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Chemical Recycling of Household Polymeric Wastes

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<http://dx.doi.org/10.5772/65667>

Abstract

This chapter provides insights on the disposal of household polymeric wastes and chemical recycling of household polymeric wastes for chemical feedstock. Waste generated may cause environmental, economic and health problems. In 2012, the EU (European Union) generated 2514 million tons of waste, of which 213,410 million tons is household waste. Household waste has lots of polymeric materials. The two most important of the polymers are polystyrene (PS) and low-density polyethylene (LDPE). In this study, the results of PS and LDPE obtained from various processes related to polymeric wastes' chemical recycling were given. Main products of PS chemical recycling were obtained as follows: styrene monomer, toluene, ethylbenzene, α -methyl styrene and other valuable chemicals. When LDPE undergoes thermal degradation in a solvent setting in autoclave, oil like diesel can be obtained.

Keywords: chemical recycling, plastic wastes, pyrolysis, thermal degradation, polystyrene, LDPE

1. Introduction

The development of technology has led to an increased welfare level throughout the world and also increased consumption per capita. As a result of this consumption, waste amount has increased gradually. The efficient management of wastes generated, the efficient usage of energy sources and creating new energy source are important aspects for the abatement of environmental pollution and health. The European Union (EU) is one of the most important waste generating regions worldwide. The household waste amount in the EU waste is 213,410 million tons [1]. Composition of these wastes is given in **Table 1**.

Municipal solid waste	Composition, wt%
Kitchen waste	25
Paper and board	18
Plastic (polymeric materials)	12
Other combustible products	10
Garden wastes	6
Rubble	5
Glass	5
Textiles	4
Nappies and other sanitary	3
Steel	2
White goods	1
Aluminum	1
Others	8

Table 1. Municipal solid waste composition in EU [1, 2].

Table 1 shows that plastic wastes (polymeric wastes) is the third predominant consisting of 12% in household wastes. Plastic wastes in household waste comprise lots of polymeric materials. In the EU-27, 6.9% polyethylene terephthalate (PET), 12.1% high-density polyethylene (HDPE), 10.4% polyvinyl chloride (PVC), 17.5% low-density polyethylene (LDPE), 18.9% polypropylene (PP), 7.4% polyurethane (PUR) and 19.7% other polymers are used in the generation of plastic material [1, 2]. Although they differ in polymer, other materials are added mostly in plastic materials as they (polymeric materials) are generated from polymers. The amount of polymer from which plastic material would be generated without adding any material is restricted. Each additive used in the production of plastics has got different purposes except for the ones, such as calcium carbonate, carbon black and caolin, which are used to provide economical production of polymers. These additives are materials such as plasticizers (phthalates), antioxidants, antistatic agents (tertiary amine derivatives, ethylene oxide/propylene oxide copolymers, glycerol derivatives and ethoxylated amines), UV stabilizer (thioethers, hindered amine light stabilizer), antimicrobial agent (10,10-oxybisphenoxarsin, copper-bis-(8-hydroxyquinoline), trichloromethyl thiophthalimides), colorants (azo dye, anthraquinone dyes, quinopthalone, benzodifuranones) and blowing agents (chlorofluorocarbons, hydrochlorofluorocarbons, methylene chloride, pentane, cyclopentane and isopentane, azodicarbonamide, azoisobutyronitrile, hydrazine derivatives). Even if these materials are insignificant in plastic generated, it is known that they have toxic, carcinogenic and endocrine deforming chemicals that are harmful to human health [3]. Moreover, it is possible for polymeric wastes in household wastes to cause pollution. The usage of plastic and the disposal of waste following acceptable environmentally sound standards are important [4, 5].

2. Disposal of household polymeric wastes

The disposal of household polymeric wastes differs from one country to the other and also it is not generally possible to collect and eliminate them. In 2013, nearly 25 million tons of polymeric wastes were collected and processed in EU-27+Norway and Switzerland [1, 2]. Plastic wastes (polymeric materials) are utilized using three methods: recycling, landfill and energy recovery. 26% of these wastes were regained by recycling, and energy was obtained from 36% by energy recovery and 38% of eliminated by landfill [2].

2.1. Landfill

In this process, household wastes are neatly stored underground. Landfill process has several disadvantages such as wastes have great volumes, decrease of the areas in which the wastes are stored, high cost and contamination of water and soil. Due to these reasons, the process of landfill should be preferred in a situation when the process of chemical recycling cannot be performed [4, 5].

2.2. Energy recovery

In this process, wastes are burned and their energy is used. Even if the disposal of wastes through burning process has advantages in terms of obtaining energy, it has disadvantages because of toxic, carcinogenic and endocrine deforming components generated during this process [5–7]. Moreover, facilities using burning process are not cost-effective and environmentally friendly [8].

2.3. Recycling

The main goal is to make plastics (polymers) using mechanical processes (mechanic recycling) or to produce various chemicals or valuable raw materials (chemical recycling) from household polymeric wastes. Even if mechanic recycling has economic advantages, it has been decreased in the molecular weight of polymer and lost of mechanical and physical properties. Therefore, qualified products cannot be manufactured. In other words, there is difference between the material being obtained from the used plastic and the product being obtained from original polymer during mechanical recycling and also it is not processed after definite cycling. In this case, the main solution to eliminate household polymeric wastes is chemical recycling. Although chemical recycling methods may seem economically disadvantaged compared to other methods, in terms of bringing a final solution to waste disposal is important.

3. Chemical recycling of household polymeric wastes

Household polymeric wastes mainly include materials which are made of PET, HDPE, PVC, LDPE, PP, polystyrene (PS) and PUR. The two most important of these polymers are PS and LDPE. PS is used for producing disposable plastic cutlery and dinnerware, CD, smoke detector,

license plate frames, plastic model assembly kits, Petri dishes, test tubes, microplates and many other objects. LDPE is used for production of plastic bags, containers, dispensing bottles, wash bottles, tubing, plastic bags for computer components and various molded laboratory.

In recent years, research has been done concerning the management of household plastic wastes by chemical recycling [9–12]. Thermal decomposition (pyrolysis and thermal degradation) gasification, hydrolysis methanolysis and glycolysis by the type of household polymeric material are the different methods applied in chemical recycling. Thermal decomposition was applied into great part of polymers. During the thermal decomposition, reactive radicals occur in the body of plastic as a result of bond rupture or chain fracturing, and these radicals reveal gas, liquid and solid products as reacting in serial. This method is applied to polymers as pyrolysis and thermal degradation. Pyrolysis is a chemical recycling method. It can be described as degrading a polymeric waste under nonoxygen media (inert, reductive/oxidative etc.) by heat effect. Necessary thermal amount in pyrolysis should be at a level to degrade the chemical structure of polymeric material and to provide the formation of new chemical materials. Polymeric material is fully transformed to solid, liquid and gas products without getting any additives by pyrolysis. The process does not cause air, water and soil pollution. All of the obtained products can be used as raw materials in other industries. There are many kinds of pyrolysis such as batch, semibatch, vacuum, catalytic pyrolysis, etc. Important pyrolysis parameters are heating rate, temperature, medium and time. The medium may be catalytic, inert, oxidative or reductive. According to heating rate, pyrolysis can be classified as slow pyrolysis and flash pyrolysis. The most definite feature distinguishing flash pyrolysis from slow pyrolysis is the stay-duration of products in pyrolysis setting occurring in the splitting and heating rate. Heating rate is in the range of 1–10°C/min in slow pyrolysis and it is higher than 100°C/hour in flash pyrolysis. Thermal degradation is the deformation process of polymeric materials as thermal in a solvent setting at respectively lower temperature as pyrolysis.

In this chapter, chemical recycling of PS and LDPE wastes was investigated. The results of the experiment of PS and LDPE obtained from various processes related to polymeric wastes' chemical recycling are discussed in the following sections. These experiments demonstrated that valuable chemicals and feedstock can be produced from household polymeric wastes with chemical recycling.

3.1. Pyrolysis of polystyrene waste in batch reactor under inert medium

Polystyrene can be degraded to the monomer and other valuable chemicals by slow pyrolysis. Schematic representation of the system, including polystyrene plastic wastes' chemical recycling by slow pyrolysis is shown in **Figure 1**. The system comprises of Pyrex glass reactor, PID controlling oven to heat reactor (1.5 kW), condenser and liquid product gathering container (phase median). Reactor is in diameter of 4.2 cm and at the length of 40 cm. Internal and external temperatures of reactor were measured by Ni-Cr-Ni thermocouple. Plastic waste was located in the bottom of the tube part in a reactor. Reactor was vertically located in high-temperature ceramic oven. Pyrolysis was done in nitrogen atmosphere.

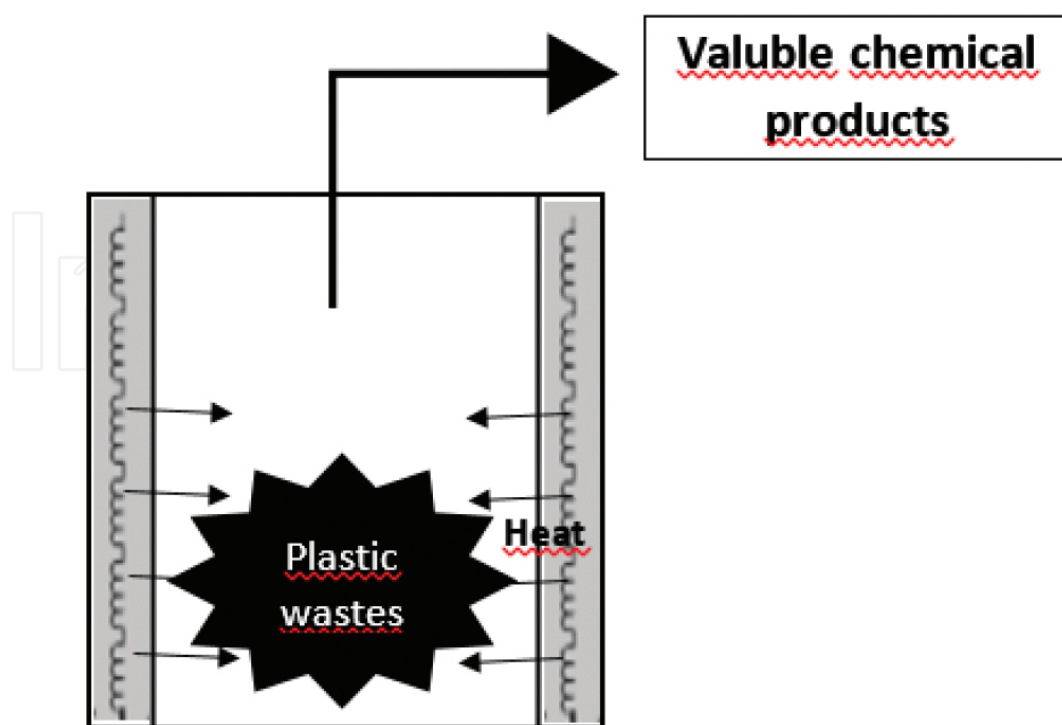


Figure 1. Chemical recycling of plastic wastes via slow pyrolysis.

Liquid products of PS pyrolysis were obtained in a condenser located out of reactor and from liquid-receiver plug vessels (phase separator) having salt-ice mixture in its cover added to its output. Moreover, total conversion was calculated by the amount of liquid and gas products. The liquid products were analyzed to illuminate the content of liquid products by Thermo Finnigan DSQ-250 gas chromatography-mass spectrometry (GC-MS). Rtx-5MS capillary column at a length of 30 m and 0.25 mm in internal diameter was used as a column. The analysis terms of GC-MS: injection temperature is 225°C, temperature of transfer line is 300°C and helium flow rate is 0.7 ml/min. Temperature program being applied to column was heated to 300°C at the heating rate of 5°C min⁻¹ after 5-min stand-by at 50°C and it was waited for 20 min at this temperature (300°C).

For chemical recycling of PS polymeric wastes, PS wastes were pyrolyzed using this experiment system. The yields were investigated in the experiment at 450°C. Those values were respectively 59.46% liquid, 2.29% solid, 43.46% gas + loss and 92.92% total conversion. According to the results, the majority of the polymer was converted into liquid and gas chemicals. GC-MS chromatogram of the liquid products obtained from chemical recycling of PS polymeric waste is given in **Figure 2**.

Compounds in this chromatogram were defined by using Wiley library. The list of chemical materials defined is given in **Table 2**.

As depicted in **Table 2**, carbon number ranges of liquid products were found as % 68.8 C₆-C₉, % 4.36 C₁₃-C₁₅ and % 26.8 C₁₆-C₁₈.

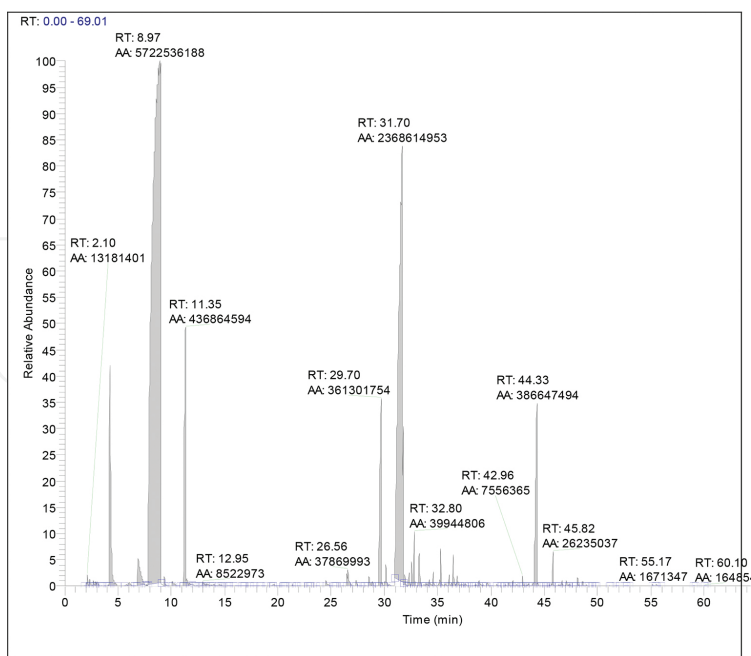


Figure 2. GC-MS chromatogram of pyrolysis liquid products obtained from chemical recycling of PS.

Residence time, min	wt%	Compound
2.34	0.08	1-Hexane
2.65	0.07	Bütane-2,3-dimethyl
3.07	0.12	Hexane-3-methyl
4.23	3.62	Benzene methyl
6.03	0.1	1-Heptene-5-methyl
6.9	0.97	Ethylbenzene
8.97	55.52	Styrene
11.35	4.24	Alpha-methylstyrene
24.51	0.1	Diphenylmethane
26.56	0.37	Bibenzyl
28.55	0.12	1,2-Diphenyl cyclopropane
29.7	0.12	Benzene-1,1'(1,3-propanedyl)bis-
31.7	22.98	Naphthalene 1,2,3,4-tetrahydro-2-phenyl
31.91	0.09	Benzene-1,1(1,4-butanedyl)bis
32.34	0.1	Benzene-1,1(1,4-butanedyldiene)bis
32.56	0.16	1,3-Pentadiene-1,1-diphenyl
32.8	0.39	Benzene, 1,1'-(2-pentene-1,5-diyl)bis-
33.29	0.43	3,5-Diphenyl-1-pentene
34.21	0.09	Benzene-1,1(1-methyl-2-butyldiene)bis
34.58	0.1	2,5-Diphenyl-1,5-hexadiene
35.28	0.35	Benzene-1,1(2-pentene-1,5-dyl-bis
36.07	0.09	1,3-Pentadiene-1,1-diphenyl
36.46	0.3	1-Pentadiene-1,5-diphenyl
36.85	0.11	1,5-Diphenyl-1,5-hexadiene
38.87	0.08	5(2-Propylvinyl)dibenzocycloheptane
42.96	0.07	1-Ethyl-2-methyl-3-phenylindane

Table 2. Compounds of PS pyrolysis liquid products at 450°C.

3.2. Thermal degradation of LDPE in autoclave with solvent

Thermal degradation is a thermal deformation of polymeric wastes in a solvent setting. In this study, LDPE polymeric wastes were put to thermal degradation with vacuum gas oil (VGO) as a solvent supplied from Kirkuk refinery (Iraq). The schema of experiment system where chemical recycling has been done by thermal degradation is given in **Figure 3**. Experiments were done in a stainless steel reactor called autoclave being durable against high pressure and temperature, gastight, having 250 ml volumes, having pressure and temperature indicator and being screw-capped one. Heating reactor was done by a PID controlling high-temperature oven. Experiments were carried out under nitrogen atmosphere.

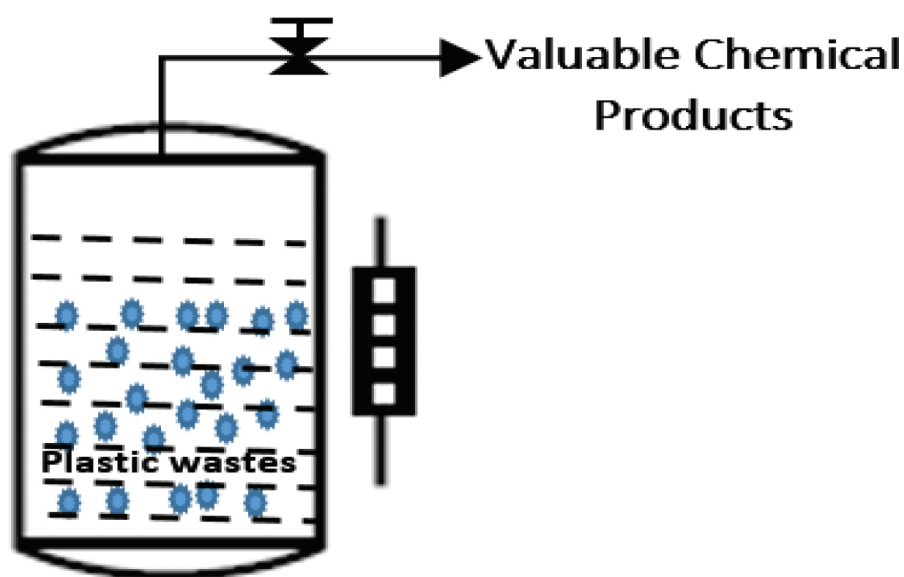


Figure 3. Chemical recycling of plastic wastes via thermal degradation.

At the end of the experiments, autoclave was cooled to room temperature and gas product obtained was taken. Then, the content amount in the reactor was determined as being separated into liquid and solid. The amount of gas product was found from variation in the beginning of process. On thermal degradation, experiments being done as getting LDPE/VGO: 1/1, 85.94% liquid, 5.76% solid, 8.3% gas and 94.24% total transformation (liquid + gas) were found.

The content of liquid products for thermal degradation has been illuminated by GC-MS device which is given above. However, the column temperature program has been set at 50°C at the beginning and after 2-min duration, it has been set at 270°C at the heating rate of 5°C/min and it was waited for 20 min at this temperature. GC-MS chromatogram of the liquid products is given in **Figure 4**. As it can be seen in **Figure 4**, each pick of the liquid products has tripled. One of them is alkane (saturated hydrocarbon), another is alkene (unsaturated hydrocarbon) and the other is diene (hydrocarbon that contains two carbon double bonds).

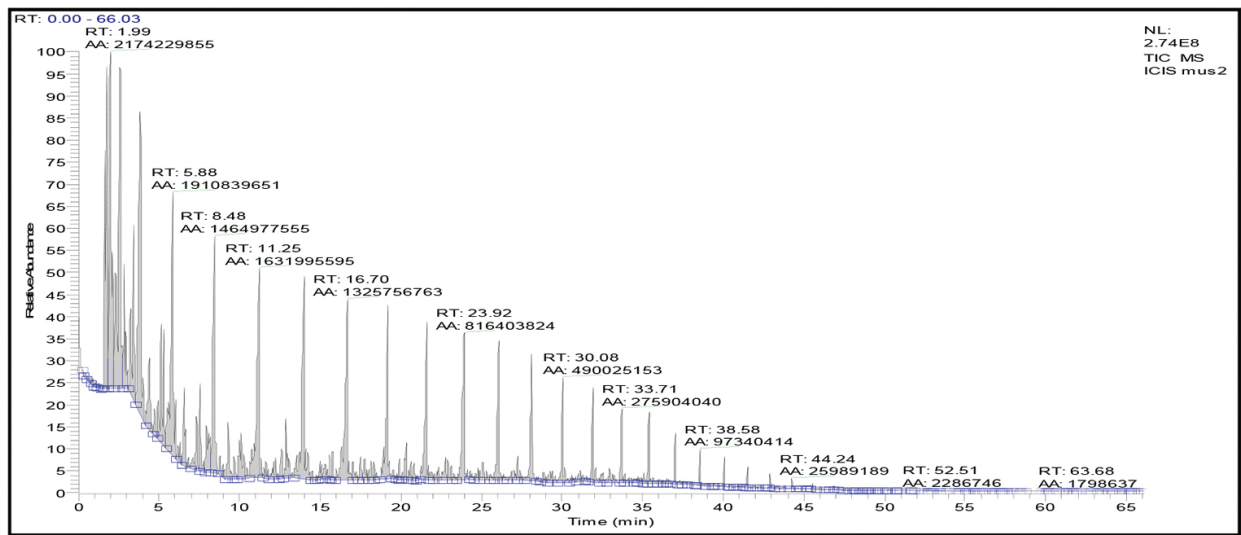


Figure 4. GC-MS chromatogram of LDPE/VGO:1/1 and at 400°C.

Thermal degradation liquid products obtained from Autoclave were analyzed by using GC-MS. The list of chemicals is given in **Table 3**.

Residence time, min	wt%	Compound
1.76	5.67	Pentane
1.99	7.46	Hexane
2.58	8.47	Heptane
2.83	1.87	Methylcyclohexane
3.44	3.51	Toluene
3.84	6.55	Octane
4.43	1.71	Ethylcyclohexane
4.7	0.48	Unknown
5.14	3.46	Ethylbenzene
5.88	6.56	Nonane
6.31	0.35	2-Methyl-bicyclo-octane C9
6.58	1.24	7,7-Dimethyl-tetracycloheptane
7.34	0.98	n-Propylbenzene
7.57	1.11	1,3,5-Trimethylbenzene
7.96	1.31	Cyclodecane
8.48	5.03	Decane
8.79	0.50	Cyclodecane C10
9.27	0.84	Cyclodecane C10
10.09	1.44	Undecane C11
11.25	5.60	Undecane
11.65	0.36	Unknown
12.24	0.51	1-Dodecene C12
12.57	0.54	1-Cyclopropyl-1-methyl-benzene C12
12.87	1.06	1-Cyclopropyl-1-methyl-benzene C12

Residence time, min	wt%	Compound
14.04	5.36	Dodecane
15.28	0.46	Dodecane
16.7	4.55	Tridecane
17.04	0.38	Spiro(tricycloundeca-2,4,6-triene),7,1-cyclopropane
17.77	0.43	7-Tetradecene
19.24	3.54	Tetradecane
20.34	0.64	Unknown
21.63	3.10	Pentadecane
22.79	0.71	Unknown
23.92	2.80	Hexadecane
25.15	0.49	Hexadecane
26.07	2.16	Heptadecane
27.01	0.34	2,6,10-Trimethyl tetradecane
27.24	0.33	Unknown
28.12	1.88	Octadecane
29.24	0.41	Unknown
30.08	1.68	Nonadecane
31.93	1.25	Eicosane
32.95	0.41	Docosane C22
33.71	0.95	Docosane
35.39	0.73	Tricosane
37.03	0.47	Pentacosane
38.58	0.33	Pentacosane

Table 3. Compounds of LDPE/VGO:1/1 thermal degradation Liquid products at 400°C.

Liquid products of LDPE/VGO:1/1 at 400°C were examined to evaluate the cetane number of fuel specifications. Cetane number of liquid products from thermal degradation has been found to be nearly 50. This value can be an initiative indicator for this product as it would be used for diesel oil. Consequently, when LDPE is put to thermal degradation in a solvent setting in autoclave, an oil like diesel can be obtained.

3.3. Flash pyrolysis of household polymeric wastes in free fall reactor (FFR)

Flash pyrolysis, one of the chemical recycling methods of household polymeric wastes, can be used with various reactors such as fluidized bed and rotating cone. One of these reactors is FFR. Valuable chemicals can be obtained by flash pyrolysis of household polymeric wastes in FFR. The schema of flash pyrolysis system with FFR is given in **Figure 5**. Polymeric wastes are made as a granule at a definite size in this system and it is fed suddenly to high-temperature heated reactor by a solid feeding system.

Reactor of FFR experimental system using chemical recycling of PS and LDPE household polymeric wastes is 140 cm in length and in 5 cm diameter quartz. It is heated to pyrolysis temperature with a high-temperature oven of 120 cm length and 10 cm internal diameter. Reactor was fed at 2 g/min feeding rate with a polymeric waste feeding system. There is tar receiver under reactor, serial condenser connected on outlet of tar receivers (which has got salt-

ice mixture in its cell) and vacuum pump getting all system under vacuum. Temperature measurements were done in the inner and outer parts of the reactor.

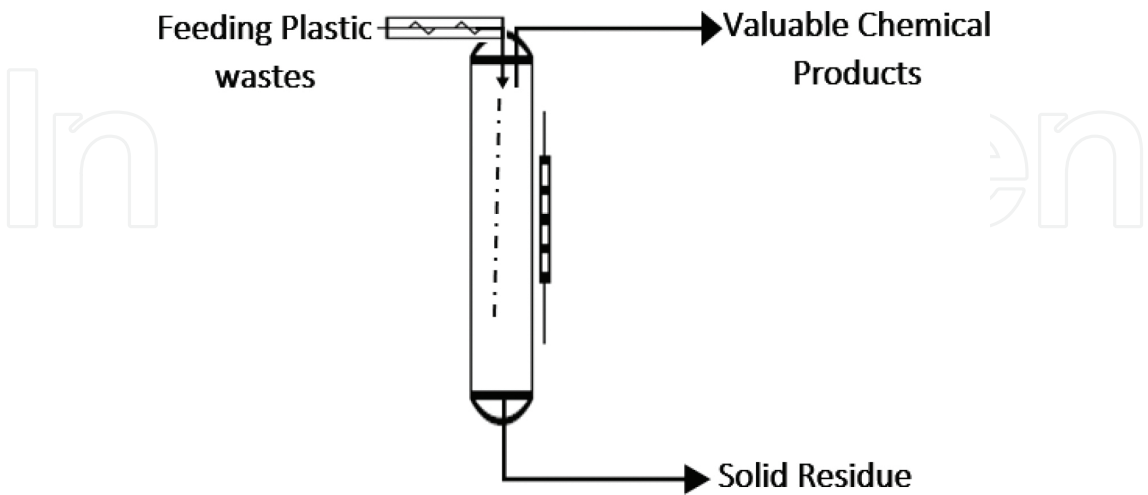


Figure 5. Flash pyrolysis of polymeric wastes in FFR.

Particles of PS and LDPE polymeric wastes at size range of 150–75 micro particle were used in PS and LDPE’s flash pyrolysis in FFR. 825°C flash pyrolysis liquid products were analyzed in GC-MS in conditions of the above given PS and LDPE liquid product analysis both for PS and LDPE in FFR.

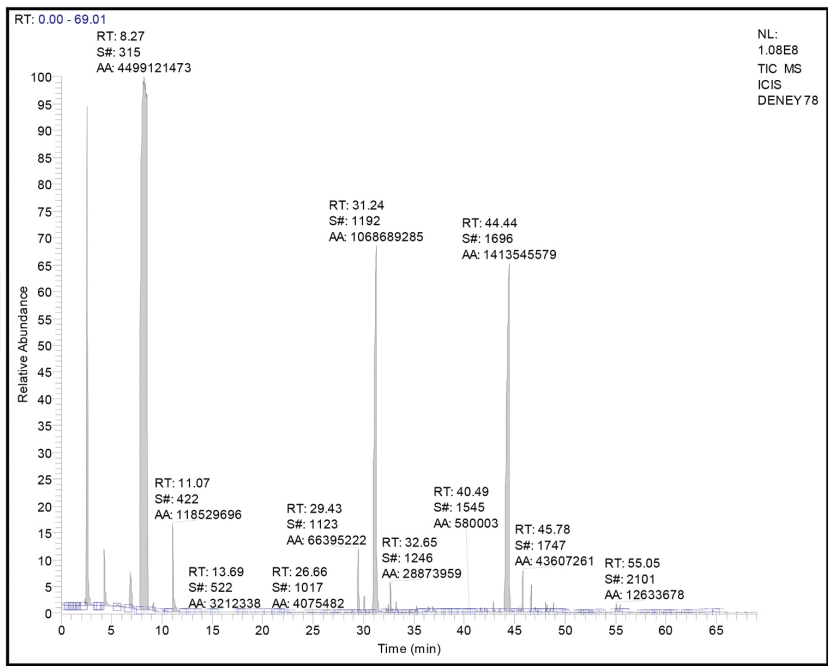


Figure 6. GC-MS chromatogram of PS flash pyrolysis liquid.

GC-MS chromatogram of liquid products from 825°C flash pyrolysis of household polystyrene polymeric wastes is given in **Figure 6**. Moreover, the description study of chemical compounds in liquid products was done and the obtained results are given in **Table 4**.

Residence time, min	wt%	Compound
4.26	1.29	Toluene
6.85	1.05	Ethylbenzene
8.27	53.60	Styrene
11.07	1.41	α -methylstyrene
29.43	0.79	1,3-Diphenyl propane
31.24	12.73	1,1-Diphenyl-2-methyl propane
32.65	0.34	1,3-Diphenyl-butane
33.23	0.21	1,1-Diphenyl-2-methyl propane
42.87	0.14	Dimer
44.44	16.84	Trimer (2,4,6-triphenyl-1-hexane)
45.78	0.52	Trimer
	11.60	Others

Table 4. Compounds of PS flash pyrolysis liquid products in FFR at 825°C.

Table 4 depicts that the main product is styrene monomer. Flash pyrolysis of polystyrene in FFR showed that it can obtain important liquid chemicals such as toluene, ethylbenzene, α -methyl styrene and others besides styrene monomer.

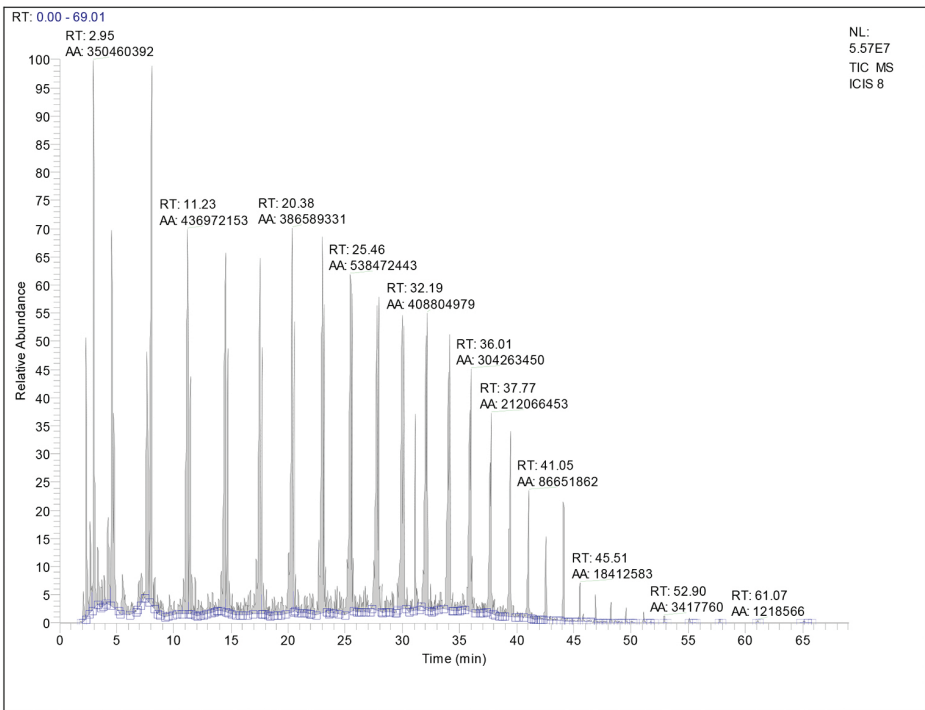


Figure 7. GC-MS chromatogram of liquid products for LDPE wastes in FFR at 825°C.

GC-MS chromatogram of liquid products for LDPE polymeric wastes in FFR from 825°C is given in **Figure 7**. As it can be seen from **Figure 7**, peaks in chromatogram were given respectively as homologues. This indicates that products have got aliphatic character. LDPE pyrolysis liquid products were described by using GC-MS library.

The products obtained with the use of GC-MS are given in **Table 5**. As it can be seen in **Table 5**, the liquid product which obtained LDPE is paraffinic hydrocarbons. Liquid products obtained from chemical recycling of LDPE polymeric wastes at 825°C in FFR were divided into three groups. The percentage of groups, The percentages of the groups are % 30.44 C₆-C₉, % 42.83 C₁₀-C₁₃ and % 26.73 C₁₄-C₁₈.

Consequently, gathering, dissection and innovator chemical recycling processes are important for the method of HHW plastic wastes. Gathering and dissection processes for polymeric wastes in HHW have direct effect on chemical recycling. Thus, polymeric wastes should be collected separately from other wastes without being contaminated and should be distinguished from each other. Each of separated polymeric waste should be put to chemical recycling by a proper process. An innovator chemical recycling process should create a harmful waste for environment, health and economy. Recycling of polymeric wastes is advantageous with pyrolysis. Pyrolysis of polymeric waste is carried out in a closed area, where gas, liquid and solid products are obtained. An innovator pyrolysis process should include units making these products usable as raw materials. The following situation is suitable: to gather polymeric wastes in HHW, to dissect them by polymer and their chemical recycling method by polymer. Pyrolysis is suggested for the recycling of PS polymeric wastes and thermal degradation is suggested for LDPE.

Residence time, min	wt%	Compound
2.37	2.06	1-Hexane
2.74	0.28	1,3-Pentadiene-2-methyl
3.03	4.15	1-Heptene
3.42	0.77	Cyclohexane-1-methyl
3.99	0.24	Cyclopentane-1-methyl
4.28	1.06	Cyclohexane-1-ethyl
4.91	6.83	1-Octane
5.6	0.79	Cyclohexane-1,2-dimethyl
6.03	0.38	1-Hexadiene-2,5-dimethyl
6.95	0.68	Cyclohexane-1-ethyl
8.18	10.12	Nonane
8.98	0.4	Cyclohexane(1-methylethyldiene)
9.65	0.27	Cyclopentene-1-butyl
11.33	5.49	1-Decane
11.57	3.48	2-Decene
11.69	0.33	Cyclohexene-1-butyl
11.96	0.32	2-Decyne
12.46	0.34	Bicyclo(3,1,1)heptane-2,6,6-trimethyl
14.63	4.88	Undecane

Residence time, min	wt%	Compound
14.87	3.86	Undecyne
16.73	0.51	1,3-Di(1-propyl) cyclopentane
17.67	4.76	Dodecane
17.91	4.23	3-Dodecyne
18.17	0.38	3-Dodecyne
18.83	0.37	1,1,2-Tridecadiene
19.51	0.36	4-Tridecene
20.43	4.83	4-Tridecene
20.71	4.13	Tridecane
20.95	0.39	1,1,2-Tridecadiene
21.73	0.37	4-Nonene-5-butyl
22.2	0.4	1,1,2-Tridecadiene
23.04	5	1-Tetradecene
23.31	4.09	Tetradecane
24.54	0.37	5-Tetradecene
25.74	7.61	Pentadecene
27.95	4.76	Pentadecene
30.02	2.01	Hexadecane
32.01	0.77	1-Octadecene

Table 5. Compounds of LDPE flash pyrolysis liquid products in FFR at 825°C.

4. Conclusion and assessment

Pyrolysis of PS, thermal degradation of LDPE and flash pyrolysis of PS and LDPE are chemical recycling methods of household polymeric wastes. It is possible to obtain valuable chemicals and oil from these wastes by chemical recycling. Results and evaluations from the experimental results are given below.

Most of the liquid products from slow pyrolysis of PS polymeric wastes constitute styrene being its own monomer. Moreover, valuable chemicals such as toluene, ethylbenzene and methylene styrene have been produced. It is possible to obtain valuable chemicals via chemical recycling of PS wastes without damaging environment and people's health. In other words, successfully designed chemical recycling processes are not harmful for environment and people's health.

Products from heated degradation of LDPE household polymeric wastes in autoclave in a solvent setting are hydrocarbons in the aliphatic structure. Liquid product obtained from the experiments using VGO is oil like diesel. It is seen that condensable liquid products are obtained when this polymer's flash pyrolysis in FFR is made. It is thought that alpha-olefins, lubricating oil, alcohols, surface chemicals, carboxylic nitrogen and similar valuable products would be obtained with the evaluation of LDPE household wastes.

Products in reactor in flash pyrolysis occurring under the vacuum in FFR are excluded quickly from reaction region. Polymer falling from upper part to bottom in reaction setting goes beyond

as having degradation. In this case, low-molecule-weighted chemicals occur as thermal splitting occurs in macromolecules with the effect of temperature in polymeric waste. Degradation in molecule changes depending on temperature and size of particle. Parameters such as temperature, particle size, feeding rate and reactor size (standby duration) should be considered in chemical recycling in such reactors.

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