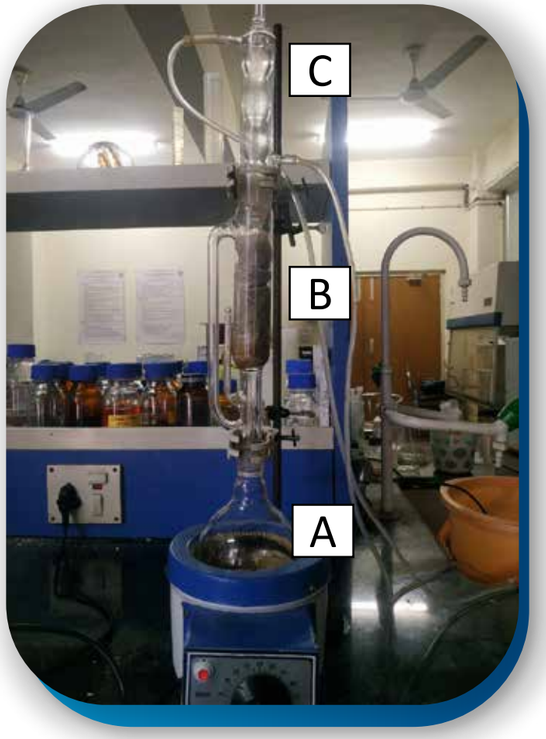


Figure.1.4 – Soxhlet Extraction setup

Table.1.6 – Total number of runs carried out with Soxhlet Extraction

Runs	Size	Moisture content	Weight of the sample (g)	Volume of solvent (ml)	Ratio	Run time (hr)
1	1 x 1	MCas	20	100	1 : 5	2
2	1 x 1	MCas	33.3	100	1 : 3	2
3	1 x 1	MCs	20	100	1 : 5	2
4	1 x 1	MCs	28.03	100	1 : 3	2
5	powder	MCas	20	100	1 : 5	2
6	powder	MCas	33.3	100	1 : 3	2
7	powder	MCs	20	100	1 : 5	2
8	powder	MCs	33.3	100	1 : 3	2

*MCas and MCs refers to moisture content of as received (44.24%) and solar dried (11.1%) samples respectively
Particle size 1 x 1 and powder refers to 1 cm x 1 cm and 300-600 micron respectively

Soxhlet extraction is used for the liquid-solid extraction when the compound has the limited solubility in solvent. Here the solvent we used is water. All the eight runs were done for two hours with the same parameters that are considered in simple distillation. The sample is loaded in thimble which is then place in the extraction column “B”. The solvent is place in round bottom flask “A”. The main focus here is to reflex the solvent used with the help of condenser “C” and to increase the extraction time.

Soxhlet extraction for oil from Eucalyptus spp. leaves

Table.1.7 – Yield and volume of extract collected for 8 runs

Runs	Weight of the extract (g)	Volume of the extract (ml)	Weight of the spent leaves (g)	Density (g/ml)	Yield (%)	Avg pH
1	78	79	31.00	0.99	7.00	5.09
2	72	75	61.00	0.96	3.88	4.91
3	58	60	44.97	0.98	5.29	5.09
4	54	53	61.21	1.02	3.46	4.90
5	73	76	39.31	0.96	6.55	4.64
6	54	54	56.24	1.00	2.91	4.66
7	58	58	42.80	0.99	5.20	4.99
8	19	17	71.50	1.12	1.02	4.60

- The Extracts we obtained is in crude form which has no fragrance.
- Here also the particle size of 1x1, ratio of 1:5, moisture content of MCas gives the higher yield
- The pH was checked for all the eight runs and it lies between the range of Eucalyptus Essential oil's pH

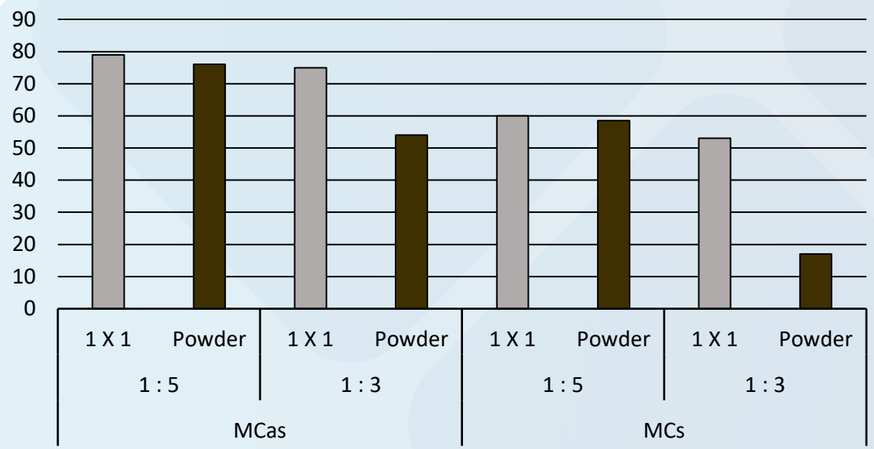


Figure: Effect of moisture content, particle size and ratio on extract yield.

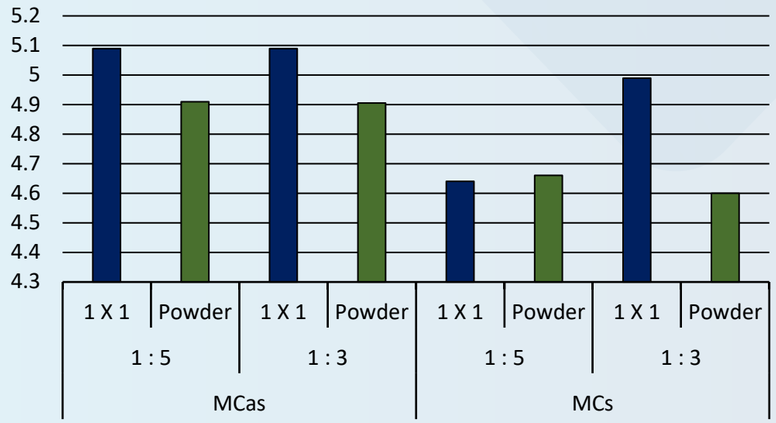


Figure: Effect of moisture content, particle size and ratio on extract pH.

2. PYROLYSIS OF EUCALYPTUS WOOD

Figure.2.1 :
Pyrolysis setup

Figure.2.2 :
Bio- oil obtained from pyrolysis

Figure.2.3 :
Biochar obtained from pyrolysis

Modelling the yield and composition of products from Eucalyptus wood pyrolysis

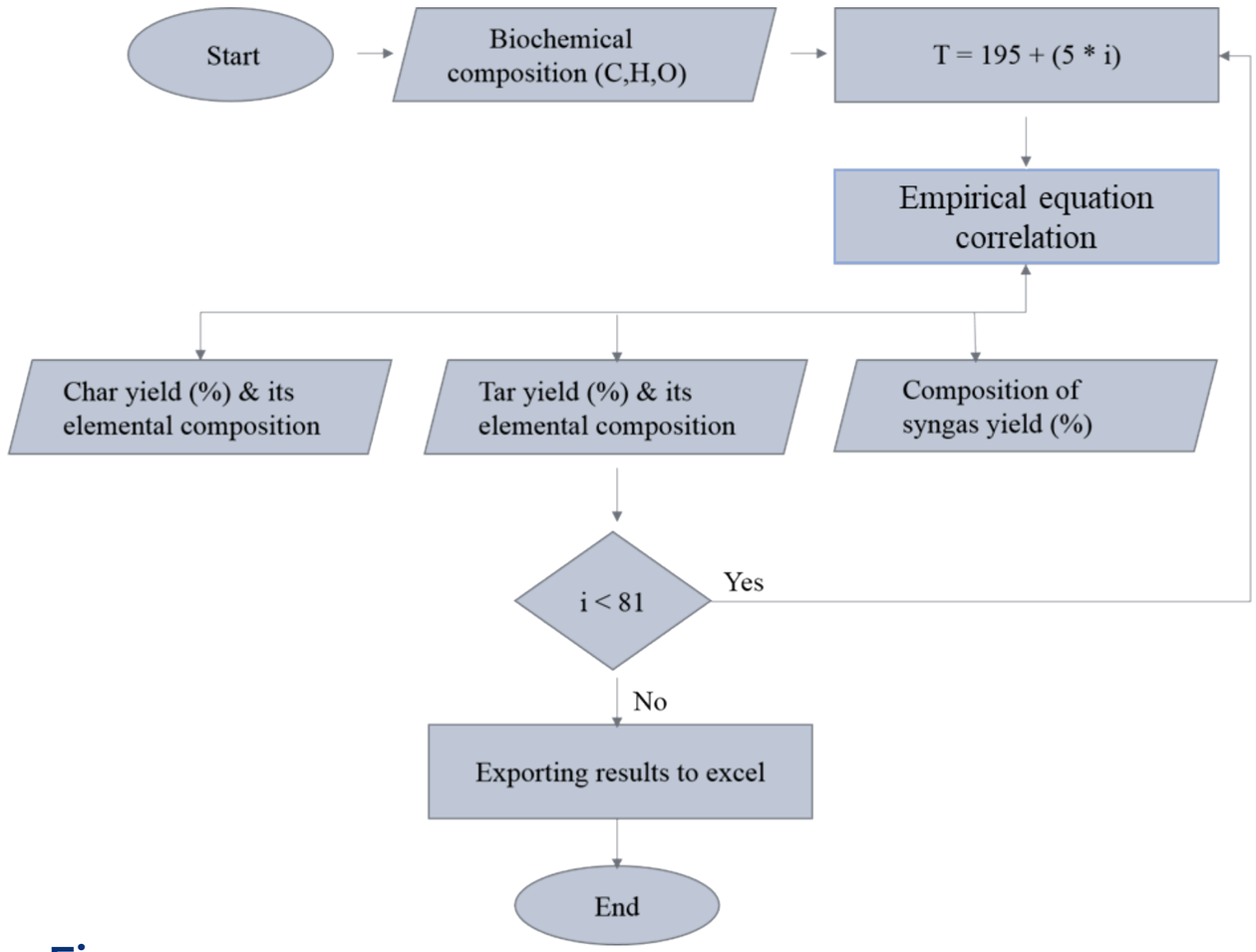


Figure:
Flowchart of MATLAB coding to obtain pyrolytic product composition

Empirical equations were used in MATLAB platform to predict the individual pyrolysis products

Model assumptions:

- Drying and devolatilization takes place at entry of biomass itself from inlet
- Overall reaction will be carried out under inert conditions with nitrogen
- Gasification and pyrolysis of carbon takes place under isothermal condition
- Biomass is well mixed
- Particle size of the feedstock is uniform

Yield of char, Y_(ch,F) = 0.106 + 2.43 * exp (-0.66 * 10^(−2)T)

Modelling the yield and composition of products from Eucalyptus wood pyrolysis

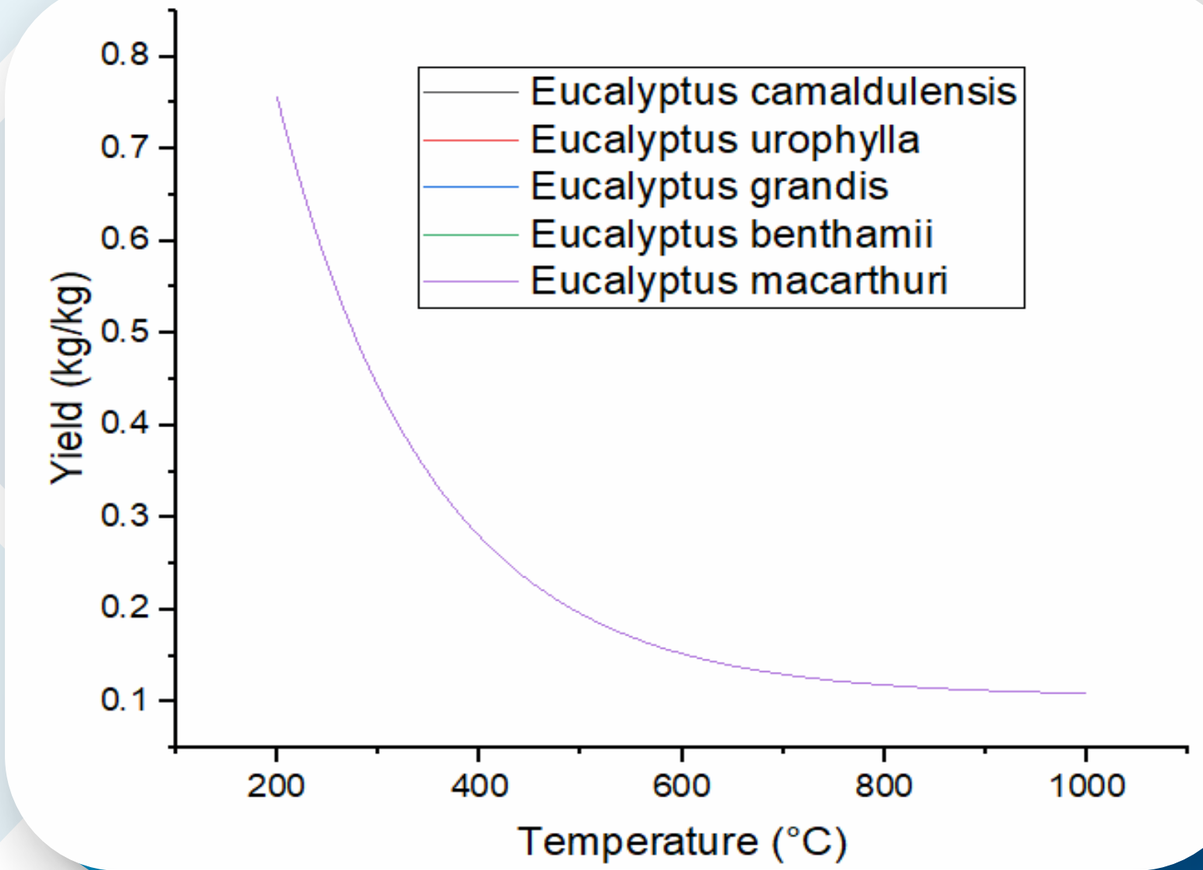


Figure 2.1 :
Effect of different wood biomass of Eucalyptus on char yield

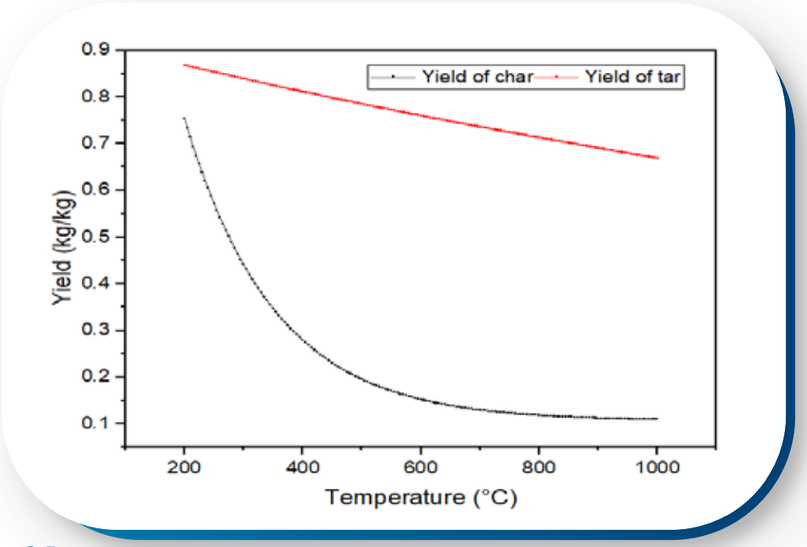


Figure 2.1 :
Effect of temperature on char and tar yield from Eucalyptus

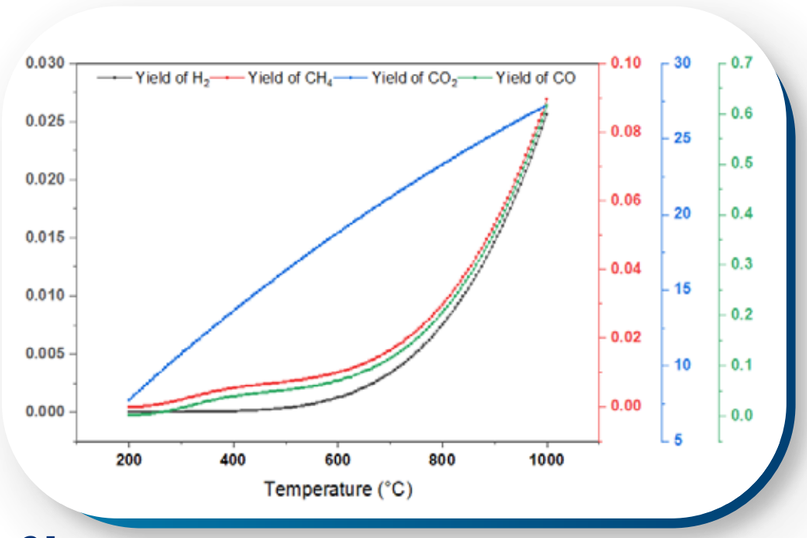


Figure 2.1 :
Effect of temperature on syngas composition from Eucalyptus

Experimental results of Eucalyptus wood pyrolysis

Table.4 – Yield of biochar and bio oil collected from the pyrolysis of Eucalyptus wood

Time (hr)	Maximum temperature rise(°C)	Weight of biochar(g)	Weight of bio-oil collected(g)	Volume of bio-oil collected (ml)	Density of bio-oil(g/ml)	Yield of biochar %(w/w)	Yield of bio-oil %(v/w)
2	360	276	59	68	0.87	27.6	6.8
2	580	126	40	41	0.98	25.6	8.2
1	420	193	15	15	1	38.6	3

- Pyrolysis is done for the different time and temperature.
- Here the completion of the pyrolysis is done by examining the rigidity of the bio-char. The completely processed biochar will break easily.
- With increase in temperature, volatiles are released out resulting in decreased yield of biochar and increased yield of bio oil.

Conclusion

- The best parameter for the production of higher yield of distillate with good fragrance is moisture content of 44.4%, particle size of 1 cm x 1 cm, and sample: water ratio of 1:5 at 100 °C yielding 7% of eucalyptus oil. Obtained oil contains dominant of β-pinene compound and 1,8 cineole compounds. The hydro-distillation for oil extraction is better than soxhlet extraction for better fragrance Pyrolysis of Eucalytpus wood resulted in biochar containing fully carbonized and shiny appearance with yield of ~27%

Future work

- Proximate characterization of biochar to evaluate for briquetting application
- Proximate characterization of spent leaves to evaluate its potential for pelletization
- Characterization of oil obtained from soxhlet extraction to analyze its biochemical compounds

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