

ORIGINAL RESEARCH PAPER

## Investigating the impact of process parameters on waste tire pyrolysis and characterizing the resultant chars and oils

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### ABSTRACT

**BACKGROUND AND OBJECTIVES:** The escalating global population, coupled with increased transportation needs and car production, has led to a surge in waste tire generation, reaching billions annually. Recognizing the environmental impact, there's a growing focus on utilizing waste tires as secondary raw materials and energy sources. Despite challenges posed by tire resilience and resistance to degradation, the current study advocates for pyrolysis as an eco-friendly method to recycle tires. It explores key process parameters (temperature and residence time) in pyrolysis, emphasizing qualitative and quantitative analyses of resulting oils compared to other products. The goal is to contribute to sustainable tire waste management and resource recovery.

**METHODS:** The study employed Thermogravimetric analysis for rubber's thermal characteristics, Fourier Transform Infrared Spectroscopy for pyrolysis oil analysis, Detailed Hydrocarbon Analysis using a VARIAN CP-3800 instrument, and a bomb calorimeter (Parr 1256) for measuring heat capacity in obtained oils.

**FINDINGS:** The yields of pyrolysis oil, char and gas were 20–32.5, 45-60, and 5-30 wt.%, respectively. The Detailed Hydrocarbon Analysis results ranged from 5-20 showed that pyrolysis oils consisted mainly of 2,3-dimethylbutene, 2-methylbutene, t-isobutyl-4-ethyl-benzene, and 1-m-4-Isopropyl-benzene. Fourier Transform Infrared Spectroscopy showed the increase of aromatic components with increasing pyrolysis temperature. The measuring of highest calorific value of pyrolysis oil was 10309 Cal/g which showed good compatibility with commercial heating oils.

**CONCLUSION:** Pyrolysis oils from waste tires exhibit calorific values comparable to commercial heating oils, suggesting a promising alternative fuel source with versatile compositions. The findings help to understand the feasibility and potential applications

DOI: [10.22034/IJHCUM.2024.03.10](https://doi.org/10.22034/IJHCUM.2024.03.10) of waste tire pyrolysis in sustainable energy solutions.



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## INTRODUCTION

The substantial global rise in population and concurrent growth in transportation demands, along with increased car production, have contributed to a surge in the generation of waste tires. Annually, billions of scrap tires are produced globally (Hita *et al.*, 2016; Nejatian *et al.*, 2023; Pazoki and Ghasemzadeh, 2020). It is estimated that the amount of 198,346-339,678 tons of tire waste in Iran reached the end of life during 2003-2015 (equals 2.3955-4.52 kg per capita per year) (Zarei *et al.*, 2018). This has prompted a growing interest in addressing environmental concerns (Karbassi and Pazoki, 2015) and recognizing the valuable potential of waste tires as secondary raw materials and energy resources. The focus on recycling or reusing rubber waste has intensified in recent years (Pazoki *et al.*, 2018). Effectively managing the recycling and disposal of worn-out tires presents notable hurdles owing to their durability and resistance to natural breakdown. Although the considerable calorific content of discarded tires renders them suitable for burning, the release of harmful gases during the combustion process raises environmental apprehensions, constituting a noteworthy obstacle in the current era. At present, the utilization of pyrolysis emerges as a distinctive and eco-conscious approach to repurpose and recycle scrap tires. This method entails transforming tires into valuable commodities, such as liquid fuels, offering a sustainable alternative (Kaminsky *et al.*, 2009). Numerous methods and reactors have been utilized in the pyrolysis of discarded tires, and thorough investigations have been carried out to analyze and define the products derived from the pyrolytic process (Choi *et al.*, 2014; López *et al.*, 2010; Rushdi *et al.*, 2013; Williams and Brindle, 2003). A major output of pyrolysis is oil, holding the potential for the production of valuable chemicals like toluene, xylene, benzene, and limonene (Islam *et al.*, 2008). Additionally, pyrolysis produces gas and char, both valuable materials. The char primarily comprises carbon black, and the gases consist of C<sub>1</sub> to C<sub>4</sub> organic compounds (Sahouli *et al.*, 1996; Tajfar *et al.*, 2023). Certain research endeavors focused on scrutinizing the influence of residence time, oxygen concentration, and feedstock flow rate on the production and features of carbon black obtained from the pyrolysis oil of used tires. This examination maintained a consistent temperature throughout the

study (Heidary, 2017; Wen *et al.*, 2023). Toth *et al.* (2018) supported and validated these conclusions by illustrating a surge in carbon black yield from biomass pyrolysis oil with an elevation in temperature from 900 to 1300 °C. Nevertheless, the yield experienced a decline, reaching as low as 7% at a temperature of 1700 °C (Toth *et al.*, 2018). The results revealed a decrease in output as residence time and oxygen concentration increased. Notably, the BET surface area demonstrated an augmentation at elevated oxygen concentrations but saw a decline with prolonged residence times. Distinct variations in both yield and primary particle size of carbon black were observable across diverse samples of pyrolysis oil derived from spent tires. In a separate study, Ono *et al.* (2012) utilized benzene as a feedstock and observed a reduction in the primary particle size of carbon black from 40 to 31 nm as the temperature increased from 1200 to 1400 °C (Ono *et al.*, 2012). More recently, Okoye *et al.* (2022, 2021b) explored the influence of process variables (reaction temperature, residence time, and oxygen concentration) on the yield and quality of carbon black produced from spent tire pyrolysis oil and heavy residue fraction (Okoye *et al.*, 2022, 2021). Their findings indicated a significant improvement in carbon black properties (BET surface area, carbon content, and volatile matter) with increasing residence time, oxygen concentration, or reaction temperature. However, this enhancement came at the cost of carbon black yield, suggesting a trade-off in selecting process variables (Wen *et al.*, 2023; Pazoki *et al.*, 2020). Presently, numerous studies have investigated the impact of process parameters on the co-pyrolysis of coal and waste tires. These studies revealed that factors such as temperature and blending ratio exert varying degrees of influence on the co-pyrolysis process (Abdoli and Ghasemzadeh, 2024; Ghasemzadeh *et al.*, 2022a). In another study, the inclusion of 67 wt% of tires into lignite was found to enhance tar yield consistently across various pyrolysis temperatures. This indicates favorable synergies for tar production at all mixing ratios (Acar *et al.*, 2011). In a separate investigation, it was reported that there existed a positive synergy for tar production with coal/tire mixing ratios of 9/1 and 8/2 at temperatures ranging from 400 to 800 °C during co-pyrolysis (Brat *et al.*, 2022). Moreover, the rate of heating emerged as a pivotal factor influencing the synergy between coal and tires. Onay and Koca (2015)

found that higher heating rates promoted interactions between coal and waste tires for tar production, leading to improved yield and quality (Onay and Koca, 2015). Conventional heating approaches, like electrical resistance heating, encounter difficulties in attaining high heating rates. Conversely, the infrared fast-heating fixed-bed reactor presents a solution, leveraging its benefits of a rapid heating rate of up to 30 °C/s and a relatively substantial sample loading (gram). This allows for thoroughly exploring and comprehending interactions during co-pyrolysis (Onay and Koca, 2015; Tokmurzin *et al.*, 2019). This study deals with the process parameters including temperature and residence time in pyrolysis of waste tire products like chars and oils. Also, the aim was both qualitative and quantitative analyses of the produced oils in comparison to other derived products. The current study was carried out at the University of Tehran in 2022.

## **MATERIALS AND METHODS**

### *Feedstock preparation*

In general, the waste tires consisted of a combination of at least three distinct rubber types, namely Natural Rubber (NR), Styrene Butadiene Rubber (SBR), and Polybutadiene Rubber (PB). The powder derived from waste tires, devoid of any steel or fiber components, was acquired from Yazd Tire Company in Iran. The waste tire powder, with particle sizes ranging from 0.2 to 1 mm, served as the direct feedstock for the pyrolysis experiments. Also, the sampling of the input raw materials of the pyrolysis process was done at one time so that the change error in the input feedstock could be greatly reduced. Acetone and 2-propanol were used for the separation of oils and was purchased from Merck, Germany.

### *Pyrolysis process*

The decomposition of waste tire took place within a fixed bed reactor, comprising a 316 SS tube with a diameter of 33mm, length of 25cm, and a volume of 105ml. Before each experiment, 20g of rubber powder was introduced into the reactor and subjected to heating using an electric laboratory chamber furnace (Oxiton model 1200 made in Iran). The specific time and temperature parameters were chosen based on the experimental design for each trial. To mitigate the influence of residual oxygen on pyrolysis products, nitrogen gas was purged into the reactor. Upon

reaching the designated reaction temperatures (400-600 °C), the sealed reactor was positioned inside the furnace for a predetermined duration. Subsequently, the reactor was removed from the electric furnace and allowed to cool gradually to room temperature, followed by further cooling in a refrigerator for 2 hours. To assess the impact of temperature on pyrolysis products, the reactor was weighed, and the generated gas was extracted upon opening the reactor. The difference in weight was reported as gas yields. The residual pyrolysis products were immersed in a solution of 2-propanol and acetone solvent at room temperature, and the mixture was stirred for 1 hour. Afterward, the solution was meticulously rinsed and filtered, and the remaining solid underwent overnight drying in an oven at 80°C. This residue constituted the char yield, predominantly composed of carbon black. The solvents utilized in the filtration process were separated under reduced pressure using a vacuum evaporator (Hahan Wapor model HS-2005s from Hahanshin company, South Korea). Following distillation, a nearly light phase was obtained from each liquid. These collected phases were identified as pyrolysis oil and stored for subsequent characterization. Experiments were performed three times each, and the mean of each group was presented. A schematic representation of the pyrolysis process is illustrated in Fig. 1.

### *Analysis and characterization*

Thermogravimetric Analysis (TGA) was conducted using a thermogravimetric analyzer (TGA Mettler-Toledo/DSC/TGA1 Instruments, Switzerland) to explore the thermal characteristics of the feed material. Approximately 10–15 mg of rubber samples were employed for TGA experiments. All experiments were executed within the temperature range of 30 to 700 °C, employing a heating rate of 10 °C/min, and conducted under a nitrogen gas purge. Fourier transform infrared spectroscopy (FTIR) was employed to analyze all pyrolysis oils (model EQUINOX55, Bruker). The FTIR analysis involved 64 scans at a resolution of 4/cm in the 400 to 4000/cm range to obtain the spectra of pyrolysis oils. Detailed Hydrocarbon Analysis (DHA) was performed using a VARIAN CP-3800 instrument equipped with a Flame Ionization Detector (FID). Separations were carried out using a fumed silica capillary column (CP-SIL PONA CB) with a length of 100 m and an internal diameter

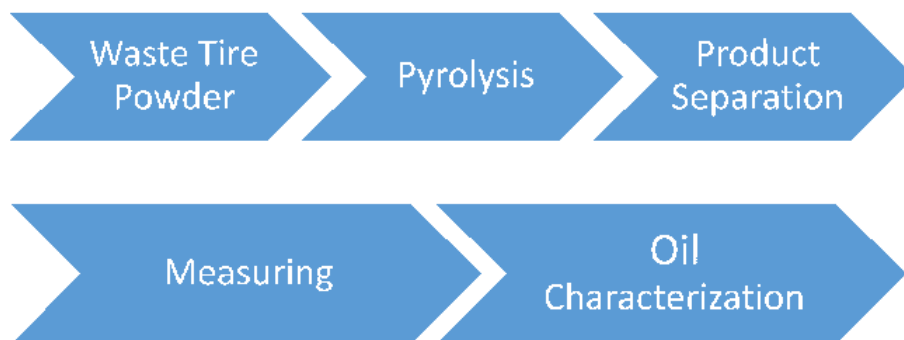


Fig.1: Schematic diagram of the pyrolysis process

of 0.25 mm. The temperature was programmed from 50 to 295 °C at a rate of 4 °C/min, with helium serving as the carrier gas. For measuring the heat capacity of obtained oils, a bomb calorimeter (Parr instrument, model 1256) was employed. The bomb calorimeter, a type of constant-volume calorimeter, was utilized to determine the heat of combustion for various types of burning samples (Abdoli and Ghasemzadeh, 2024). Each experiment involved the use of approximately 1-1.5 g of samples. The values of the results presented in each section are the average of three times of analysis.

## RESULTS AND DISCUSSION

### TGA experiments

Fig. 2 displays the TGA and Differential Thermogravimetric (DTG) curves for the scrap tire powder. The TGA curves indicate that thermal degradation occurred across a broad range from 200 to 500 °C, while the DTG curves distinctly reveal three decomposition regions. The initial weight loss at 200-300 °C is attributed to the degradation or volatilization of additives such as stearic acid and aromatic-naphthenic oils employed in the tire manufacturing process. The subsequent weight loss (300-380 °C) primarily results from the degradation of NR, and the third weight loss (380-500 °C) is associated with the decomposition of SBR and BR. NR and SBR constitute the main components of tires, while BR is commonly used as a liner for certain types of tires. These findings align with a prior study that utilized DTG peak temperatures of 378 °C, 458 °C, and 468 °C for NR, SBR, and BR, respectively (Moustafa et al., 2016). The insights gained from this TGA study provide a foundation for designing our pyrolysis experiment.

According to the TGA analysis, an optimal pyrolysis temperature range of 400-600 °C is recommended to achieve complete decomposition of waste tires.

### Effect of temperature

Table 1 outlines the impact of pyrolysis temperatures (400, 450, 500, 550, and 600 °C) on the product yields. The gas yield was determined by the disparity between the quantity of feed material and the combined amount of oil and pyrolysis char. Pyrolysis oil yields ranged from 20% to 32.5% by weight of the tire waste. Char yield calculations were performed after washing and drying the pyrolysis char in an oven overnight.

Table 1 illustrates that the yields of pyrolysis products exhibit a strong dependency on the chosen pyrolysis temperatures. Optimal pyrolysis conditions seem to be around 500 °C, as at this temperature range, the yields are most favorable. Notably, increasing the pyrolysis temperature beyond this point primarily promotes the degradation of oil, converting it into gas products, mainly due to secondary cracking reactions. The solid residue from pyrolysis comprises reinforcing carbon black used in tire production and other inorganic compounds incorporated during the manufacturing process (Czajczyńska et al., 2017; Ghasemzadeh et al., 2022b, 2022c). Char yield fluctuates within the range of about 45 to 60 wt.%, while the gas obtained from pyrolysis can vary from 5-30 wt.% of the products, depending on the pyrolysis temperature. Interestingly, as the pyrolysis temperature increases, the char yield remains relatively constant, hovering around 45 wt.%. The correlation between product yields and temperature is visually represented in Fig. 3.

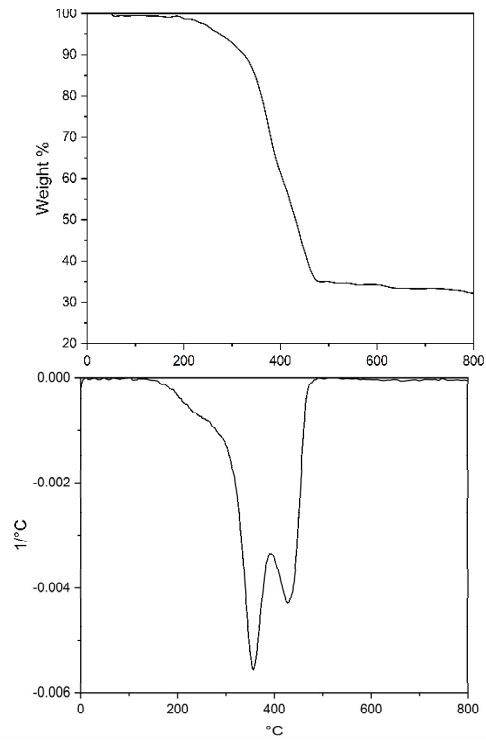


Fig. 2: Thermogravimetric analysis of the scrap tire powder

Table 1: Effect of temperature on the yields of pyrolysis products

Pyrolysis temperature (°C)	Char yield (%)	Oil yield (%)	Gas yield (%)
400	60	32.5	5
450	55	30	10
500	50	27.5	17.5
550	45	22.5	25
600	45	20	30

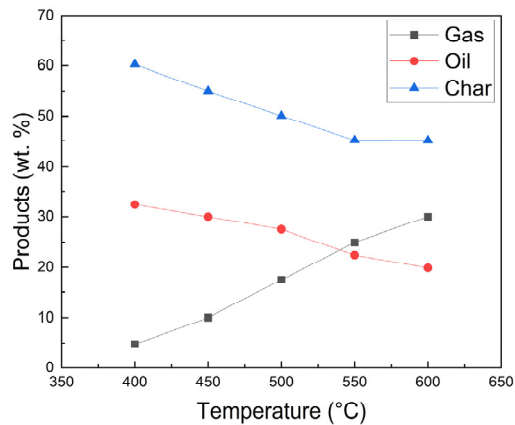


Fig. 3: Correlation between product yields and temperature

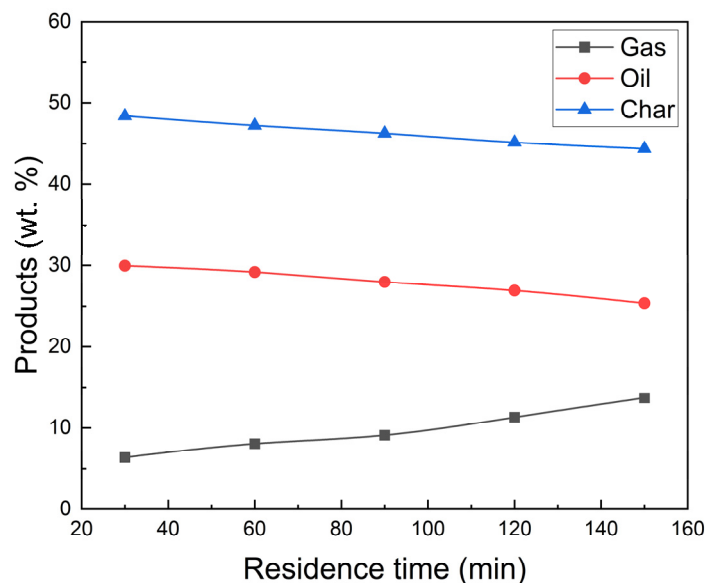


Fig.4: Effect of residence time on yields of pyrolysis products

#### Effect of residence time

Fig.4 shows the effect of residence time on yields of pyrolysis products at 500 °C. Different residence times such as 30, 60, 90, 120, and 150 min were examined for the pyrolysis procedure at 500 °C. As the figure shows, only slight changes are obvious in trends of yielding products. This is more obvious in the case of gases, which are increasing with increasing residence time. So in conclusion, a longer residence time has a weak influence on the yields of pyrolysis products but the direction was the same as the pyrolysis temperature for the gas product.

#### FTIR analysis

FTIR was employed to characterize pyrolysis oils obtained through the degradation of tire wastes at four distinct temperatures ranging from 400 to 550 °C. The analysis was conducted at room temperature using Potassium Bromide (KBr) discs and recorded over a wavenumber range of 4000–400  $\text{cm}^{-1}$  (Fig. 5a). The IR spectra exhibit several characteristic peaks, including broadband indicative of the OH group at 3400/cm (Samimi, 2024). Additionally, absorption peaks at 3045 and 3014/cm were assigned to the C=C stretching vibrations. A narrow and moderately intense peak at approximately 1600/cm corresponds to characteristic vibrations of the aromatic nucleus

(C=C), such as the benzene ring. An intense peak at 1200/cm is typical of C-O bonds. Bands observed in the range of 1000–1150/cm were attributed to the C–O–C stretching vibrations, while the stretching and bending vibrations at 2796–2950/cm and 1310–1465/cm were assigned to alkyl groups, such as  $\text{CH}_2$  and  $\text{CH}_3$ , present in the obtained chemicals. The sample spectra of pyrolysis oil at 500 °C, along with comparisons at four different temperatures, are provided in Fig. 5b.

Although the obtained results show more or less similar absorption there are some differences between FTIR absorption of the produced oils in different pyrolysis temperatures. The evidence of FTIR spectroscopy wavenumber around 1600 and 3000/cm shows by increasing the pyrolysis temperature from 400 to 550 °C the amount of aromatic structure increases. This observation is presented in Fig. 6. This is quite obvious from sharper absorption peaks around 1600/cm and above 3000/cm which belong to aromatic structures. These bands were very small at lower temperatures and increased with increasing pyrolysis temperature, indicating that higher concentrations of aromatics were presented in higher pyrolysis temperatures. Similar results have been reported by other studies (López et al., 2013; Maleki Delarestaghi et al., 2018;

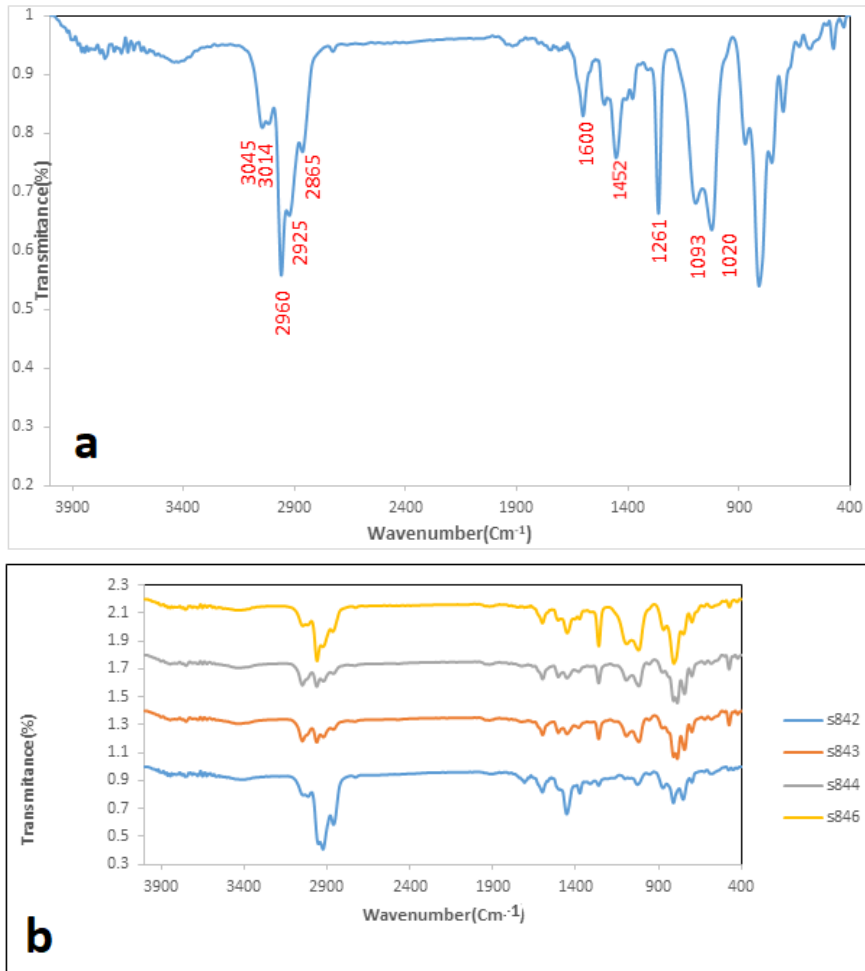


Fig. 5: FTIR spectra of pyrolysis oil a) as a sample at 500°C, b) comparing at 4 different temperatures

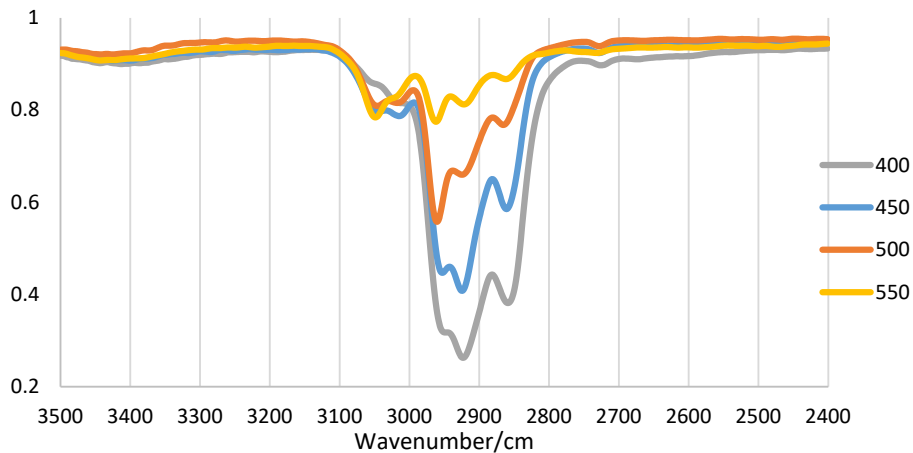


Fig. 6: Comparison between FTIR spectra of the oils produced in different pyrolysis temperatures.

Table 2: Detailed Hydrocarbon Analysis of pyrolysis oils

Sample number	Ret. Time (min)	Peak name (compound)	Pyrolysis temperature (°C)
1	8.398	1-Pentene	500
2	8.637	NC5	550
3	9.064	Cis-2-Pentene	400
4	9.215	2-Methylbutene	500
5	9.305	2,3-DMC4	400-550
6	81.979	1-m-4-Isop-benzene	400-550
7	100.115	t-IB4Ebenzene	550

Table 3: Calorific value results of waste tire pyrolysis oils

Sample number	Pyrolysis temperature (°C)	Energy (Cal/g)
1	400	10309
2	450	9800
3	500	9667
4	550	8973

Ramirez-Canon *et al.*, 2018). Aromatic formation reaction at higher temperatures is mainly due to Diels-Alder and recombination reactions between aliphatic and aromatic free radicals. It is noteworthy to mention that the chemical compositions of the pyrolysis oils are intricate due to the utilization of various types and mixtures of scrap tires in the pyrolysis process. The diverse nature of the input materials contributes to the complexity of the resulting chemical compositions in the pyrolysis oils.

#### Detailed Hydrocarbon analysis

DHA was performed on pyrolysis oils obtained within the temperature range of 400-550°C, and the outcomes are presented in Table 2. In summary, it can be noted that the composition of pyrolysis oils is highly intricate due to the variations in scrap tires and pyrolysis temperatures. The results indicate a diverse mixture of organic compounds ranging from 5 to 20 carbons, with a predominant presence of aromatics. Some of the identified chemicals include 1-pentene, 2-methylbutene, 2,3-dimethylbutane (DMC4), 1-m-4-Isop-benzene, and t-IB4Ebenzene. The complexity of the obtained chemicals underscores the influence of both the tire composition and the pyrolysis conditions on the resulting hydrocarbon composition. It is quite obvious that by increasing the degradation temperature, the amount of aromatic structures increases. This may be because of free radical recombination reactions and cyclization of aliphatic

chains. Similar results have been obtained by another study and are also confirmed by our FTIR spectroscopic study (Diez *et al.*, 2004).

#### Calorific value

Bomb calorimetry was employed to measure the calorific value (heat of combustion) of pyrolysis oils according to the ASTM D4809 standard method. In general, pyrolysis oils obtained from recycled tires exhibited a high calorific value comparable to that of commercial heating oils. This similarity renders them an attractive alternative and valuable fuel source. The results obtained through bomb calorimetry or energy content per gram of oil (Cal/g) at different pyrolysis temperatures are presented in Table 3, providing insights into the heat release capabilities of the pyrolysis oils. Within the temperature range of 400 to 550 °C, the energy content of the produced oil has decreased from 10,309 to 8,973, indicating a 13% reduction. This implies that when the temperature of the pyrolysis process increased, the energy content of the produced oil decreased. These findings align with other studies in the field (Heidary, 2017; Diez *et al.*, 2004).

The utilization of waste tire pyrolysis emerges as a valuable avenue for extracting oil from discarded materials, offering a solution with multifaceted economic benefits. The resulting oil showcases a spectrum of properties contingent upon diverse process parameters, including residence time and temperature. While some studies have



explored the economic feasibility of pyrolysis processes, in Iran's context, characterized by relatively low energy prices, economic viability pivots on addressing environmental concerns or capitalizing on government incentives directed toward bolstering sustainable energy production. Various factors, spanning plant longevity, feedstock composition, technological variables, and biomass pricing, collectively shape the economic feasibility of oil extraction through pyrolysis. Key to evaluating market competitiveness against other biofuels is the production cost linked with char and oil. This cost is susceptible to influence from a range of factors encompassing pretreatment methods, upgrading techniques, and recycling practices. Approaches such as biomass torrefaction as a pretreatment strategy and the adoption of cost-efficient catalysts for oil refinement show promise in rendering biomass pyrolysis for oil extraction financially viable. Additionally, implementing a self-sustaining pyrolysis process holds the potential for substantial cost reductions, positioning it as the most economically advantageous option on a commercial scale. The study focused on examining key parameters such as temperature and residence time in pyrolysis processes, aiming to evaluate resulting oils qualitatively and quantitatively in comparison to alternative products. However, potential sources of error exist within the analysis, and the underlying assumptions require improvement to enhance reliability. These sources of error include inaccuracies in measurement precision, risks of sample contamination, variability in feedstock characteristics, and assumptions made during analysis. To address these issues, researchers could refine measurement techniques, implement robust quality control measures, standardize feedstock characteristics, and validate assumptions through sensitivity analyses or control experiments. By mitigating these potential sources of error and enhancing underlying assumptions, the study can achieve greater reliability and validity in its findings, ultimately strengthening the significance of its conclusions.

## **CONCLUSION**

The rising global population and increased transportation needs have led to a significant annual production of billions of waste tires. To address the

environmental impact, there is a growing focus on repurposing these tires as secondary raw materials and energy sources. Despite challenges posed by tire resilience, a recent study advocates for using pyrolysis as an eco-friendly method for recycling tires. The research explores key parameters like temperature and residence time in pyrolysis, emphasizing both qualitative and quantitative analyses of resulting oils compared to other products. The goal is to contribute to sustainable tire waste management and resource recovery. This study investigated the pyrolysis of waste tire powder at various temperatures and yielded pyrolysis oils, with a notable range in yields (20 to 32.5 wt.%), dependent on the final pyrolysis temperature. This process also generated pyrolysis char (45 to 60 wt.%) and gas (5 to 30 wt.%). Detailed Hydrocarbon Analysis identified key compounds in the pyrolysis oils, including 1-pentene, 2-methylene, 2,3-dimethylbutane, 1-m-4-Isop-benzene, and t-IB-4Ebenzene. Fourier Transform Infrared Spectroscopy revealed a temperature-dependent shift, with an increase in aromatic structures and a decrease in aliphatic components. Moreover, the calorific value comparison demonstrated that pyrolysis oils from waste tires are on par with commercial heating oils, indicating their potential as a valuable alternative fuel source. This comprehensive analysis underscores the versatility of waste tire pyrolysis in producing oils with diverse compositions and energy characteristics. The findings contribute to understanding the feasibility and potential applications of waste tire pyrolysis in sustainable energy solutions.

## **AUTHOR CONTRIBUTIONS**

A. Pazoki performed conceptualization, investigation, and interpreted the data. R. Ghasemzadeh wrote the draft manuscript and reviewed the literature. M. Barikani performed experiments and investigation in the literature review and manuscript preparation. M. Pazoki analyzed and interpreted the data.

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### CONFLICT OF INTEREST

The authors declare no potential conflict of interest regarding the publication of this work. In addition, the ethical issues including plagiarism, informed consent, misconduct, data fabrication and, or falsification, double publication and, or submission, and redundancy have been completely witnessed by the authors.

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### ABBREVIATIONS (NOMENCLATURE)

<i>BR</i>	Polybutadiene Rubber
<i>DHA</i>	Detailed Hydrocarbon Analysis
<i>DTG</i>	Differential Thermogravimetric
<i>FTIR</i>	Fourier Transform Infrared Spectroscopy
<i>FID</i>	Flame Ionization Detector
<i>KBr</i>	Potassium Bromide
<i>NR</i>	Natural Rubber
<i>SBR</i>	Styrene Butadiene Rubber

TGA Thermogravimetric Analysis

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