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Study of solid residues obtained from the pyrolysis of commercial plastic waste bottles by FTIR and TG methods

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ABSTRACT

The composition of solid residues-products of thermal pyrolysis of plastic waste (polyethylene terephthalate (PET), polypropylene (PP)) at 350-550 °C was studied by Fourier transform infrared spectroscopy (FT-IR) and differential thermal analysis (DTA) methods. On the basis of the transition band (T%) the absorption and Abs parameters were calculated. It was observed that the Abs parameters of the peaks observed in the initial samples appear to change depending on the pyrolysis temperature, with the appearance of new peaks at higher temperatures. It was observed that during the pyrolysis of PET polymer waste, a number of bands with wavenumber 1692, 1670, 1262, 755, 694, and 464 cm^{-1} occurred at above 450 °C. It would seem that the Abs parameters for the 2923, 1453, and 846 cm^{-1} peaks observed in the initial samples are equal to zero. It would appear that during the pyrolysis of the PET polymer waste at 550 °C, only three peaks with wavenumbers of 1686, 1062 and 707 cm^{-1} are observed. Similarly, during the pyrolysis of the PP polymer waste at the same temperature, only one new peak (1092 cm^{-1}) is observed. The solid residues of the pyrolysis processes for the samples taken at 550 °C are calculated and are equal to 13.6 and 0.6%, respectively, for PET and PP. The data shows that solid residues from the pyrolysis of PP wastes have a structure similar to that of charcoal.

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1. Introduction

The plastic pyrolysis process involves the thermal degradation of post-consumer plastic waste in the absence of oxygen to produce valuable products such as fuel oil and solid residues such as charcoal and gas. Post-consumer plastic waste is believed to be a significant source of feedstock for plastic waste pyrolysis, as it is widely available and relatively easy to collect [1,2]. Unlike gas and fuel oil fractions, the composition of charcoal is not clear unambiguous, creating difficulties in its application for various purposes. The pyrolysis process of polyethylene terephthalate (PET) and polypropylene (PP) plastic waste has been studied in a number of works. In experiments conducted by different authors, the yield values of pyrolysis products were obtained differently. The amounts of gaseous, liquid products and solid residues of PET waste pyrolysis at 500 °C and heating rates of 10 °C/min are 52.13, 39.89 and 8.98 %, respectively [3]. The yields of gaseous, liquid products and solid residues during the pyrolysis of PP waste at 380 °C were 6.6, 80.1, and 13.3%, respectively [4]. In general, the yield of products during the pyrolysis of both polymers depends on the temperature and the type of pyrolysis reactor. The yield of liquid products during the pyrolysis of PP decreases at temperatures above 500 °C. At 740 °C, the yields of liquid, gaseous, and coal were 48.8, 49.6, and 1.6%, respectively [5-7]. Char formation increased from 2.0 to 4.0% in PP pyrolysis and from 0.7 to 2.0% in PE pyrolysis as the temperature increased from 668 to 746 °C. Unfortunately, the char obtained from both plastics consisted mainly of inorganic matter up to 98.9%, which originated from the inorganic substance in the feed fraction. In this case, the high inorganic matter made the application of char as fuel difficult. However, it still has the potential to be used as road surfacing and as a building material, char can also be used as an adsorbent in water treatment to remove heavy metals through an upgrading treatment [8-10].

The purpose of this study is to gain insight into the composition of solid residues resulting from the pyrolysis of different types of plastic waste, namely PET and PP experiments carried out at temperatures of 350, 450 and 550 °C. Polymer waste selected for the study exhibited a range of chemical structures, and a comparative analysis of the observed chemical changes may prove beneficial in understanding the pyrolysis of plastic wastes.

2. Experimental

2.1. Materials

Plastic bottles were acquired from local markets, super-markets and retail stores, where these products are regularly

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(a) (b) (c)

Figure 1. Photo of solid residues after 10 min of pyrolysis at (a) 350 °C, (b) 450 °C, and (c) 550 °C of PET-1 samples.

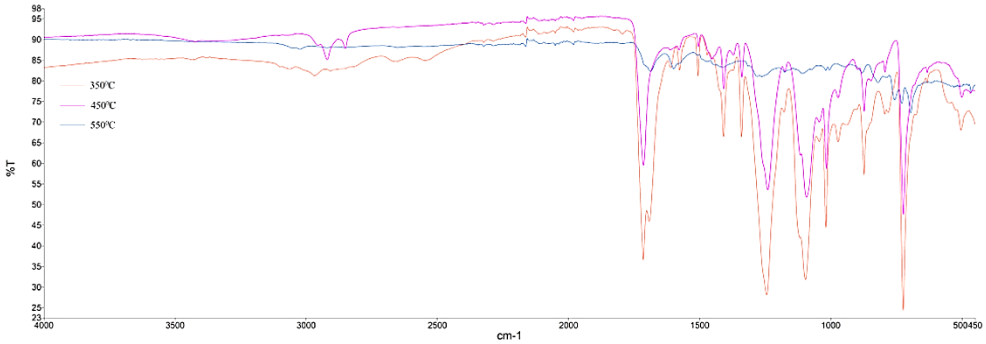


Figure 2. FT-IR spectra of the solid residues obtained from PET-1 pyrolysis.

sold for consumer use. This selection was made to reflect the typical plastic waste found in commercial and household environments. In our experiments, we used commercially available plastic bottles: PET-Transparent fizzy lemonade bottles (PET-1), PET-Light blue transparent Sirab bottles (PET-2), and PP-Gray colored perfume bottles (PP-3). These bottles were collected and carefully selected based on their composition and structural characteristics. Before analysis, the bottles were cleaned, dried, and cut into appropriate sizes for further processing. Samples were collected during 2024, ensuring that they were representative of commonly used commercial plastic packaging materials. After washing and drying, the collected samples were collected in a quartz ampoule in the form of small pieces with a width of 3 mm and a length of 4 mm weighing 0.06 g and placed in a special volume in the oven. The pyrolysis of the samples was carried out at 350, 450 and 550 °C in quartz vials for 10 minutes. The temperature of the furnace was measured with a thermocouple in the volume in which the sample was placed. Following the pyrolysis process, the solid residues were collected for FT-IR analysis. The obtained samples were characterized to determine their chemical composition and structural changes resulting from thermal decomposition. The solid residues shown in Figure 1 correspond to the pyrolysis of polyethylene terephthalate (PET). These residues were obtained after 10 minutes of pyrolysis at different temperatures: (a) 350 °C, (b) 450 °C, and (c) 550 °C. The observed morphological changes indicate the thermal degradation behavior of PET under these conditions.

2.2. Instrumentation

2.2.1. TG analysis of waste samples

The total yield of solid residues at the pyrolysis of PET and PP samples was calculated from TG curves. The Perkin Elmer Simultaneous Thermal Analyzer STA 6000 (USA) was used. PET samples (5.66-11.76 mg) with dimensions of 0.03-0.06 cm^2 were taken for analysis. In the device, these samples were heated up to a temperature of 700 °C under thermal conditions. The heating rate was maintained at 5 °C/min to ensure uniform thermal decomposition. The experiments were conducted under nitrogen gas conditions to analyze the degradation behavior of the polymers and accurately determine the yield of solid residues.

2.2.2. FT-IR analyses of solid residues

Samples of solid residues obtained from pyrolysis were compressed to a certain volume on a diamond surface under pressure and their spectra were obtained. The spectra of solid residues obtained from the pyrolysis by FT-IR Spectrophotometer (Spectrum 400 instrument, PerkinElmer, US) in the frequency range (T%) of 400-4000 cm^{-1} under normal incidence (ϕ = 90°).

3. Results and discussion

3.1. FT-IR analyses of solid residues

The FT-IR spectra of the solid residues obtained from the pyrolysis of PET transparent plastic bottles (PET-1) at 350, 450, and 550 °C are presented in Figure 2. The spectra display characteristic peaks corresponding to various functional groups and structural motifs present in PET and its degradation products. The broad peak of O-H stretching at 3424 cm^{-1} reflects the presence of hydroxyl groups, possibly from carboxylic acids or alcohols formed during degradation. Asymmetric C-H stretching vibrations are observed at 2966, 2923, and 2855 cm^{-1}, typical for aliphatic hydrocarbon chains. The strong absorption at 1713 cm^{-1} is attributed to the stretching of the C = O of the ester or carboxyl groups, a hallmark of the PET backbone and its decomposition intermediates. Aromatic character is evident from bands at 1611, 1504, and 1117 cm^{-1}, assigned to *mono*- and *para*-substituted benzene rings. The peaks at 1578 and 1262 cm^{-1} are associated with C=C and other aromatic stretching vibrations. Alkyl deformation vibrations are represented by CH_2 bending at 1453 cm^{-1} and alkane-related deformation at 1407 cm^{-1}.

Table 1. Abs parameters and band energy calculation reports for initial and pyrolyzed PET samples *.

ν (cm^{-1})	**Energy value, ×10^{-2}, Joule**	**Initial**		**350 °C**		**450 °C**	**550 °C**	
		PET-1	**PET-2**	**PET-1**	**PET-2**	**PET-1**	**PET-1**	**PET-2**
3424	6.78	0.04	-	0.05	-	-	0.05	-
2966	5.87	0.05	-	-	-	0.08	-	-
2923	5.79	0.06	0.04	0.07	0.02	-	-	-
2855	5.65	0.04	-	0.06	-	-	-	-
1713	3.39	1.00	-	0.22	-	0.43	-	0.02
1693	3.35	-	-	-	-	0.33	0.08	-
1611	3.19	-	-	-	-	0.08	0.08	-
1578	3.12	0.04	0.04	-	-	0.08	0.08	-
1504	2.98	0.07	0.06	0.05	0.03	0.09	-	-
1453	2.88	0.08	0.06	0.07	0.05	-	-	-
1407	2.78	0.19	0.18	0.10	0.09	0.17	-	0.03
1336	2.65	0.22	0.19	0.09	0.06	0.17	-	0.02
1262	2.50	-	-	-		-	0.09	
1244	2.46	0.82	0.75	-	0.22	0.55	-	0.03
1239	2.45	-		0.27		0.55	-	
1117	2.21	0.54	0.49	-	-	-	-	-
1097	2.17	0,70	0.73	0.28	0.24	0.49	-	-
1014	2.01	0.41	0.44	0.23	0.19	0.35	-	0.04
970	1.92	0.15	0.17	0.11	-	0.18	-	-
873	1.73	0.26	-	0.14	-	0.24	0.08	-
846	1.67	0.15	-	-	-	-	-	-
793	1.57	0.10	-	0.08	-	0.14	-	-
756	1.50	-	-	-	-	-	0.12	-
724	1.43	0.92	-	0.32	-	0.61	0.13	0.04
695	1.38	-	-	-	-	-	0.14	-
503	1.00	0.12	-	0.11	-	0.17	-	-
464	0.92	-	-	-	-	-	0.11	-

* PET-1 and PET-2 correspond to the different types of PET plastic bottles used in the pyrolysis experiments.

In the fingerprint region, the functionalities of the ether and ester contribute bands at 1244, 1239, and 1097 cm^{-1}, corresponding to the C-O and C-O-C stretches. Out-of-plane C-H bending and wagging modes related to aromatic structures are detected in the 873–724 cm^{-1} region [11].

Comparison of the spectra at different temperatures reveals progressive intensity changes and peak broadening, particularly at higher temperatures, indicating extensive chain scission, decarboxylation, and formation of new aromatic and aliphatic compounds. The FT-IR profile of PET samples from PET-1 showed similar peak positions and intensities, suggesting comparable chemical composition and thermal degradation behavior.

From the FT-IR spectra, the absorption (Abs) values for each observed peak were calculated and presented in Table 1. The calculations were performed using % Absorption = 100 - %T for percentage absorption and Abs = log 10 (I/I_0) = log 10 (1/T) = log 100/100-% Absorption for absorption (Abs). These equations were applied to calculate the Abs parameters from the FT-IR spectra for the chemical compounds in the solid residue of PET pyrolysis processes at different temperatures for PET-1 and PET-2 bottles.

The distinction between these two types of PET bottles is important because variations in additives, dyes, or manufacturing processes can influence the thermal degradation behavior, chemical composition, and absorbance (Abs) parameters obtained from FT-IR spectra. From the comparison of the Abs parameters, it can be seen that the highest absorption is 1713 cm^{-1} (C=O in the carbonyl group), 1244 cm^{-1} (in the plane the C-H bending bond and the C-O stretching bonds corresponding to ether (alkoxy) or alkonoate ester groups), 1097 cm^{-1} (C-O-C antisym.), 724 cm^{-1} (Ar-C-H wagg.) bands are observed [12]. The obtained results are related to the structure of PET polymer molecules. The intensity of peaks observed in the region of 2966, 1713, 1407, 1336, 1244, 1097, 1014 and 724 cm^{-1} decreases with increasing temperature, and is almost imperceptible in the sample pyrolyzed at 550 ℃. As the temperature of the pyrolysis process increases, a decrease in the value of the Abs parameters is observed, while the Abs parameters of some bands decrease to zero. That is, these bands are not observed at high temperatures. For some absorption bands, the value of the Abs parameters increases. 1693, 1611 cm^{-1} (monosubstituted benzene rings), 1262 cm^{-1} (C=C double bonding modes of vibration), 756 cm^{-1} (trans. conf. γ CH_2), bands at 695 and 464 cm^{-1} (parasubstituted benzene ring) appear only at high temperatures. This observation indicates that the chemical structure of the solid residue changes as the pyrolysis temperature increases, leading to the disappearance of some functional groups and the formation of new ones. The absence of certain bands at high temperatures suggests that the corresponding chemical structures decompose or transform as a result of thermal degradation. For example, ester groups in PET can break down, leading to the loss of their characteristic absorption bands. The increase in Abs parameters for some bands (eg, 1693, 1611, 1262, and 756 cm^{-1}) implies that certain chemical bonds become more prominent or concentrated in the residue as a result of pyrolysis reactions. The bands at 695 and 464 cm^{-1}, which are associated with parasubstituted benzene rings, appear only at high temperatures. This suggests that at elevated temperatures, PET undergoes structural rearrangements, leading to the formation of aromatic compounds with parasubstituted benzene rings. Overall, these spectral changes indicate the progressive breakdown of PET into smaller molecular fragments and the formation of new stable structures, such as aromatic rings, as the temperature increases. The temperature-dependent behavior of the absorption bands highlights the structural changes in PET during pyrolysis. At lower temperatures, functional groups such as carbonyls (C=O) and ethers dominate the spectrum. As the temperature rises, these groups degrade or transform, leading to a reduction in their Abs parameters. At higher temperatures, new bands emerge, signifying the formation of structures such as monosubstituted and parasubstituted benzene rings (mono- and parasubstituted) and new bonding modes, likely as a result of polymer decomposition and aromatic compound formation. This analysis aligns with the typical thermal degradation behavior of PET, where ester bonds break and volatile products form at elevated temperatures, leaving more stable aromatic structures [13].

Spectra of solid residue obtained from thermal pyrolysis of the PP sample pyrolyzed at 550 °C for 10 min and the initial PP sample are shown in Figure 3.

Table 2. Abs parameters and energy value calculated from FTIR spectra of chemical compounds in the solid residue of PP-3 pyrolysis.

ν (cm^{-1})	**E (Energy value), $\times 10^{-26}$ Joule**	**Abs**	
		Initial	**550 °C**
2950	5.84	0.11	-
2917	5.78	0.15	-
2839	5.62	0.073	-
1736	2.88	0.04	-
1456	2.88	0.08	0.01
1376	2.72	0.10	0.01
1167	2.31	0.05	-
1092	2.16	-	0,03
998	1.98	0.03	0.01
973	1.93	0.03	-
841	1.67	0.02	-

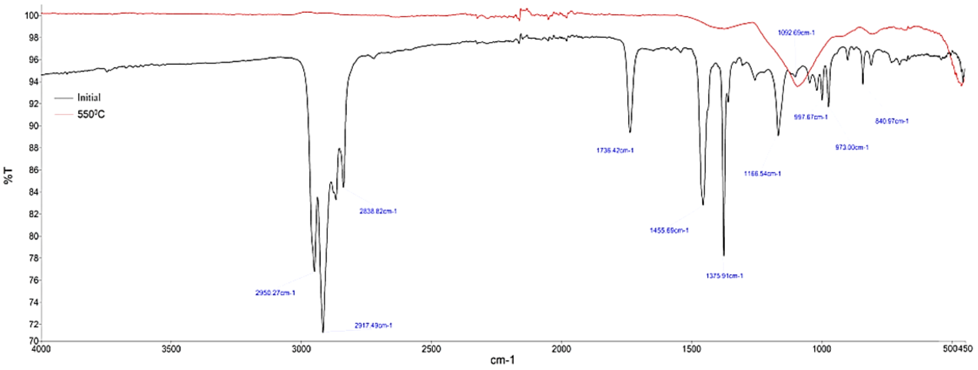
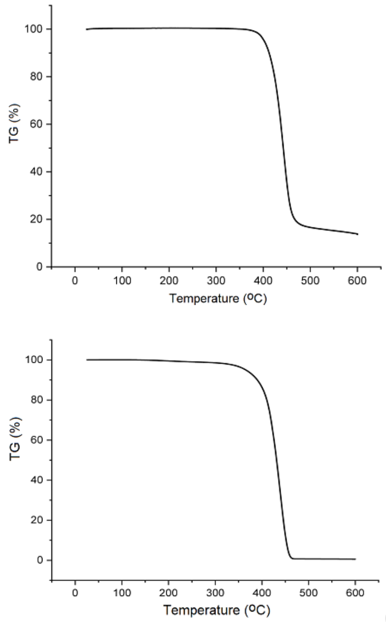


Figure 3. FT-IR spectra of PP (Gray-colored perfume bottles) at room temperature and after pyrolysis at 550 °C.



(a)

(b)

Figure 4. TG curves of the PET (a) and PP (b) waste samples.

From the spectral analysis the following transmitted peaks are identified: asymmetric and symmetric in-plane C-H ($-CH_3$) (2950, 2917, 2839 cm^{-1}), CH_2 deformation (1456 cm^{-1}), CH_3 symmetric bend, CH_2 wag (1376 cm^{-1}), wagging C-H (1167 cm^{-1}), Rocking CH_3, Rocking C-H (998 cm^{-1}), Rocking CH_3 (973 cm^{-1}) and CH_2 rock, CH bend (841 cm^{-1}). The calculated Abs for each wavelength number are presented in Table 2. Abs parameters of 2917 and 1376 cm^{-1} bands in the FT-IR spectra of the initial samples appear to be higher than others (Abs = 0.10-0.15). Shifts in the wavenumbers from the initial to 550 °C can indicate changes in molecular structure or bonds during the pyrolysis process. For example, the shift from 2950 (initial) to the 2917 cm^{-1} range suggests a possible change in the CH stretching vibrations of the alkyl groups. The absorption intensity (Abs) at various wavenumbers will tell you how much of the sample absorbs infrared light at specific frequencies. Higher absorption suggests the presence of a strong vibrational mode. For example, the peak at 2950 cm^{-1} shows a significant Abs value in the initial stage, but it is absent or not recorded for the 550 °C condition. This could mean that the chemical bonds at this wavenumber have been altered or degraded. The peaks around 2917, 2950, and 2838 cm^{-1} represent alkyl C-H stretching vibrations from alkyl groups, and changes here may suggest the breakdown or modification of polymer chains. The peak at 1736 cm^{-1} typically represents C=O stretching, which could indicate the presence of aldehydes or ketones, particularly when it becomes more prominent after heating. Peaks around 1455 and 1375 cm^{-1} often correspond to bending vibrations of the methylene (CH_2) or methyl (CH_3) groups, which could shift or change with thermal degradation.

The pyrolysis process significantly alters the molecular structure of the polypropylene (PP) bottle, breaking down long alkyl chains and leading to the formation of smaller molecules [14,15]. The C-H stretching vibrations decrease due to the breakdown of polymer chains, while the formation of carbonyl and oxygenated species increases due to thermal oxidation. The reduction in energy values at higher wavenumbers indicates bond weakening or the breaking of bonds, leading to smaller molecular fragments that absorb less energy. The shifts in absorption intensity and the emergence of carbonyl-related peaks highlight chemical transformations during pyrolysis, such as the creation of aldehydes, ketones, or other oxygenated compounds.

3.2. TG analysis of waste samples

TG curves of PET and PP waste samples are shown in Figure 4. The solid residues of the pyrolysis processes for the taken samples at 550 °C is calculated and are equal to 13.6 and 0.6%, respectively, for PET and PP. The data shows that solid residues from the pyrolysis of PP wastes has similar structure as charcoal. The calorific value of the char was approximately 18.84 MJ/kg and suitable for use as fuel, for example, in combustion with coal or other wastes. The chars were mainly mesoporous and macroporous material with adsorption capacities for methylene blue dye in the range of 3.59-22.2 mg/g. This indicates that upgraded chars should have good adsorption properties in bulky molecules and can also be used as an adsorbent in water treatment to remove heavy metals through an upgrade treatment [16-18].

4. Conclusions

The results of the calculations show that the parameters of the Abs peaks observed in the initial samples change as a function of the pyrolysis temperature, with new peaks appearing even at higher temperatures. It is found that during pyrolysis of PET polymer waste, parameters with wave-numbers 1693, 1670, 1262, 755, 694 and 464 cm^{-1} appear only at 450 °C. At the same time, the Abs parameters for the peaks 2923, 1453, and 846 cm^{-1} observed in the initial samples are zero. Only 3 peaks with wavelengths of 1686, 1062 and 707 cm^{-1} are observed in the pyrolysis of waste polymer PET. Only one new peak (1092 cm^{-1}) is observed at 550 °C in the pyrolysis of the PP waste polymer. The chemical transformations studied during pyrolysis were analyzed on the basis of obtained results.

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Disclosure statement

Conflict of interests: The authors declare that they have no conflict of interest.
Ethical approval: All ethical guidelines have been followed.
Sample availability: Samples of the compounds are available from the author.

CRediT authorship contribution statement

Conceptualization: Muslum Ahmed Gurbanov; Methodology: Ulviye Aydin Guliyeva, Elshen Valeh Mirzazada; Software: Muslum Ahmed Gurbanov, Ulviye Aydin Guliyeva; Validation: Muslum Ahmed Gurbanov, Ulviye Aydin Guliyeva; Formal Analysis: Muslum Ahmed Gurbanov, Ulviye Aydin Guliyeva; Investigation: Ulviye Aydin Guliyeva, Elshen Valeh Mirzazada; Resources: Muslum Ahmed Gurbanov, Elshen Valeh Mirzazada; Data Curation: Ulviye Aydin Guliyeva, Elshen Valeh Mirzazada; Writing - Original Draft: Ulviye Aydin Guliyeva, Elshen Valeh Mirzazada; Writing - Review and Editing: Muslum Ahmed Gurbanov, Ulviye Aydin Guliyeva, Elshen Valeh Mirzazada; Visualization: Muslum Ahmed Gurbanov, Ulviye Aydin Guliyeva; Funding acquisition: Ulviye Aydin Guliyeva, Muslum Ahmed Gurbanov; Supervision: Ulviye Aydin Guliyeva, Muslum Ahmed Gurbanov; Project Administration: Ulviye Aydin Guliyeva, Muslum Ahmed Gurbanov.

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