CHARACTERIZATION OF ALIPHATIC COMPOUNDS OBTAINED FROM PROLYSIS OF SUNFLOWER-EXTRACTED BAGASSE

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Biomass, pyrolysis, aliphatics, characterization, column chromatography

Abstract

In this study, aliphatic compounds obtained by pyrolysis of sunflower-extracted bagasse were characterized by column chromatography method. The pyrolysis liquid product was separated into two fractions using pentane: pentane-soluble (aliphatic, aromatic, polar fractions) and insoluble (asphaltic) compounds. The chemical composition of the aliphatic fraction was investigated using chromatographic and spectroscopic techniques (FTIR, column chromatography and GC). According to column chromatography results, pyrolysis oil consists of 52 % aliphatic, aromatic, polar fractions and 48 % asphalten. Column chromatography indicated that the pyrolysis liquid product consists of 8,58 % aliphatic, 19,76 % aromatic and 23,66 % polar fractions. Furthermore, it was shown that the aliphatic compounds mixtures (including from C_{23} to C_{31}) could be separated into their compounds by the column chromatography method. Chemical characterization of the pentane soluble fraction obtained from the pyrolysis of sunflower extraction bagasse indicated that the pentane soluble fraction could be a raw material source for aliphatic chemicals. The aliphatic compounds obtained by this method can be considered to be a useful alternative to diesel fuels because their carbon distribution is similar to that of diesel fuels.

AYÇİÇEK EKSTRAKSİYON KÜSPESİNİN PROLİZİNDEN ELDE EDİLEN ALİFATİK BİLEŞİKLERİN KARAKTERİZASYONU

Anahtar Kelimeler

Biyokütle, piroliz, alifatikler, karakterizasyon, kolon kromatografisi

Öz

Bu çalışmada, ayçiçek ekstraksiyon küspesinin pirolizinden elde edilen alifatik bileşikler kolon kromatografisi yöntemi ile karakterize edilmiştir. Piroliz sıvı ürünü pentan kullanılarak pentanda çözünen (alifatik, aromatik, polar fraksiyonlar) ve çözünmeyen (asfaltenler) bileşikleri olmak üzere iki fraksiyona ayrılmıştır. Alifatik fraksiyonun kimyasal bileşimi kromatografik ve spektroskopik teknikler (FTIR, kolon kromatografisi ve GC) kullanılarak incelenmiştir. Kolon kromatografisi sonuçlarına göre, piroliz yağı %52 alifatik, aromatik, polar fraksiyonlar ve %48 asfaltenden oluşmaktadır. Kolon kromatografisi, piroliz yağının %8,58 alifatik, %19,76 aromatik ve %23,66 polar fraksiyonlardan oluştuğunu göstermiştir. Ayrıca, alifatik bileşik karışımlarının (C_{23} ila C_{31} karbon içeren) kolon kromatografisi yöntemi ile bileşiklerine ayrılabileceği gösterilmiştir. Ayçiçek ekstraksiyon küspesinin pirolizinden elde edilen pentan çözünür fraksiyonunun kimyasal karakterizasyonu, pentan çözünür fraksiyonunun alifatik kimyasallar için hammadde kaynağı olabileceğini göstermiştir. Bu yöntemle elde edilen alifatik bileşikler, karbon dağılımları dizel yakıtlarına benzer olduğundan, dizel yakıtlarına faydalı bir alternatif olarak düşünülebilir.

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1. Introduction

Energy is one of the most important factors in economic and technological development of countries. Energy demand increases rapidly due to the growing population in the world. Because of the limited petroleum, natural gas and coal sources, the energy problem becomes the major concern for all countries. For this reason, the energy problem causes crises, tensions and even wars in the world. Petroleum, coal and natural gas are at present the principal sources of fuels and organic chemicals although they have high sulfur, nitrogen and metal content. In recent years, biomass and its products have been intensively investigated as alternative raw material sources for fuels and organic chemicals (Ashoor et al., 2023; Osman et al., 2023; Rahmawati et al., 2024; Zhou et al., 2023). The main advantages of using biomass are that it has negligible sulfur, nitrogen and metal content (Liu, Li, Jiang and Yu, 2017; Ngangyo et al., 2019).

In this study, sunflower-extracted bagasse was pyrolyzed in a fixed-bed Heinze retort. Pyrolysis liquid product oil was separated from asphalten by solving in pentane. The pentane eluate including aliphatic, aromatic and polar fractions was separated its fractions by column chromatography. Additionally, the aliphatic fraction was also separated into its components according to the carbon number distribution.

2. Scientific Literature Review

Various biomass sources such as sugar beet, euphorbia, sunflower, hazelnut, cotton etc. can be evaluated as potential fuel and chemical raw material sources (Afif, Anayah and Pfeifer, 2021; Avcı et al., 2025; Carrino, Visconti, Fiorentino and Fagnano, 2020; Ebrahimian, Denayer, Aghbashlo, Tabatabaei and Karimi, 2022; Pfersich et al., 2020).

Synthetic fuel production studies became significant because petroleum, a primary energy source, has limited reserves. Pyrolysis of biomass is a main method used for this purpose. In a previous biomass pyrolysis study, nut shell was selected as biomass source and thermos chemically processed by fast pyrolysis under different conditions in a fixed bad tubular reactor. In that study, GC chromatography of the aliphatic fractions of the liquid product was compared with currently utilized transport fuels such as kerosene and diesel fuel and a big similarity was seen. Pyrolysis liquid product involving 22.7 % aliphatic hydrocarbon was determined in the same study (Onay and Koçkar, 1998). Cotton seed cake was pyrolyzed in a different study and aliphatic fractions of the pyrolysis liquid product gas chromatography showed hydrocarbon distribution between C_{10}-C_{32} (Pütün, Özbay, Koçkar and Pütün, 1997). Carbon distribution of the pentane eluate of pyrolysis liquid product of sunflower-extracted bagasse is as C_8-C_{31} in the literature. This carbon distribution is similar to that of the diesel fuel also (Yorgun, Şensöz and Koçkar, 2001a).

3. Materials

The extracted bagasse sample has been taken from some sunflower oil factories around the city of Eskişehir located in central Anatolia. Silica gel 60 (Merck KGaA, Germany) was used as column packing material. Pentane (Merck KGaA, Germany), Methanol (Merck KGaA, Germany) and Toluene (Merck KGaA, Germany) were used as solvents.

Elemental analysis of sunflower extraction bagasse was carried out with Carlo Erba EA 1108 device. The results of this analysis are shown in Table 1.

Table 1. Elemental Compositions of Sunflower-Extracted Bagasse

Component	%
C	58.2
H	7.0
N	8.0
O	26.8

3.1. Methods

The sample was first dried, then ground and sieved in a high-speed rotary shear mill. The liquid product obtained after pyrolysis was separated into pentane-soluble and pentane-insoluble fractions. The pentane eluate was separated into subproducts by passing through a silica gel column and analyzed by gas chromatography. The procedures for these processes are detailed below.

3.2. Pyrolysis

Pyrolysis experiments were carried out in a 316 stainless steel fixed bed reactor with a volume of 400 cm^3 (70 mm internal diameter), at different sweep gas (N_2) flow rates, in the particle size range of $0.425<Dp<0.850$ mm, with a heating rate of 7°C.min^{-1}, up to a final temperature of 500°C. The reactor was heated externally with an electric furnace while the temperature was controlled by a thermocouple located inside the bed. The experimental results obtained from the pyrolysis of the sample are given in Table 2.

Table 2. Pyrolysis Yields of Sunflower Extracted Bagasse

N_2, $cm^3.min^{-1}$	Oil, %	Gas, %	Char, %	Water, %
0	21.1	28.0	28.5	22.4
50	22.4	22.6	27.8	27.2
100	22.0	21.1	28.0	28.9
200	21.4	22.9	27.8	27.9
500	20.1	24.0	27.7	28.2

3.3. Column Chromatography

Chromatographic methods used to separate or purify chemicals use particles with diameters ranging from 1 µm to 250 µm, depending on the method type and the targeted separation sensitivity. These particles can be silica or polymer based. Surface properties of silica or polymeric particles used in chromatography can be changed by chemically modifying their surfaces with organic chains. Si-OH groups on the surface of natural silica can form hydrogen bonds with polar compounds present in the analyte, causing secondary interactions. These interactions result in the separation of components within the analyte, with greater or lesser retention within the column.

In this study, the oil under experimental conditions where the highest oil yield was obtained as a result of pyrolysis was passed through a column filled with active silica gel (70-230 mesh ASTM) in order to separate aliphatic, aromatic and polar compounds from each other. The method used for this purpose is as follows:

Silica gel activated at 600°C for 8 hours was cooled to room temperature in a porcelain crucible in a desiccator. Exactly 1.00 g of the pyrolysis oil was kept in pentane for 24 hours. Thus, aliphatic, aromatic and polar compounds were taken into the solvent pentane. Asphaltenes in the pyrolysis oil were removed by separating the liquid phase. The obtained aliphatic, aromatic and polar compounds mixture was mixed with the activated silica gel and charged to the top of the previously prepared 20 cm height and 2.5 cm i.d. column filled with activated silica gel and pentane. Then, 100 mL pentane was passed through this column and each 10 mL sample was collected in a separate bottle. Aliphatic compounds were separated from each other by this way. Then, 200 mL of toluene and 200 mL of methanol were passed through the column, respectively. In this way, the aromatic and polar fractions were separated from each other and collected in separate containers. The solvents in the obtained fractions were evaporated and the percentages of aliphatic, aromatic and polar compounds in the mixture were calculated.

3.4. Characterization

Pentane subfraction was subjected to Infrared spectra by using Mattson 1000 FTIR spectrophotometer. Each fraction was dried and weighed and then aliphatic compounds were subjected to elemental analysis (Carlo Erba, EA 1108) to determine the amounts of the carbon, hydrogen, nitrogen and oxygen. From the elemental analysis result, H/C ratio was determined. 10 mL pentane samples were analyzed by using a Hewlett-Packard 5890 A Gas Chromatography with flame ionization detector (FID) to compare them with conventional transport fuels.

All the experiments were carried out with parallels and the consistency of the results were checked.

4. Results

The volatiles, fixed carbon, ash and moisture amounts of the sample are shown in Table 3.

Table 3. Proximate Analysis of Sunflower-Extracted Bagasse (wt% as received)

Volatiles	78.4
Fixed carbon	10.5
Ash	6.1
Moisture	5.0

When the pentane-insoluble fraction was dried and weighed, it was found that the pyrolysis oil consisted of 48% asphaltenes. It was determined that the pentane-soluble part consisted of a mixture of aliphatic, aromatic and polar compounds at a rate of 52%. This mixture was separated by column chromatography and it was calculated that the pyrolysis oil consisted of 8.58% aliphatic, 19.76% aromatic and 23.66% polar compounds.

Fourier transform infrared (FTIR) spectra, representing functional group compositional analysis of pentane soluble material is shown in Figure 1. Symmetrical and asymmetrical C-H stretching vibrations of aliphatic CH_3 and CH_2 groups between 2800-3000 cm^{-1} and C-H bending vibrations of aliphatic CH_3 and CH_2 groups between 1350-1475 cm^{-1} indicate the presence of alkane groups in pentane soluble fraction. The presence of ketone, quinone, aldehyde groups etc. could be explain with C=O stretching vibration between 1650 and 1750 cm^{-1}. Aliphatic in plane and out of plane C-H bending vibrations were observed at nearly 700-800 cm^{-1}. These peaks indicate that hydrocarbons with more than seven carbon atoms are present in the pentane soluble fraction (Pütün et al., 1997). The elemental analysis of the aliphatic compounds obtained from the pyrolysis oil is shown in Table 4 and their gas chromatograms are in Figure 2. According to the results obtained from gas chromatography, the aliphatic compounds including C_{23}-C_{31} were in the first 10-mL sample passed through the column (Figure 2 (a)). The same content was determined for the second 10-mL sample as can be seen in Figure 2 (b). In the third 10-mL sample, all the aliphatic compounds except the ones having C_8, C_{10}, and C_{12} passed according to the Figure 2 (c). The chromatogram of the fourth 10-mL sample is shown in Figure 2 (d). Since the chromatograms of the samples taken after the fourth 10 mL were similar, the GC chromatograms after the fourth sample were not given.

Table 4. Elemental Analysis of Aliphatic Compounds Obtained from Pyrolysis Oil

Compound	Amount (%)	Mol Number	H/C ratio
C	79.21	6.6008	
H	12.72	12.7200	$CH_{1,9270}O_{0,0764}$
N	-	-	H/C = 1,9270
O	8.07	0.5044	

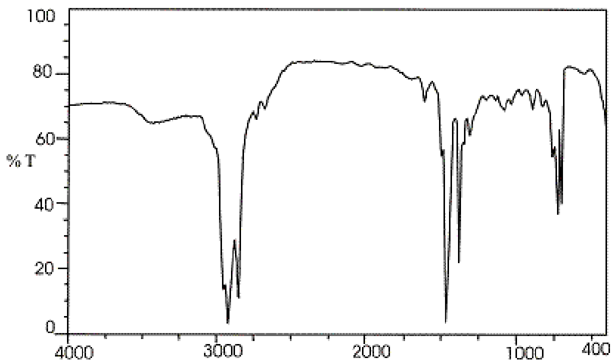
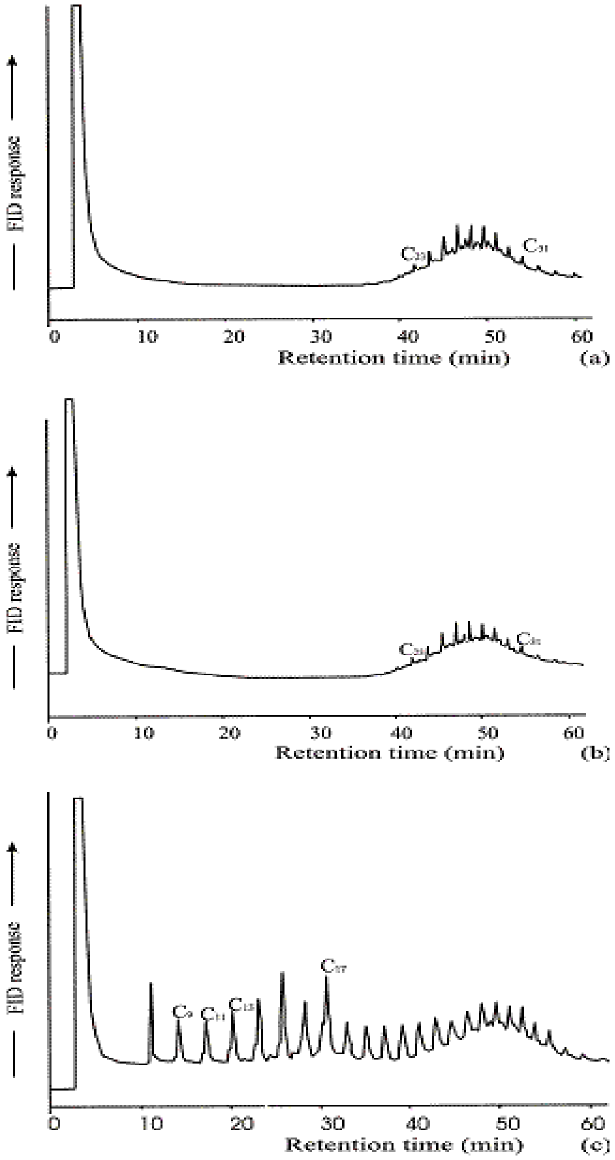


Figure 1. FTIR Spectra of Aliphatic Compounds Obtained from Pyrolysis Oil.



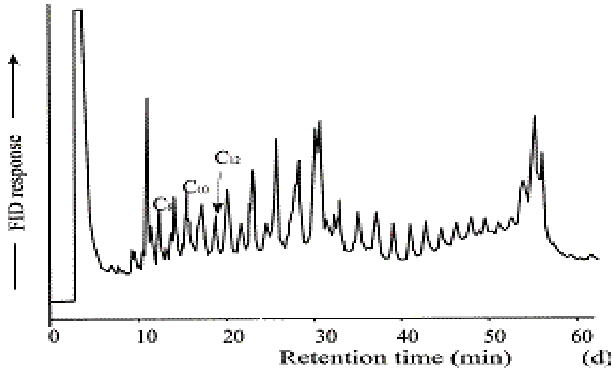


Figure 2. Gas Chromatograph of Aliphatic Compounds Obtained from Pyrolysis Oil. (a; first 10-mL, b; second 10-mL, c; third 10-mL, d; fourth 10-mL).

5. Discussion

It is known that pyrolytic oil/bio-oil, or a mixed hydrocarbon mixture, is produced by the chemical decomposition of compounds containing high amounts of organic matter, such as biomass, by heating them at high temperatures in an oxygen-free environment. The purpose of this study is not to determine the conditions that yield the highest pyrolytic oil from sunflower extracted bagasse.

The primary purpose of this study is to investigate whether pyrolytic oil obtained from the pyrolysis of any biomass can be purified into more valuable byproducts using simple chemical processes. Based on this, it was demonstrated that the pyrolytic oil obtained in this study can be separated from asphaltenes by first extracting the aliphatic, aromatic, and polar compounds present in the mixture using pentane solvent.

This study also demonstrated that pyrolysis oil can be separated into fractions using a simple, inexpensive and hand-made silica column.

When Figures 2 a and b are examined, it is seen that the first compounds to separate from the mixture are those containing between 23 and 31 C. This is likely due to the high molar mass of these compounds. This method can also be easily used in the purification of valuable chemicals. Figure 2 (d) shows that carbon distribution lies between C_8 and C_{31}.

When this results are compared with diesel chromatogram, carbon distribution of the sample is found similar to diesel fuel. For this reason, the aliphatic compounds obtained by this method are considered to be a useful alternative to diesel fuels and chemical feedstocks (Pütün et al., 1999; Yorgun et al., 2001a; Yorgun, Şensöz and Koçkar, 2001b).

Conflict of Interest

There is no conflict of interest with any person or institution in the study.

Authorship Contribution Statement

In this research, the experiments, literature review, creation of figures, tables and articles, and interpretation of the results were done by Musa ŞÖLENER.

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