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Co-pyrolysis of Heavy Aromatic Waste and Polystyrene: Analysis of Liquid Product via GC-MS

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Abstract

Plastic wastes form 18.46 % of total municipal solid wastes in 2018 around the world. Heavy hydrocarbons occur as the waste of refinery processes. In this study co-pyrolysis of waste polystyrene foam (PS) and heavy aromatic waste (HAW) which can be accepted as a derivative of an oily sludge was carried out for the first time in literature. It was aimed to investigate the effect of temperature (420, 450, 480 °C) and mixing ratio (1/5, 3/5, 1/1) on liquid product yield and compound distribution in the pyrolytic liquids. Whole experiments were done in semi-batch reactor configuration under nitrogen atmosphere. Gas chromatography-Mass spectrometry (GC-MS) was utilized to analyze the pyrolytic liquids. At 480 °C it was obtained maximum liquid yield was 87.24 % in 1/5 HAW/PS feedstock ratio. Compared to pure PS pyrolysis, it was achieved relatively low styrene, toluene yield. However, yields of ethylbenzene and cumene increased with HAW added to the feed. Naphthalene was not detected in the liquid of PS pyrolysis. Maximum naphthalene yield was taken as 12.71% at 480 °C with a 1/5 HAW/PS feedstock ratio.

Keywords: Co-pyrolysis, GC-MS, heavy aromatic waste, plastic wastes, polystyrene

Ağır Aromatik Atık ve Polistirenin Eş-pirolizi: Sıvı Ürünün GC-MS ile Analizi

Öz

Plastik atıklar dünya çapında 2018 yılındaki toplam kentsel katı atıkların %18,46'sını oluşturur. Ağır hidrokarbonlar rafineri proseslerinin atığı olarak meydana gelir. Bu çalışmada, literatürde ilk defa atık polistiren köpük (PS) ve bir petrol çamuru türevi olan ağır aromatik atığın (HAW) eş-pirolizi yürütülmüştür. Sıcaklık (420, 450, 480 °C) ve karıştırma oranının (1/5, 3/5, 1/1) sıvı ürün verimi ve pirolitik sıvıların bileşik dağılımı üzerindeki etkisini incelemek amaçlanmıştır. Tüm deneyler yarı-kesikli reaktör konfigürasyonunda azot atmosferi altında yapılmıştır. Gaz kromatografi-Kütle spektrometresi (GC-MS) pirolitik sıvıları analiz etmek için kullanılmıştır. 480 °C'de 1/5 HAW/PS besleme oranında maksimum sıvı verimi %87.24 olarak elde edilmiştir. Saf PS pirolizi ile karşılaştırıldığında, nispeten düşük stiren ve toluene verimi edinilmiştir. Bununla birlikte, etilbenzen ve kümen verimleri beslemeye HAW katılmasıyla artmıştır. Naftalin PS pirolizinin sıvısında tespit edilmemiştir. Maksimum naftalin verimi 480 °C'de 1/5 HAW/PS besleme oranında % 12,71 olarak alınmıştır.

Anahtar Kelimeler: Eş-piroliz, GC-MS, ağır aromatik atık, plastik atıklar, polistiren

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1. Introduction

Municipal solid waste (MSW) means that a mixture of solid wastes which is thrown by people in the urban and rural areas [1]. According to the World Bank, it is expected that the amount of municipal solid waste will be up 3.4 billion tonnes by 2050 [2]. According to the United States Environmental Protection Agency, 146.1 million tonnes of MSW was landfilled in 2018 around the world. Plastic wastes form 18.46 % of this value [3]. The reason for this high amount originates from several advantages of plastics. Plastics have thermal and electrical insulation properties, hygienic for food packaging, resistance against chemicals and water, low weight for reducing transportation costs, durability, and longevity feature. But, they have some disadvantages too. Stabilizers, colorants, and phthalates are harmful materials that are used in plastic production. They occur as a threat to human life. In an environmental view, plastics are non-degradable, their breaking down can take a long time. Besides that, landfilling of plastic wastes occur a problem [4]. Recycling plastic wastes can occur in three ways: mechanical recycling, energy recovery, and chemical recovery. In the mechanical recycling route, it is benefited from expensive machines. In the energy recovery route, carbon dioxide (CO_2) which is the main greenhouse gas is released. Thus, the chemical recovery route comes to the forefront [5].

Pyrolysis is by far the most studied chemical recovery technique compared to liquefaction and gasification. In the pyrolysis technique, liquid fuel, solid residue, and gas products are obtained from several materials at the same time under an oxygen-free atmosphere. The pyrolytic product distribution majorly depends on the reaction temperature, heating rate, residence time, and reactor type [6].

Polystyrene is a thermally and mechanically stable material uses in food packaging, electronic, medical, and toy sectors [5]. From past to present, polystyrene pyrolysis in several reactor configurations has taken place in literature. Karaduman et al. (2001) carried out polystyrene pyrolysis in a free-fall reactor under vacuum to investigate the effect of particle size and temperature on liquid product yield and composition. They obtained a maximum liquid yield of 32 % at 750 ºC. At this temperature toluene, styrene, naphthalene yields were nearly 10, 20, 50 % respectively. They found also that large particle size (300-212 μm) maximized the liquid yield at 825 ºC [7]. Nisar et al. (2019) investigated the effect of reaction time and temperature on the liquid yield of polystyrene by using a pyrex glass reactor. They found that 410 ºC and 70 min at which liquid yield was almost 90 % were the ideal reaction conditions. Styrene/ethylbenzene yield at this condition was 35 % [8]. Park et al. (2020) studies two-step pyrolysis set up which included an auger reactor and a fluidized reactor. They aimed to increase benzene-toluene-ethyl benzene-xylene (BTEX) yield in the liquid product. They obtained a maximum BTEX yield of 26 % at 780 ºC in a fluidized bed reactor and 300ºC in an auger reactor. They found liquid yield as 86 % under these conditions [9]. In the last years, researchers have studied on co-pyrolysis of polystyrene with various materials such as low-rank coal [10], pinewood [11], sawdust [12], grape seeds [13], macroalgae [14], rubber seed [15], coffee grounds [16], other polymers [17] to determine synergistic effect between them.

Oily sludge is an industrial waste. It can occur during the production, refining, storage, and transportation of petroleum. Oily sludge is an emulsion that consists of a solid and liquid part. Because it includes components like heavy metals and polycyclic aromatic hydrocarbons (PAHs), oily sludge is accepted as hazardous and toxic for human health and the environment [18]. The hydrocarbon content of oily sludge can be between 5 and 86 % by mass. It can be possible to find anthracene, alkanes, cycloalkanes, benzene, toluene, xylenes, naphthalene, phenols in oily sludge [19]. There are several methods to dispose of oily sludge such as freeze/thaw method, ultrasonic/microwave irradiation, water/thermochemical cleaning, solvent extraction, centrifugation, biological treatment, advanced oxidation, solidification/stabilization, landfill, and thermochemical methods. Thermochemical methods which were used in oily sludge treatment are hydrothermal liquefaction, gasification, pyrolysis, incineration, thermal desorption. Hydrothermal liquefaction requires high temperature (200-600 ºC) and pressures (5-40 MPa), In thermal desorption which was seemed like the first stage of pyrolysis recovery of all hydrocarbons don’t occur completely. In gasification, syngas product has high impurity. In incineration, harmful gas emissions and high operating costs are disadvantages. In comparison with the other thermochemical methods, pyrolysis provides to obtain maximum oil recovery and less pollutant emission by far [20].

In refinery processes, components in crude oil are separated with respect to their boiling points. Heavy hydrocarbons forms as the waste of this process. They are converted to gas, naphtha, and light cycle oil via further treatments like catalytic cracking, alkylation, and hydrogenation [21]. To the best of our knowledge, there is no study about heavy aromatic waste which can be accepted as a derivative of an oily sludge and polystyrene pyrolysis in literature. This study it was aimed to investigate the effect of temperature and mixing ratio on liquid product yield and compound distribution in the liquid of these two materials.

2. Material and methods

Heavy aromatic waste (HAW) was supplied from a Turkish petrochemical company, TÜPRAŞ. Waste polystyrene foam (PS) was used in experiments. The experimental set-up was the same as our previous research [5]. Pyrolytic experiments were carried out pyrex glass reactor in the homemade ceramic furnace under a nitrogen atmosphere. The pyrolytic setup was shown in Fig. 1. The temperature was changed between 420-480 ºC with 30 ºC step size. The residence time was selected as 60 min. The weight percentage of HAW/PS was 1/5, 3/5, 1/1. Liquid product yield was calculated as follows:

Liquid product yield (%) = [collected liquid after the experiment, g/initial feedstock amount in the reactor, g]*100

Liquid products were collected by using a phase separator and sent to Gas Chromatography-Mass Spectroscopy (GC-MS) equipment. The brand and model of GC-MS were ThermoFinnigan and DSQ-250 respectively. Rtx-5 capillary column was used in the equipment. Helium whose flow rate was 0.7 mL/min was utilized as carrier gas. The temperature program of the column was started at 50 ºC. The column stayed at this temperature for 5 min. Then, the column temperature was increased by 270 ºC with a 5 ºC/min heating rate. And finally, the column was kept at this temperature for 20 min.

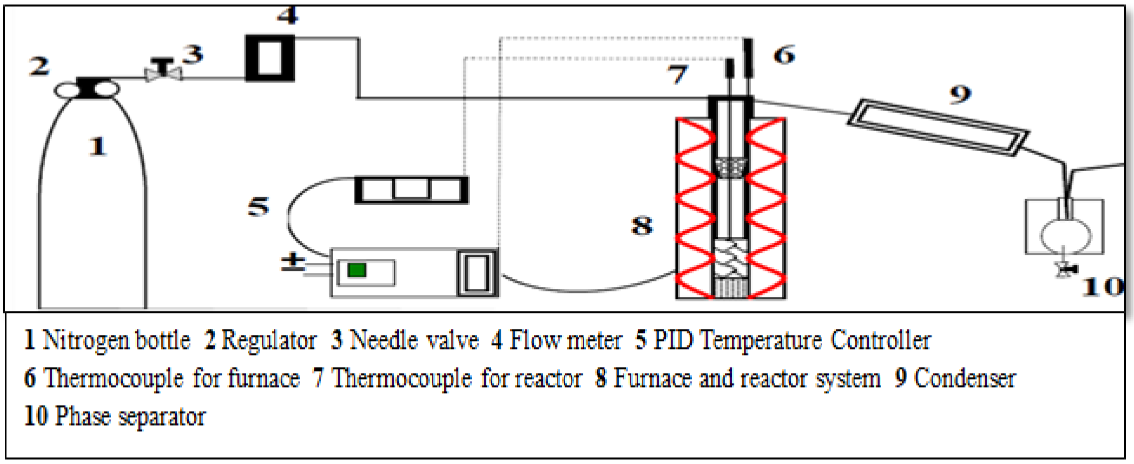

Figure 1. Experimental pyrolysis set-up [5]

3. Results and discussion

As shown in Fig. 2, it was found that liquid yield affected both temperature and feedstock composition. At 420 ºC it was obtained maximum liquid yield was 78.67 % in 1/5 feedstock ratio. This matter did not change at other temperatures. 480 ºC and 1/5 feedstock ratio formed the optimum condition for liquid yield. At this condition, the liquid yield was found as 87.24 %. It can be also said that there was no liquid yield difference between 480 and 450 ºC. Then, it was not tried further temperatures to investigate liquid yield. At 480 ºC PS pyrolytic liquid yield was found as 87.02 % which was lower than co-pyrolysis yield.

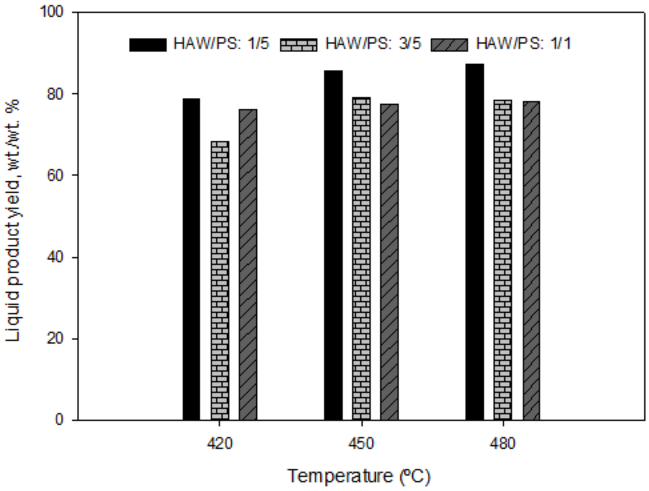

Figure 2. Changing in liquid yield with temperature and feedstock ratio

At the optimum condition, liquid product composition was investigated comprehensively and was compared to PS pyrolytic liquid at 480 ºC. The Chromatogram of PS pyrolytic liquid was shown in Fig. 3. Determined compounds in pyrolytic liquid of PS were given in Table 1. As shown in Table 1, the liquid product of polystyrene pyrolysis formed mainly C7-C17 hydrocarbons. There were some components whose peak areas were bigger than others. For example, Styrene yield was found as 43.74 %. Besides that, ethylbenzene and toluene yields were obtained as 4.16 and 6.15 % respectively. These components can be occurred because of further decomposition of styrene at 480 ºC [8].

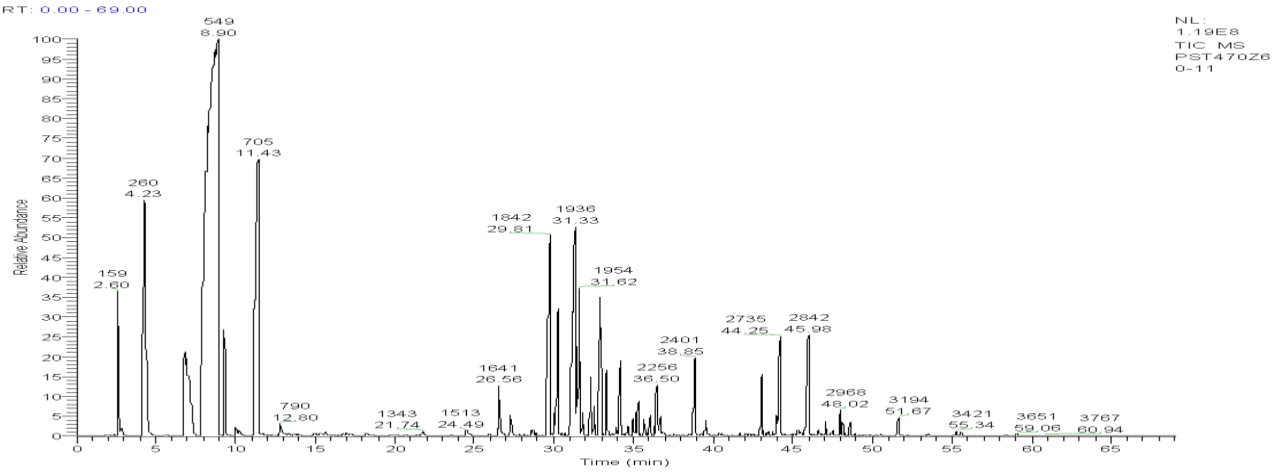

Figure 3. Chromatogram of the liquid product of PS at 480 ºC

Because maximum liquid yield was taken at 480 ºC and 1/5 HAW/PS ratio, this liquid was analyzed comprehensively. The results were given in Fig.4 and Table 2. As shown in Table 2, the liquid is composed of C5-C14 hydrocarbons mainly. With adding HAW to the feed, the styrene amount decreased to 8.08 % while the ethylbenzene amount increased a little bit. The main component of the liquid was found as naphthalene with a 12.71 percentage of peak area. It has been known that 2-methyl naphthalene which was in liquid with a relatively high amount can be utilized in polyethylenenaphthalate (PEN) production. PEN is a material that can be a rival to polyethylene terephthalate (PET) [22]. Toluene amount decreased with co-pyrolysis compared to PS pyrolysis.

Table 1. Compounds in PS pyrolytic liquid at 480 ºC

Retention time, min	Component	Formula	Peak area, %
4.23	Toluene	C_7H_8	6.15
6.82	Ethyl benzene	C_8H_{10}	4.16
8.90	Styrene	C_8H_8	43.74
9.31	Cumene	C_9H_{12}	0.86
11.43	Alpha-methyl styrene	C_9H_{10}	7.58
26.56	bibenzyl	$C_{14}H_{14}$	0.66
29.81	1,3-Diphenyl propane	$C_{15}H_{16}$	5.63
31.33	1,2,3,4-Tetrahydro-1-phenyl naphthalene	$C_{16}H_{16}$	6.10
31.62	1,2-Diphenyl cyclo propane	$C_{15}H_{14}$	2.36
32.98	1,3-Diphenyl-1-Butene	$C_{16}H_{16}$	3.20
33.30	1,2-Dihydro-1-phenyl naphthalene	$C_{16}H_{14}$	0.78
34.22	1,1-Diphenyl-1,3-Pentadien	$C_{17}H_{16}$	0.90
35.31	1,5-Diphenyl-1-Penten	$C_{17}H_{18}$	0.66
36.50	2-phenyl naphthalene	$C_{16}H_{12}$	1.17
38.85	1-benzyl naphthalene	$C_{17}H_{14}$	1.06

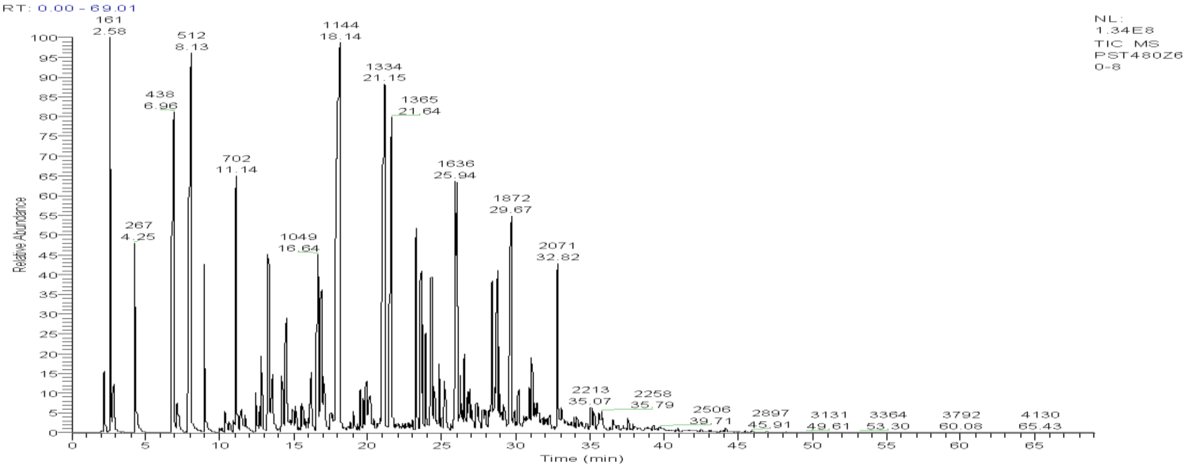

Figure 4. Chromatogram of the liquid product of 1/5 HAW/PS at 480 ºC

To see the effect of the HAW/PS ratio and temperature on liquid composition, a group of the component was selected to compare. Detected compounds were given in Fig. 5. At 420 ºC, it was observed that naphthalene, 2-methyl naphthalene, and 1-methyl naphthalene yield increased with increasing HAW ratio in feed. However, styrene and alpha-methyl styrene yield decreased with this rising. This was an expected situation because naphthalene derivatives and styrene derivatives originated from HAW and PS respectively. It was observed that increasing HAW amount in HAW/PS mixture effect on toluene yield was ignorable. Samely, Shelves et al. determined that adding petroleum waste to the feed did not affect Benzene-Toluene-Xylene yield in the pyrolytic liquid [23]. Maximum ethyl benzene yield was found as 15.88 % in 3/5 HAW/PS ratio at 420 ºC. At 450 ºC, styrene and alpha-methyl styrene peak area in the liquid was also decreased with increasing HAW in the feed. But if it comes to 480 ºC, styrene yield increased while naphthalene yield decreased with HAW adding. Because at high temperatures pyrolysis products of HAW is mainly gaseous compounds like light olefins which don't easily undergo aromatization reactions [23]. This result was commented also on as synergistic interaction between the two feedstock materials. At 1/5 HAW/PS ratio, it was determined that naphthalene, 2-methyl naphthalene, and 1-methyl naphthalene yields increased with increasing temperature (from 420 to 480 ºC). This situation was clarified that polycyclics converted to the aromatics for petroleum residue [24].

Table 2. Compounds in co-pyrolytic liquid of HAW and PS at 480 ºC

Retention time, min	Component	Formula	Peak area, %
2.21	1,3-cyclo pentadien	C_5H_6	0.51
4.25	toluene	C_7H_8	2.17
6.96	Ethyl benzene	C_8H_{10}	6.32
8.13	styrene	C_8H_8	8.08
8.99	cumene	C_9H_{12}	1.47
11.14	Alpha-methyl styrene	C_9H_{10}	2.84
12.85	indane	C_9H_{10}	0.69
13.30	indene	C_9H_8	2.05
13.53	2-Ethyl-1,4-Dimethyl Benzene	$C_{10}H_{14}$	0.56
16.16	2,5-dimethyl styrene	$C_{10}H_{12}$	0.86
16.64	Methyl indene	$C_{10}H_{10}$	2.48
16.86	1-methyl-4-(1-propenyl) benzene	$C_{10}H_{12}$	2.37
18.14	naphthalene	$C_{10}H_8$	12.71
20.17	1,2-dihydro-6-methyl naphthalene	$C_{11}H_{12}$	0.8
21.15	2-methyl naphthalene	$C_{11}H_{10}$	7.3
21.64	1-methyl naphthalene	$C_{11}H_{10}$	5.31
23.27	biphenyl	$C_{12}H_{10}$	2.08
23.58	2-ethyl naphthalene	$C_{12}H_{12}$	2.18
23.89	1,6-dimethyl naphthalene	$C_{12}H_{12}$	0.87
24.30	1,2-dimethyl napthalene	$C_{12}H_{12}$	2.55
24.76	1,8-dimethyl napthalene	$C_{12}H_{12}$	0.69
25.94	3-methyl 1,1' biphenyl	$C_{13}H_{12}$	4.32
26.51	Isopropyl naphthalene	$C_{13}H_{14}$	0.67
26.84	2,3,6-tirimethyl naphthalene	$C_{13}H_{14}$	0.89
28.39	Fluoren	$C_{13}H_{10}$	1.87
28.75	1,4-dihydro fluoren	$C_{13}H_{12}$	2.96
29.67	1,3-diphenyl propane	$C_{15}H_{16}$	2.63
31.10	2-methyl fluorene	$C_{14}H_{12}$	1.23
32.82	anthracene	$C_{14}H_{10}$	2.28

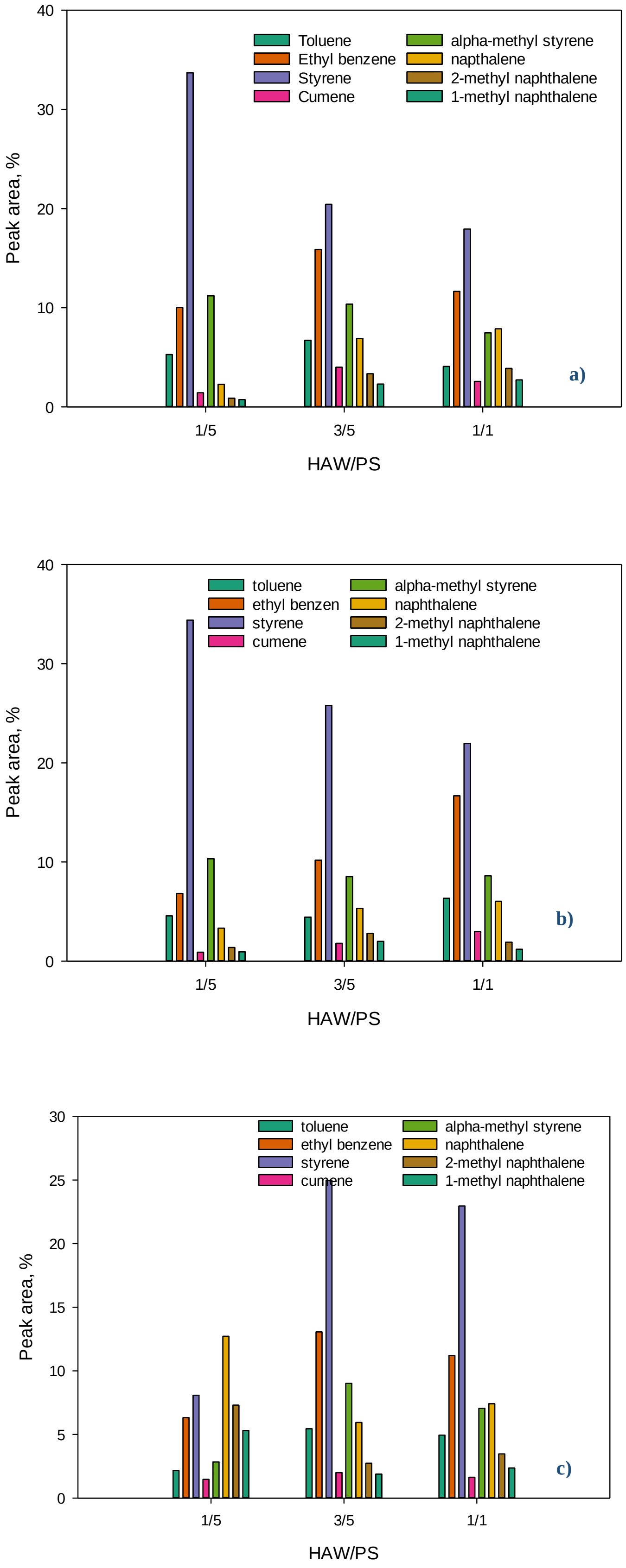

Figure 5. Component distribution of pyrolytic liquid obtained at a) 420, b) 450, c) 480 °C

4. Conclusion

It was performed co-pyrolysis of heavy aromatic waste and polystyrene in a semi-batch glass reactor at 420, 450, 480 ºC with 1/5, 3/5, and 1/1 HAW/PS weight ratios. Product yields were computed and pyrolytic liquids were detected via GC-MS. Maximum liquid product yield was obtained as 87.24 % at 480 ºC for thermal degradation of HAW/PS (1/5). At that temperature liquid yield of pure PS, pyrolysis was found as 87.02 %. Considering that result, it was concluded that liquid yield did not affect HAW adding to the feed. The liquid of HAW/PS (1/5) at 480 ºC is composed of mainly naphthalene derivatives and aromatic hydrocarbons like toluene, ethylbenzene, styrene. The liquid product of polystyrene pyrolysis formed mainly C7-C17 hydrocarbons. However C5-C14 hydrocarbons formed a liquid product of HAW/PS (1/5) at 480 ºC. Naphthalene was not found in PS pyrolytic liquid. However, it was observed at the ratio of 12.71 % in HAW/PS (1/5) pyrolytic liquid at 480 ºC. At 480 ºC, styrene yield increased while naphthalene yield decreased with HAW adding. This result was commented as synergistic interaction between the two feedstock materials. Besides that, because of HAW aromatization reactions in high temperatures can not occur easily. At 1/5 HAW/PS ratio, it was determined that increasing temperature affected positively naphthalene, 2-methyl naphthalene, and 1-methyl naphthalene yields. However, at 1/5 HAW/PS ratio, it was observed that increasing temperature affected negatively toluene, ethylbenzene, and alpha-methyl styrene yields. With that study, two waste materials combined to give a valuable hydrocarbon-rich product that can be a fuel.

5. Contribution of researchers

In this study; M.A.: as the undergraduate thesis student contributed to do pyrolysis study, analyzing the products, discussing the results, and writing the paper. G.Ö.: as the graduate student contributed to do pyrolysis study, analyze the products, discuss the results and write the paper. A.K.: the supervisor proposed the idea, developed the methodology, and contributed to doing pyrolysis study, analyzing the products, discussing the results, and writing the paper.

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