

Utilization of Plastic Waste with Biomass for Production of Co-Pyrolysis Oil

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ABSTRACT: This study evaluates the pyrolysis suitability of pine needles and HDPE, highlighting their compositional differences and impacts on bio-oil production. Pine needles exhibit 7.92% moisture, 71.21% volatile matter, and 23.44% fixed carbon, making them favourable for bio-oil generation due to their low fixed carbon and significant volatile matter. However, their 17.45% extractive content, which includes various organic compounds, can decrease bio-oil yield and impede levoglucosan formation. Pine needles also contain 27.17% cellulose, 24.15% hemicelluloses, and 29.15% lignin. In contrast, HDPE has negligible moisture, 100% volatile matter, and no ash, offering a higher calorific value of 20.42 MJ/kg and facilitating easier conversion to hydrocarbon fuels due to its higher carbon and hydrogen content. Combining HDPE with biomass like pine needles can enhance energy production, utilizing the highly volatile matter of pine needles and the high calorific value of HDPE to optimize overall pyrolysis efficiency.

KEYWORDS: Biomass, Co-pyrolysis, Energy recovery, HDPE, Upgradation, Plastics waste

I. INTRODUCTION

The extensive use of fossil fuels over the past century has elevated greenhouse gas (GHG) emissions and hastened global warming [1]. The increasing focus on renewable sources and waste valorization is driven by factors such as global warming, the negative environmental impact of fossil fuels, rising energy demand, and the availability of waste materials. However, the exponential population growth and ongoing urbanization have heightened energy demand, highlighting the need to explore alternative resources. Consequently, the past few decades have seen a rising demand for transportation fuels driven by demographic changes.

Biomass, a renewable and carbon-negative fuel source, is one of the most explored and

environmentally benign alternatives. It is considered a potential feedstock for the rapidly advancing bio-economy, capable of being converted into high-quality fuel through processes like thermochemical conversion, fermentation, and anaerobic digestion. Pine needles, widely available biomass worldwide, are especially abundant in regions such as the North Western Himalayas in India, which has 7.62 million hectares of pine forests [2]. In India, the annual availability of pine needles is approximately 6.3 tons per hectare [3]. Biomass constitutes three major compounds: cellulose (40-50%), hemicellulose (20-40%), and lignin (16-22%) [4]. Using biomass to produce liquid fuel not only helps conserve fossil fuel reserves but also reduces GHG emissions. Pyrolysis is a widely studied technique for converting biomass into liquid oil, which can be further upgraded to gasoline and diesel range transportation fuels [5]. Additionally, it produces solid and gas fractions that can be used as fuels or for producing activated carbon. Therefore, studying pyrolysis processes to determine optimal operational conditions is of significant interest. Raw bio-oil from biomass pyrolysis typically shows 30-50% selectivity for undesired oxygenated compounds like aldehydes, ketones, and acids. The high oxygen content in biomass leads to these compounds, making the oil acidic and corrosive, which is undesirable for fuel [6].

To improve the fuel stability of bio-oil, co-pyrolysis of biomass and organics containing higher hydrogen contents can be considered. Therefore, plastics could potentially be a beneficial feedstock due to their higher hydrogen content than biomass and the small amount of water in their pyrolysis derived oil. The heating value of the plastic pyrolysis oil is nearly of conventional fuel, which is around 40 MJ/Kg, that it is also relatively clean and its fuel properties are similar to the petroleum fuels. Hence, the presence of plastic in the pyrolysis of biomass can make a positive

contribution towards the co-pyrolysis yield. Several co-pyrolysis of biomass and plastics investigations have been carried out to improve the bio-oil quality and to reduce the wax formation [7,8].

On the other hand, plastic pollution is one of the global environmental challenges [9]. Therefore, the utilization of plastics is important to preserve the environment. High-density polyethylene (HDPE) is significantly present in landfills and ocean litter [10,11]. Therefore, HDPE is selected as a co-feedstock in this study. The addition of HDPE with biomass in the pyrolysis process typically improves the quality of pyrolysis liquid by enhancing the aromatic hydrocarbon compounds [12]. In addition, HDPE does not contain oxygen content, which will significantly reduce the oxygenated compounds in pyrolysis liquid [13].

Therefore we hypothesized that adding HDPE plastics to biomass in a batch-type reactor would reduce both the fixed carbon and oxygen content in the feedstock. This, in turn, would lead to lower oxygenated compounds in the pyrolysis liquid and higher selectivity towards gasoline-range hydrocarbons (C6-C12). The quality of bio-oil depends not only on the pine needles to HDPE ratio but also on the temperature [14, 15]. Given that the decomposition temperatures of biomass and plastics typically differ, it is essential to study each pine needle to HDPE ratio at various pyrolysis temperatures.

This research aims to enhance the yield and quality of bio-oil through the co-pyrolysis of pine and HDPE at a laboratory scale. A key challenge in plastic pyrolysis is wax formation [16, 17]. The reaction mechanisms involving the hydrogen-rich HDPE and oxygen-rich pine, as well as the dewaxing process, are predicted based on observed end products analyzed through GC-MS, and are supported by relevant literature. The specific objective of this study is to identify the optimal combinations of temperature and pine-to-HDPE ratio by examining the effects of various pine/HDPE ratios (5/95, 10/90, 15/85, 20/80) and temperatures (450°C, 500°C, 550°C) on the pyrolysis products.

II. MATERIALS AND METHODS

Pine needles were collected from the pine forest of Bhowali, Nainital, India (coordinates: 29.3823°N 79.5196°E) and hereafter referred to as biomass. It was sieved to (I.S. sieve No. 20) get less than ≤ 0.841 mm particle size. HDPE waste was collected from Ramdas Enterprises Rudrapur which produces HDPE products and often has a significant amount of HDPE scrap. The ground material was then sieved into three different size groups: < 1.25 mm, 1.25 – 2.0 mm, and 2.0 – 3.0 mm by standard sieves. The elemental composition of different size groups was carried out to check the homogeneity.



Fig.1 Raw materials and its powdered form
(Pine needle and HDPE)

In the same way, the HDPE waste was made into powder with an average size of less than 1 mm, for making it into powder to reduce its volume by heating it in hot air oven for 2 hours at 100 °C. Before performing pyrolysis, the powdered

samples of pine needles and HDPE (PN: HDPE) were blended in order to achieve homogeneity. Fig.1 shows the pictorial representation of raw materials.

Characterization of Feedstock

The feedstock characterization involved analyzing both the biomass and HDPE through proximate and elemental analyses. Biomass typically contains a considerable amount of moisture, whereas HDPE contains almost none. Therefore, the biomass was dried at 105 ± 5 °C for 24 hours using the Oven before use. After the drying process, a weight loss of approximately 10 % was observed, attributed to the intrinsic moisture content in the biomass [18].

Proximate analysis

The proximate analysis included the determination of moisture, ash, volatile matter, and fixed carbon.

Moisture Content

Moisture is defined as the quantity of liquid present per unit mass of the wet solid. According to the ASTM D 3173 standardized test method for moisture analysis, a 1 g sample is placed in a hot-air oven at 105°C for 1 hour. After drying, the sample is weighed. The moisture content is then determined as follows:

$$\text{Moisture Content (\%)} = \frac{W_2 - W_3}{W_2 - W_1} \times 100 \dots (1)$$

Where W_1 is the weight of the empty crucible (g), W_2 the weight of the empty crucible + sample (g) and W_3 is the weight of the empty crucible + sample after drying (g).

Volatile matter

Volatile matter quantifies the percentage of gaseous products released under specific test conditions from a moisture-free sample. According to the ASTM D 3175 standard test method for volatile matter analysis, an oven-dried sample placed in a covered crucible is heated in a muffle furnace at 950 ± 20 °C for 7 minutes. The crucible is then cooled first in ambient air and subsequently in a desiccator, after which the weight loss is calculated.

$$\text{Volatile matter (\%)} = \frac{W_3 - W_4}{W_2 - W_1} \times 100 \dots (2)$$

Where W_1 is the weight of the empty crucible (g), W_2 the weight of the empty crucible + sample (g) and W_3 is the weight of the empty crucible + sample after oven-drying (g). W_4 is the weight of the empty crucible + weight after heating in the muffle furnace (g).

Ash content

Ash content was measured by weighing the residue left after burning the coal under strictly controlled conditions, including sample weight, temperature, time, atmosphere, and equipment specifications. According to the ASTM D 3174

standardized test method for ash content analysis, the remaining sample residue after the volatile matter test is heated in a muffle furnace at 700 ± 50 °C for 4 hours. The ash content in each sample is then estimated using the following formula:

$$\text{Ash content (\%)} = \frac{W_5 - W_1}{W_2 - W_1} \times 100 \dots (3)$$

where W_1 is the weight of the empty crucible (g), W_2 the weight of the empty crucible + sample, after volatile matter (g) and W_5 is the weight of the empty crucible + ash left in the crucible (g).

Heating value

The heating value of raw materials is determined using a bomb calorimeter, following the ASTM E711-87 standard test method for calorific value. The bomb calorimeter features a solid cylindrical stainless-steel bomb where fuel combustion occurs. A fuel sample weighing less than 1 g is placed in the crucible, and a fuse wire ignites the fuel. The bomb is filled with oxygen gas at 25–30 atmospheric pressure. The electrodes are connected to an electrical supply, and the initial water temperature is recorded. After the fuel sample combusts, the rise in water temperature is measured to determine the calorific value using the following formula:

$$\text{Calorific value (kcal/kg)} = \frac{WX\Delta T}{M} \dots (4)$$

where M is the Mass of fuel placed in the crucible (g), W is the water equivalent of the bomb calorimeter or heat capacity (cal/°C), $\Delta T = t_2 - t_1$, where t_1 is the initial temperature of water in the calorimeter (°C) and t_2 is the final temperature of water in the calorimeter (°C).

To determine the heat capacity of the bomb calorimeter, wherever nichrome fuse wire and cotton thread are used simultaneously, pure benzoic acid is used in the bomb. In this case, cotton thread was also used along with nichrome fuse wire. The heat capacity for nichrome fuse wire is 333.68 cal/g and for cotton thread, it is 4180 cal/g [19].

$$W = \frac{MXH + (E_w) + (E_t)}{\Delta T} \dots (5)$$

where W is the water equivalent of the bomb calorimeter (2283.32 cal/°C), M is the mass of the test sample, ΔT the rise in temperature, E_w is the correction of heat of combustion for nichrome fuse wire and E_t is the correction of heat of combustion for cotton thread

$$E_w = M_w \times H_w \dots (6)$$

Where M_w is the mass of nichrome fuse wire and

Hw is the heat capacity per gram of nichrome fuse wire

$$E_t = M_t \times H_t \quad \dots(7)$$

where M_t is the mass of cotton thread and H_t is the heat capacity per gram of cotton thread.

Elemental analysis

Elemental analysis (EA) refers to the process of determining of mass fraction of carbon, hydrogen, nitrogen, and sulphur to find out the structure of an unknown compound. EA of Pine needle and HDPE was carried out in a CHN analyzer. Oxygen content was determined by the difference in weight considering that the whole biomass was made of C, H, N, O, and ash.

Composition analysis

The hemicellulose, lignin, and cellulose content of pine needles (*Pinus roxburghii*) were determined using direct methods [20]. Prior to measuring cellulose, hemicellulose, and lignin, the extractives in the cattle dung were determined using a Soxhlet extractor. A 5.0 g biomass sample was placed in a cellulose thimble inside the Soxhlet apparatus receiver, and 300 ml of acetone was added to the heating flask. The acetone was heated to 90°C for 2 hours, during which several extraction cycles occurred. The extracted sample was then oven-dried at 105°C until a constant weight was achieved. The extractive content was calculated as the weight difference between the raw

biomass and the extractive-free biomass.

To determine the hemicellulose content, 30 ml of 0.5 M NaOH solution was added to 1 g of extractive-free biomass in a 100 ml Erlenmeyer flask, and the mixture was heated at 80°C for 3.5 hours since hemicellulose is soluble in 0.5 M NaOH solution. The sample was then filtered and washed with deionized water until the pH reached 7.0. The residue was dried at 105°C in a hot air convective oven for two hours. The hemicellulose content was calculated as the difference in weight before and after this treatment.

The lignin content was determined by adding 30 ml of 98 wt.% H₂SO₄ to 1 g of dried extractive-free biomass in a 100 ml Erlenmeyer flask. The sample was kept at ambient temperature for 24 hours with periodic shaking to ensure complete hydrolysis of cellulose and hemicellulose. Afterward, it was boiled at 100°C in a water bath for 1 hour and then filtered through a filtering crucible.

The residue was then washed with de-ionized water until it reached a neutral pH and dried to a constant weight at 105°C.

To determine the ash content, the obtained residue was incinerated in a muffle furnace at 575°C. The lignin content was calculated as the difference in weight after deducting the ash content. The cellulose content was determined by difference, assuming that extractives, hemicellulose, lignin, ash, and cellulose are the only components of the entire biomass.

Co- pyrolysis process and experimental setup

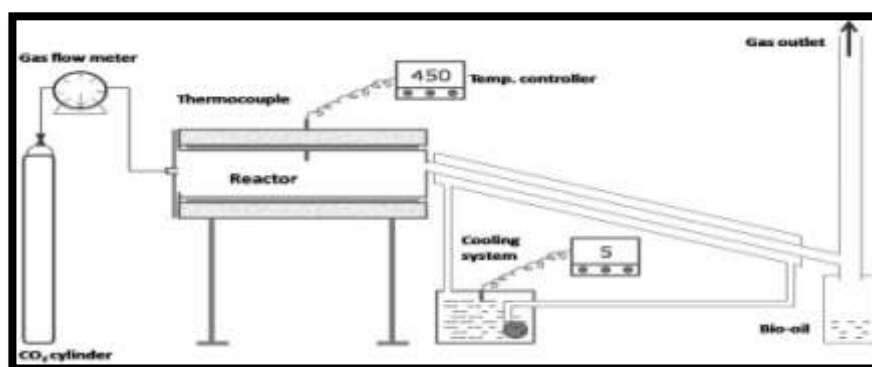


Fig 2 Schematic diagram of laboratory scale bio-oil production unit

Batch pyrolysis of *Pinus roxburghii* needles and HDPE was conducted in a reactor constructed from a 5mm stainless steel sheet, with an internal diameter of 400 mm and a length of 400 mm. For each run, 1000 g of dried feedstock (PN: HDPE) was loaded into the reactor. Prior to loading the feedstock, CO₂ was purged into the reactor at a

flow rate of 3 l min⁻¹ for 15 minutes to establish an inert atmosphere, with the CO₂ flow rate monitored using a gas flow meter with a range of 0.1 to 3 l min⁻¹ and a least count of 0.1 l min⁻¹.

The temperature within the reactor was monitored using a Ni-Cr-Ni (K type) thermocouple and controlled by a high-sensitivity PID controller.

The desired pyrolysis temperature, ranging from 450°C to 550°C, was achieved at a constant heating rate of 15°C/min and maintained according to the experimental design parameters, and the maximum yield of the liquid product was obtained at 550°C. The reactor was heated using an electrical furnace, and the volatiles generated during pyrolysis were promptly condensed using a water-cooled condenser operating at 10 °C, while non-condensable gases were vented out.

The resulting condensate, comprising both aqueous and organic phases, was collected in a pre-weighed, dried beaker. The organic phase was then extracted by adding an equal volume of dichloromethane to the mixture, which caused the organic phase to settle below the aqueous phase due to its higher density. This mixture was separated using a separating funnel, and the organic phase was filtered using Grade 4 filter paper, which had a diameter of 110 mm, a thickness of 205 µm, and a pore size of 20-25 µm. Dichloromethane from the organic phase was separated using distillation at a temperature of 40-45 °C. The residual was then weighed and considered as the recovered bio-oil [21]. The pyrolysis reactor used in this study is shown in Figure 2.

Post-pyrolysis, the reactor was cooled to room temperature and weighed to estimate the char yield, calculated as the weight percentage of the solid residue. The gas yield, representing the non-condensable volatiles, was determined by the difference between the total weight percentage of

the liquid products and char. The optimum temperature for maximum pyrolytic oil produced from PN and HDPE co-pyrolysis was decided on the basis of the highest weight percentage yield of the pyrolytic oil. The weight percentage yields of the liquid, solid, and gaseous products were calculated using the following equations:

- Liquid yield % = (Weight of the liquid product / Weight of the feed) X 100 ... (8)
- Char yield % = (Weight of the char / Weight of the Feed) X 100 ... (9)
- Gas yield % = 100 – (Total liquid yield % + Char yield %) ... (10)

III. RESULTS AND DISCUSSION

Properties of feedstock

The average moisture content of ground pine needles was 7.92 %, while HDPE had negligible moisture. Moisture in raw materials increases bio-oil water content during pyrolysis. Pine needles had 71.21% volatile matter and 2.02% ash, while HDPE had 100% volatile matter and no ash. High volatile matter enhances bio-oil yield, and low ash content ensures high-quality bio-oil. The fixed carbon content of pine needles was 23.44%, negligible in HDPE. Low fixed carbon content favors bio-oil production by reducing biochar and pyrolysis gas production, making these solid fuels suitable for large-scale pyrolysis oil production.

Table 1. Results of proximate, composition, and ultimate analysis of Pine needles and HDPE particles

Characteristics	Pine needles	HDPE
Proximate Analysis (wt. %)		
Moisture Content	7.92	0
Volatile matter	71.21	100
Ash content	2.02	0
Fixed carbon	23.43	0
Composition Analysis (wt. %)		
Cellulose	27.17	-
Hemi cellulose	24.15	-
Lignin	29.15	-
Extractives	17.45	-
Ultimate Analysis (wt. %)		
C	44.99	83.360
H	5.46	14.787
N	0.99	0
S	-	0.464
O	48.55	-
H/C	1.46	2.13

O/C	0.81	-
N/C	-	0.0020
Empirical formula	$CH_{1.46}N_{0.02}O_{0.81}$	$CH_{2.13}S_{0.002}$
HHV(MJkg⁻¹)	17.67	46

The extractives content of pine needles was 17.45%, comprising fats, waxes, proteins, phenolics, sugars, gums, resins, terpenes, starches, glycosides, saponins, and essential oils (Mohan et al., 2006). Extractives reduce bio-oil yield and inhibit levoglucosan formation (García-Pérez et al., 2007). Pine needles contained 27.17% cellulose, 24.15% hemicelluloses, and 29.15% lignin, within typical biomass ranges.

The ultimate analysis shows that carbon, hydrogen, nitrogen, and oxygen contents for pine needles (PN) were 44.99%, 5.46%, 0.99%, and 48.55%, and for HDPE were 49.57%, 7.2%, 1.05%, and 34.12%, respectively. HDPE's higher carbon and hydrogen content makes it easier to convert into hydrocarbon fuels. Pine needles have more oxygen, leading to a lower calorific value compared to HDPE. Mixing plastic and biomass enhances energy production. The chemical compositions were $CH_{1.46}N_{0.02}O_{0.81}$ for PN and $CH_{2.13}S_{0.002}$ for HDPE, with higher heating values of 17.67 MJ/kg and 20.42 MJ/kg, respectively.

Effect of plastic composition in feed blend on bio-oil yield

Table 2 presents compelling evidence of the potential benefits of co-pyrolyzing pine needles (PN) with HDPE at varying ratios, highlighting significant variations in the yields of solid, liquid (bio-oil), and non-condensable gas phases. Pure HDPE (0:100) serves as a baseline, yielding 31.7% solid, 28.1% bio-oil, and 40.2% non-condensable gas. As the proportion of pine needles increases, notable trends emerge. At a 95:05 ratio (A1), bio-oil yield slightly improves to 31.435%, with solid and gas yields at 32.08% and 36.485%, respectively. The 90:10 blend (A2) further enhances bio-oil yield to 34.19%, while solid and gas yields adjust to 31.10% and 34.71%.

A more pronounced effect is observed at an 85:15 ratio (A3), where bio-oil yield significantly increases to 38.12%, accompanied by 30.40% solid and 31.48% gas yields. The most substantial improvement is seen with an 80:20 blend (A4), achieving the highest bio-oil yield of 41.57%, with the lowest solid yield of 27.0% and a gas yield of 31.43%. These results underscore the synergistic effects of co-pyrolysis, where the inclusion of pine needles enhances bio-oil production, making the 80:20 ratio particularly promising for optimizing liquid fuel output.

Table 2 Product distribution of pyrolysis using pine needles and HDPE

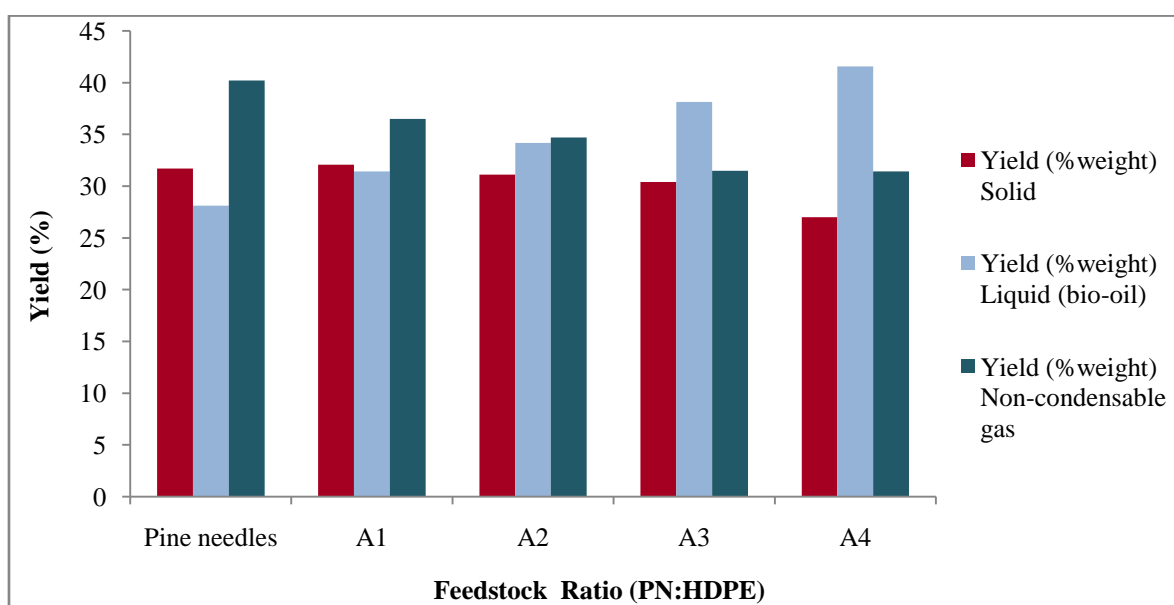
Feedstock blend Type	Pine needle to plastic ratio (PN:HDPE)	Yield (% weight)		
		Solid	Liquid (bio-oil)	Non-condensable gas
Pine needles	0:100	31.7	28.1	40.2
A1	95:05	32.1	31.4	36.5
A2	90:10	31.1	34.2	34.7
A3	85:15	30.4	38.1	31.5
A4	80:20	27.0	41.6	31.4

Fig. 3 illustrates the yields of solid, liquid (bio-oil), and non-condensable gas phases from the co-pyrolysis of pine needles and HDPE at various ratios. It shows that the effect of plastic composition in the feed blend on bio-oil yield reveals intriguing trends. The solid yield decreases as the proportion of pine needles increases, indicating that pine needles contribute to a lower

solid yield compared to HDPE. Conversely, the bio-oil yield significantly increases with a higher ratio of pine needles, reaching its peak at the 80:20 ratio (A4). This suggests that the addition of pine needles enhances bio-oil production during co-pyrolysis, making it an effective strategy for maximizing liquid fuel output. Additionally, the non-condensable gas yield decreases with an

increasing ratio of pine needles, highlighting that HDPE produces more non-condensable gases compared to pine needles. These findings underscore the synergistic potential of co-pyrolyzing pine needles with HDPE, optimizing bio-oil yield while reducing unwanted by-products, thereby presenting a promising avenue for sustainable energy production.

According to [22], co-pyrolysis of plastics and biomass, where plastics have a higher H/C mole ratio than biomass, can improve bio-oil yield compared to mixing yields from pure plastics and biomass pyrolysis. Thus, the graph in Fig. 1 suggests that bio-oil yields from co-pyrolysis should be higher than those obtained by mixing pure feedstock yields.



(PN:HDPE ; A1- 95:05, A2- 90:10, A3- 85:15, A4- 80:20)

Fig. 3 Yield of bio-oil from pyrolysis using different blends of Pine needles and HDPE

IV. CONCLUSION

The analysis of pine needles and HDPE reveals distinct differences impacting their suitability for pyrolysis. Pine needles, with 7.92% moisture, 71.21% volatile matter, and 23.44% fixed carbon, offer a favorable profile for bio-oil production due to their low fixed carbon and moderate volatile matter. However, their extractive content of 17.45%, including various organic compounds, can reduce bio-oil yield and hinder levoglucosan formation. Pine needles contain 27.17% cellulose, 24.15% hemicelluloses, and 29.15% lignin, aligning with typical biomass compositions. HDPE, with negligible moisture, 100% volatile matter, and no ash, provides a higher calorific value (20.42 MJ/kg) and easier conversion to hydrocarbon fuels due to its higher carbon and hydrogen content. The blending of HDPE with biomass like pine needles can enhance overall energy production by leveraging the highly volatile matter of pine needles and the high calorific value of HDPE.

Co-pyrolysis of pine needles and HDPE at varying ratios shows that increasing the proportion

of pine needles in the blend enhances bio-oil yield while reducing solid and non-condensable gas yields. The optimal ratio for bio-oil production is 80:20 (PN:HDPE), making it the most efficient blend for maximizing liquid fuel output.

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