Baku State University Journal of Chemistry and Material Sciences, 2026, v. 3 (1), p.43-48

journal homepage: http://bsuj.bsu.edu.az/en
https://doi.org/10.30546/209501.201.2026.03.001.024

INVESTIGATION OF THE CHEMICAL AND PHYSICAL CHARACTERISTICS OF PRODUCTS DERIVED FROM THE PYROLYSIS OF PINE WOOD BIOMASS

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Received: 22 February 2026
Accepted: 10 April 2026
Published: 13 April 2026

In this article, the phase distribution and the main physicochemical properties of the solid (biochar), liquid (bio-oil), and gaseous products obtained during the pyrolysis of pine (Pinus spp.) biomass under an inert atmosphere were comprehensively investigated. Pyrolysis of lignocellulosic feedstocks is one of the widely used thermochemical conversion methods for the production of biofuels and carbonaceous materials. The experiments were carried out in the temperature range of 450–550 °C at a heating rate of 300 °C/min under a nitrogen flow of 400 cm³/min. The liquid fraction was analyzed by GC–MS, and it was found that phenolic derivatives (~45.5%) and ketones (~17%) were the predominant components, which is consistent with the high lignin content of pine wood.

Thermal analysis (TG/DTG/DTA) of the raw material showed that the main devolatilization stage occurs in the temperature range of 267-356 °C. Chromatographic analysis of the gaseous fraction revealed the predominance of methane and C_2–C_3 hydrocarbons. The calorific value of the solid residue, estimated using the Dulong-type approach, was about ~18.9 MJ/kg. The obtained results indicate that pine wood waste is a promising feedstock for both energy and material-oriented utilization.

Keywords: pine wood, biomass, pyrolysis, bio-oil, biochar, gas composition, TG/DTG/DTA, GC–MS

INTRODUCTION

The growing accumulation of forestry and agricultural residues has intensified interest in their rational utilization not only as energy resources but also as sources of value-added carbon-based materials [1–3]. Among the available thermochemical conversion routes, pyrolysis occupies a special place due to its flexibility with respect to feedstock type and operating conditions, as well as its ability to generate three useful product streams, namely biochar, bio-oil, and combustible gases [1-4]. In recent years, increasing attention has been paid to small- and medium-scale pyrolysis systems intended for decentralized processing of biomass residues, particularly in regions with developed forestry and agricultural sectors [2,5]. In this context, the investigation of widely available and structurally uniform feedstocks is of both scientific and practical relevance [6,7].

Pine wood represents one of the most abundant lignocellulosic resources in many regions and is characterized by relatively stable composition and high lignin content [6,8]. These features make it an attractive model material for studying the relationships between biomass structure, thermal conversion behavior, and product distribution [9-11]. A detailed

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examination of the pyrolysis of pine wood not only contributes to a better understanding of the decomposition pathways of lignocellulosic components but also provides a basis for evaluating the potential of this feedstock for the integrated production of energy carriers and value-added carbonaceous materials [3,4,12].

EXPERIMENTAL

Pine wood sawdust and crushed wood particles were used as the feedstock. Prior to the experiments, the samples were air-dried under laboratory conditions, sieved to obtain a uniform particle size fraction, and subsequently dried to constant weight in order to remove residual moisture.

The pyrolysis experiments were carried out in a laboratory-scale tubular reactor under an inert atmosphere. Nitrogen was used as the carrier gas to ensure oxygen-free conditions. The heating rate was fixed at 300 °C/min, while the final temperature was varied in the range of 450–550 °C. The nitrogen flow rate was maintained at 400 cm^3/min throughout the experiments. For each temperature regime, the experiment was repeated at least three times (n = 3), and the reported results represent average values.

Thermogravimetric and differential thermal analyses (TG/DTG/DTA) were performed under a nitrogen atmosphere at a heating rate of 10 °C/min using sample masses in the range of 8–10 mg in order to evaluate the thermal decomposition behavior of the raw material.

The composition of the gaseous products was determined by gas chromatography equipped with thermal conductivity (TCD) and flame ionization (FID) detectors. The analytical results were initially obtained on a volumetric basis and then converted to mass percentages taking into account the molar masses of the individual components. The liquid product (bio-oil) was analyzed by gas chromatography–mass spectrometry (GC–MS) in order to identify its main groups of compounds.

For each experiment, the solid, liquid, and gaseous products were collected separately and weighed. The overall mass balance of the process was established on the basis of these measurements and used to assess the reproducibility and reliability of the experimental data.

RESULTS AND DISCUSSION

The TG/DTG/DTA results indicate that the main devolatilization stage of pine wood biomass occurs in the temperature range of 267–356 °C, which is mainly associated with the decomposition of hemicellulose and cellulose components [6,8]. This interval corresponds to the region of the most intensive mass loss observed on the TG curve and to the main peaks on the DTG and DTA curves (Figure 1). At higher temperatures, the mass loss rate decreases and the process becomes dominated by the gradual decomposition of lignin and the development of carbonization reactions, which proceed over a broader temperature range due to the more complex and thermally stable structure of lignin [8,11].

It is well established that particle size and heating rate exert a strong influence on the kinetics of biomass thermal decomposition. Smaller particles provide more efficient heat transfer, which facilitates the release of volatile products and reduces the contribution of secondary repolymerization reactions [6,8]. In the present work, the use of pre-sieved and homogenized feedstock, together with a relatively high heating rate of 300 °C/min, ensured rapid devolatilization and limited the residence time of vapors inside the reactor, thereby restricting the extent of secondary transformations. This approach was chosen to favor the formation of volatile products and to better resolve the contribution of the gaseous and liquid fractions.

The overall product distribution obtained under the selected conditions is summarized in Table 1 (A). As can be seen, the gas fraction accounts for 34.5 wt.% of the initial feedstock, while the solid residue (semi-char) represents 65.5 wt.%. These values are in the range typically reported for the pyrolysis of lignocellulosic materials under comparable conditions

and confirm that pine wood follows the general conversion pattern observed for woody biomasses [1,3,13]. The detailed composition of the gas fraction is presented in Table 1 (B) [14].

Table 1. Yield and composition (wt.%) of the gas fraction obtained during the pyrolysis of pine wood biomass.

Parameter	(Value (wt.%)
A	
Initial feedstock	100
Gas product	34.5
Solid residue (semi-char)	65.5
B	
Methane (CH_4)	34.3
C_2 fraction (ethane + ethylene)	18.2
C_3 fraction (propane + propylene)	16.3
i-Butane (i-C_4H_{10})	2.8
Total butenes (C_4H_8)	8.3
n-Butane (n-C_4H_{10})	4.2
Other components	15.9

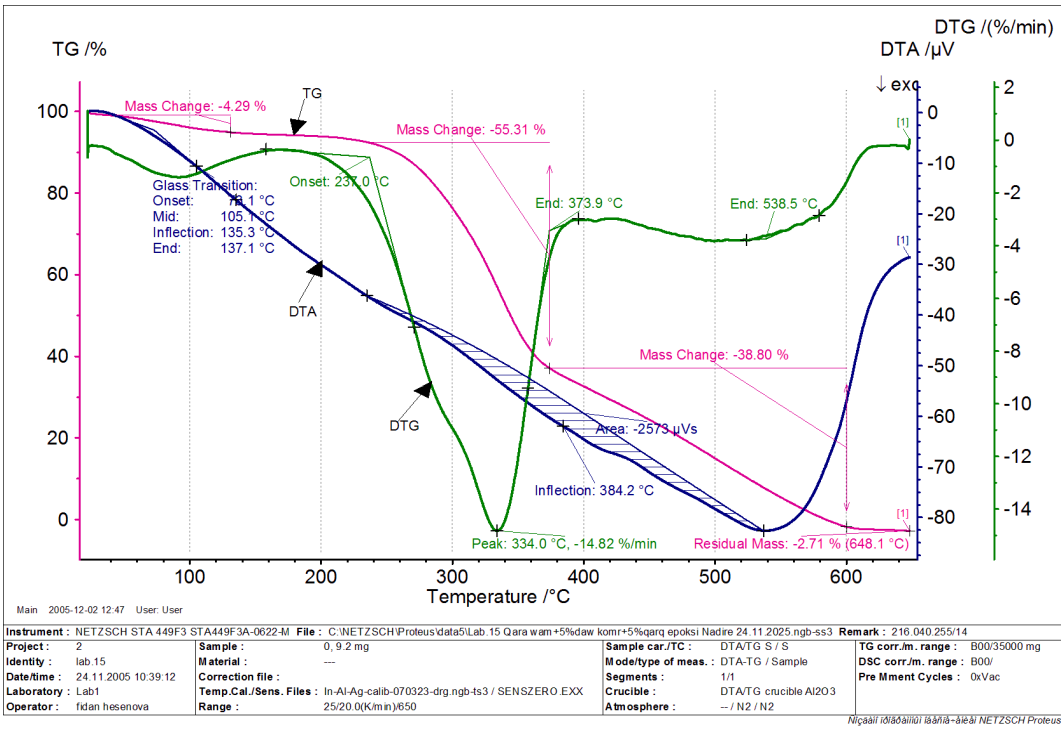

Figure 1. TG/DTG/DTA curves of pine wood biomass (in nitrogen atmosphere, 10 °C/min, sample mass 8–10 mg

The gaseous product is dominated by methane (34.3 wt.%), followed by the C_2 (ethane + ethylene, 18.2 wt.%) and C_3 (propane + propylene, 16.3 wt.%) fractions, while smaller amounts of C_3 hydrocarbons and other minor components are also present. Such a composition is characteristic of gases obtained from the pyrolysis of lignocellulosic feedstocks and is in good agreement with literature data [5,15-17]. According to previous studies, an increase in the pyrolysis temperature generally enhances the contribution of the gas fraction as a result of secondary cracking of vapors [1,3,16,18], and the present results are consistent with this overall tendency.

From an energetic point of view, the predominance of low-molecular-weight hydrocarbons indicates that the gas fraction possesses a relatively high calorific value and can be considered a valuable energy carrier. This creates the possibility of using the gaseous

products, at least partially, to cover the heat demand of the process itself, thereby improving the overall energy efficiency of the system [1,7,19].

With regard to the liquid product, the GC–MS analysis shows a clear predominance of phenolic derivatives, which can be attributed to the decomposition of lignin-derived aromatic structures. This observation is fully consistent with numerous literature reports indicating that guaiacol, syringol, and their alkylated derivatives are among the main components of bio-oils obtained from lignin-rich feedstocks [4,9,10]. At the same time, the presence of ketones and other oxygen-containing compounds is mainly associated with the thermal conversion of cellulose and hemicellulose fractions [5,6]. While such a composition increases the chemical value of the bio-oil as a potential source of valuable compounds, it also explains its relatively high acidity and limited storage stability, which are well-known drawbacks of biomass-derived pyrolysis oils.

The solid residue obtained in the process can be classified as semi-char or biochar and represents a carbon-rich material with potential energy and material applications. The energy potential of this product, estimated using the Dulong approach, is about ~18.9 MJ/kg, which is comparable with values reported for biochars derived from similar types of biomass [8,12,20]. However, as emphasized in the literature, a more accurate determination of the heating value requires direct calorimetric measurements [2,12,21]. In addition to its possible use as a solid fuel, the mineral composition of the residue suggests that, after appropriate activation or modification, this material may also be considered for use as an adsorbent or functional carbon material.

It should be noted that, in laboratory-scale pyrolysis experiments, the closure of the mass balance is an important indicator of data reliability. According to published data, uncertainties at the level of 3–7% may occur due to condensation losses and deposition of products on reactor walls [1,2,10,22]. In the present study, repeated experiments were performed in order to minimize these effects and to ensure acceptable reproducibility of the obtained results.

Overall, although the pyrolysis of pine wood has been widely investigated, the present work provides a combined and internally consistent evaluation of thermal behavior, gas composition, and biochar energy potential for a local feedstock under a high-heating-rate regime. This integrated approach allows a more comprehensive assessment of the conversion behavior of pine wood biomass and its suitability for both energy and material-oriented utilization.

CONCLUSION

The performed experiments show that the pyrolysis of pine wood biomass in the temperature range of 450–550 °C under a high heating rate leads to the formation of about 34.5 wt.% of gaseous products and about 65.5 wt.% of solid residue (semi-char). The TG/DTG/DTA data indicate that the main devolatilization stage takes place in the interval of 267–356 °C and is mainly related to the decomposition of hemicellulose and cellulose.

The gas fraction is characterized by a high content of methane (34.3 wt.%) and C_2–C_3 hydrocarbons, which explains its relatively high energy value. The solid product shows an estimated calorific value of about 18.9 MJ/kg, making it suitable for use as a solid fuel or carbon material. The liquid product is dominated by phenolic compounds of lignin origin, which points to its possible use as a chemical feedstock.

In general, the obtained results confirm that pine wood waste can be considered a suitable raw material for thermochemical conversion into gaseous, liquid, and solid products with clearly different properties and potential fields of application.

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