AJEMA GLOBAL RESEARCH NETWORK

AMERICAN Journal of Engineering, Mechanics and Architecture
Volume 2, Issue 2, 2024 ISSN (E): 2993-2637

Synthesis of Antioxidant Stabilizers AO-1, Their Application to Polyethylenes, IR Spectral and Thermogravimetric Analysis of Stabilized Polyethylenes

Karaev Sherali
An Engineer, Applicant for Shurtan Gas-Chemical Complex, Republic of Uzbekistan, Guzar

Djalilov Abdulakhat
Academician, Doctor of Chemical Sciences, Director of Tashkent Research Institute of Chemical Technology LLC, Uzbekistan, Tashkent region, Tashkent district

Karimov Masud
Professor, Doctor of Technical Sciences, Deputy Director, Uzbekistan, Tashkent region, Tashkent district

Abstract: The synthesis of the antioxidant stabilizer AO-1 was carried out in three stages. Medium-density polyethylene products were mixed with PY-342 using the CLAWSON method and processed in a Jinan Himax LSB extruder to determine the properties of the synthesized antioxidant stabilizer for stabilizing polyethylene products. In the IR spectrum of stabilized polyethylenes, asymmetric and symmetric stretching vibrations of the methyl (-CH3) and methylene (-CH2) groups were obtained in the region of 2848-2914 cm-1 of the spectrum and the presence of phenol homologues was determined in the region of 1700-2000 cm-1. It was found that low-intensity lines are characteristic of rotational and pendulum vibrations of the methylene group and are located in the region of 1305 and 717 cm-1, and stabilized polyethylene was analyzed using thermogravimetric analysis. The decomposition of polyethylene occurs at a temperature of 120°C, and the decomposition of polyethylene with the addition of the antioxidant stabilizer AO-1 occurred at a temperature of 380.57°C. In this case, a mass loss of 0.053 mg or 1.897% occurs, which indicates an improvement in the physicochemical properties of polyethylene.

Keywords: IR spectrophotometer, antioxidant stabilizer, etherification, pentaerythritol, terephthalic acid, polyethylene, pendulum vibration.

Introduction. Currently, in the world, the development of antioxidant stabilizers with high stabilization efficiency and their introduction into polymer products is one of the main solutions to problems in this area. Especially today, the economic development of industry and production is inextricably linked with the long-term operation of polymer products. However, there are a number of requirements for creating a new generation of stabilizing antioxidants, such as: the synthesis of little-used, low-concentrated, environmentally friendly and economically inexpensive stabilizing antioxidants.

It has been established that the performance characteristics of polymer products are formed under the influence of external factors (oxygen, ozone, light, radiation, etc.) [1, 2; pp. 272-307, 3; pp. 967-985, 4; pp. 57-65, 5; 227 pp., 6; pp. 41-48]. This occurs due to processes of

degradation of the polymer structure, which are usually called “aging”. One of the ways to prevent wear is to add special additives to the composition of polymers and polymeric materials - stabilizers to prevent processes. Based on the nature of their action, polymers and stabilizers of polymeric materials are divided into six groups. This division is conditional; some compounds are able to protect the polymer from several types of wear [7; pp. 27-31, 8; 1129-1155 b.].

Main part. Synthesis method for obtaining antioxidant (AO-1) using pentaerythritol (2,2-bis(hydroxymethyl)propane-1,3-diol) C(CH2OH)4 by esterification of a polyethylene product.

1. Pentaerythritol 2 mol

2. DMSO 100 mg

3. Terephthalic acid 2 mol

4. H_2SO_4 0.5 g (catalyst)

The reaction process takes place in a 3-neck flask with a volume of 250 ml and in a water bath.

Stage 1. Mix pentaerythritol and terphthalic acid in a 200 ml flask at 50°C, then add DMSO and slowly heat to 65°C for 1 hour.

Stage 2. Transfer the saturated mixture to a 250 ml three-neck flask. The flask was placed in a water bath on a hotplate, a dropper with a catalyst (H2SO4) was attached to the necks of the flask, a thermometer was placed, and the catalyst began to drip very slowly. The temperature in the flask was increased to 150°C, the process was carried out for 4.5 – 5 hours. The mixture in the flask turned light brown.

Stage 3. The synthesized substance was washed from accompanying substances in two ways. First washed with ethyl alcohol and then with water. The substance acquires a transparent white color, it is filtered and dried at room temperature for 6-8 hours until it turns into a homogeneous powder.

The synthesized antioxidant stabilizer was named AO-1. The resulting antioxidant stabilizer was mixed with high, medium and low pressure polyethylene grades PY-342. They were mixed using the CLAWSON method and processed in a Jinan Himax LSB extruder. IR spectral analyzes of the stabilized polyethylenes were then carried out.

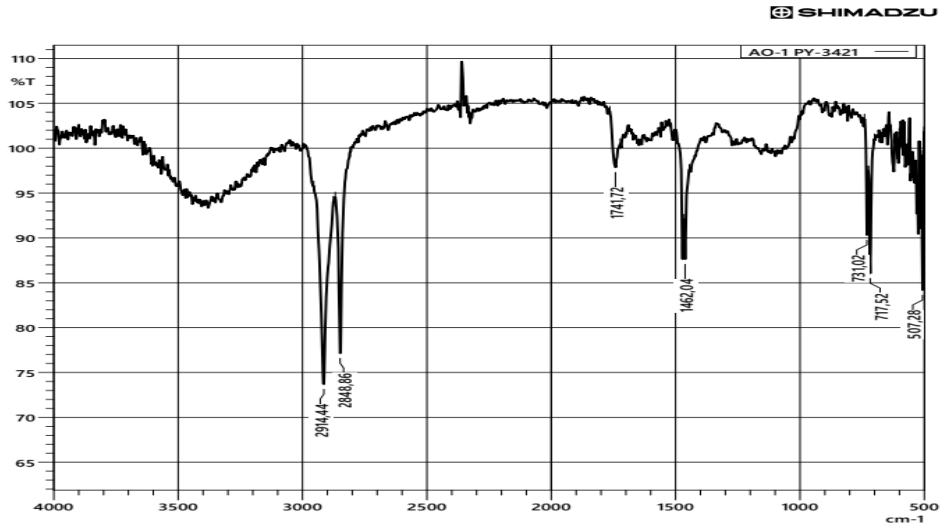

Figure 1. IR spectrum of PY-342 polyethylene stabilized with antioxidant AO-1.

In the spectral region 2848-2914 cm-1 there are bands due to asymmetric and symmetric stretching vibrations of the methyl

(-CH3) and methylene (-CH2) groups. Di-, tri-, tetra-, penta- and hexa-substituted benzene derivatives have been well studied in the literature, and as a result of research, the appearance of

a spectrum in the region of 1700-2000 cm-1 indicates the presence of phenol homologues. The bands caused by asymmetric and symmetric bending vibrations of the methyl group and shear vibrations of the methylene group correspond to the spectral region of 1462 cm-1. Low-intensity bands in the spectral region of 1305 and 717 cm-1 are due to rotational and pendulum vibrations of the methylene group.

A sample of polyethylene PY-342 with the addition of antioxidant stabilizer AO-1 was subjected to thermogravimetric analysis and oxidative degradation was analyzed to determine stabilization.

The derivative diagram (DTA) curve revealed an endothermic effect with two heat absorptions at 134.630C and 472.360C and no exothermic effect with heat release. Thermogravimetry (TGA) curve analysis shows that extensive decomposition occurs in the temperature range of the TGA curve.

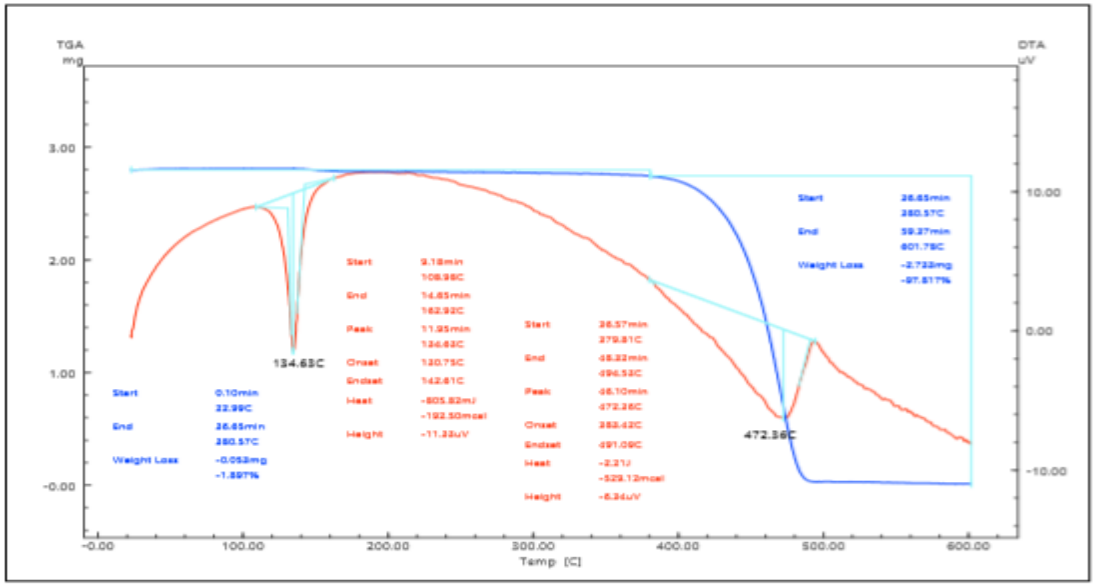

Figure 2. Derivatogram of polyethylene grade PY-342, stabilized with antioxidant AO-1.

The 1st decomposition interval was observed in the temperature range of 22.99-380.57 ° C, 0.053 mg or 1.897% of the mass was lost, the 2nd decomposition interval was observed at a temperature of 380.57-601.78 ° C and 2.733 mg or 97.817% mass. In the temperature range of 14.94 – 601.24°C, the total mass reduction was 2.786 mg or 99.714%, which took 59.37 minutes. The analysis of the thermogravimetric analysis curve and differential thermal analysis curve is shown in Table 1 below.

Based on the results of DTA and TGA analysis, kinetic parameters were determined for various temperature ranges of the process. Its advantage is that the kinetic properties of the reaction over the entire temperature range were calculated from a series of measurements and from one sample.

The degree of mass loss (wm) was determined by the method of graphic differentiation of the TGA curve:

wm=Dm/Dt

here Dm – weight loss, mg; Dt - time interval, min.

The detailed analysis of the thermogravimetric analysis curve and differential thermal analysis curve is given in the table below.

In this TGA analysis, 99.714% of the total mass underwent thermal decomposition up to 600°C. (Table 1).

Table 1. Thermogravimetry (TGA) curve analysis.

Temperature ^{0}C	Time, minutes	Weight (мг)	Mass loss (%)
22,99-380,57	36,55	0,053	1,897
380,57–601,78	22,72	2,733	97,817

The detailed analysis of the thermogravimetric analysis curve and differential thermal analysis curve is given in Table 2 below.

Table 2. The influence of temperature on the weight loss of a sample of PE brand PY-342 stabilized with antioxidant AO-1

№	dw 2.83	1/T	dw/dt	М.г	Минт	T^0+К
1	2.74	0.0026	0.014	0.09	6.42	373
2	2.68	0.0021	0.009	0.15	16.42	473
3	2.45	0.0017	0.014	0.38	26.42	573
4	1.32	0.0014	0.041	1.51	36.42	673
5	0.97	0.0012	0.040	1.86	46.42	773
6	0.06	0.0011	0.046	2.77	59.3	874.78

The activation energy values for this process are given for the results of oxidative destruction of the PY-342 PE sample using SA grade AO-1 (Table 3).

Table 3. Results of thermal-oxidative analysis of a sample of PE brand PY-342 with the addition of antioxidant AO-1.

№	dw 2.83	$Ln(W_1/W_2)$	$1/T *10^{-3}$
1	2.74	0.032	2.6
2	2.68	0.054	2.1
3	2.45	0.144	1.7
4	1.32	0.762	1.4
5	0.97	1.070	1.2
6	0.06	3.853	1.1

Thus, based on the experimental data obtained on the kinetics of processes in the temperature range from 287.94 to 874.24 K, the features of thermal oxidative destruction of a sample of PY-342 polyethylene stabilized with the antioxidant AO-1 were studied.

Conclusion

Based on the experiments conducted, we can conclude that the synthesized antioxidant stabilizer AO-1 has proven itself well when mixed with medium-density polyethylene PY-342. IR spectral analysis of polyethylenes stabilized with the antioxidant AO-1 showed the presence of methyl (-CH_3), methylene (-CH_2) groups and phenol homologues. In thermogravimetric analysis, the decomposition of polyethylene occurs at a temperature of 120°C, and the decomposition of polyethylene with the addition of the antioxidant stabilizer AO-1 occurred at a temperature of 380.57°C. In this case, a mass loss of 0.053 mg or 1.897% occurs, which indicates an improvement in the physicochemical properties of polyethylene.

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