Study of compounds emitted during thermo-oxidative decomposition of polyester fabrics

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Compounds emitted during thermo-oxidative decomposition of three commercial polyester fabrics for indoor outfit and decorations (upholstery, curtains) were studied. The experiments were carried out in a flow tubular furnace at 600ºC in an air atmosphere. During decomposition process the complex mixtures of volatile and solid compounds were emitted. The main volatile products were carbon oxides, benzene, acetaldehyde, vinyl benzoate and acetophenone. The emitted solid compounds consisted mainly of aromatic carboxylic acids and its derivatives, among which the greatest part took terephthalic acid, monovinyl terephthalate and benzoic acid . The small amounts of polycyclic aromatic hydrocarbons were also emitted. The emission profiles of the tested polyester fabrics were similar. The presence of toxic compounds indicates the possibility of serious hazard for people during fire.

Keywords: polyester fabrics, poly(ethylene terephthalate), thermo-oxidative decomposition of polyesters, combustion of polyesters.

INTRODUCTION

Polyester fabrics are used in the manufacturing of different kinds of clothes including overcoats, jackets, leisure and sportswear, protective and outdoor clothing. They are also widely used in home furnishings like bedspreads, sheets, pillows, furniture, carpets, tablecloths or curtains. This popularity is caused by very desirable properties: resistance to most chemicals, resistance to crease, stretching and shrinking. Polyester fabrics are hydrophobic, quick drying and retain their shape[1, 2].

In textile industry the predominant commercial polyester is poly(ethylene terephthalate) (PET). In some special areas of applications other aromatic polyesters, their different blends or copolymers are used, despite higher prices of raw materials and production costs[2–4]. Poly(butylene terephthalate) (PBT) fibres, due to their superior elasticity, are used for the manufacture of underwear, swimwear and sportswear, but their application to textiles is limited[1, 4]. Poly(trimethylene terephthalate) (PTT), which was developed more recently, has excellent dyeability, elastic recovery, stain resistance and low static charge generation and is intended to use in upholstery and as a carpet fibre[4–6]. Another aromatic polyester – poly(ethylene 2,6-naphthalate) (PEN) is used for manufacturing of high performance fibers that have better strength, thermal properties and dimensional stability than PET fibers. PEN has found applications in high performance sailcloth materials and in industrial filtration applications. It can also be used to prepare high-strength fibers for the manufacture of tire cord and mooring ropes[7–9].

Because of the wide use of polyesters in everyday life it is important to put attention on their destruction process which may occur at high temperatures, e.g. during fire. Thermo-oxidative decomposition of aromatic polyesters was the subject of many studies, including influence of flame retardant additives[10–12]. Thermal and thermo-oxidative decomposition of fabrics made of polyesters was the subject of kinetic studies[13], emissions of compounds evolved from used polyester fabrics (black

shorts) at different temperatures between 650–1050ºC were also determined[14]. Some studies in relation to the polyester-cotton blended fabric[15, 16] and influence of the flame retardants[16, 17] were also performed. Moreover, the flammability and burning behaviour of different textile fabrics were studied[18, 19]. The results of other authors show that during decomposition of polyester fabrics at high temperatures complex mixtures of different toxic compounds, including carbon monoxide, aromatic hydrocarbons and polycyclic aromatic hydrocarbons were formed[14, 17]. In present work the investigations results of compounds emitted during thermo-oxidative decomposition of commercial polyester fabrics for indoor outfit and decorations are presented.

EXPERIMENTAL

Material

Three samples of commercial polyester fabrics for indoor outfit and decorations were obtained from local Polish market. All materials were described by the producers as "100% polyester". There was no other information available, e.g. the kind of polyester or the presence of the flame retardants. The characteristics of tested fabrics are given in Table 1.

All analytical standards were of analytical-grade from various suppliers: Aldrich, Supelco, Fluka. All solvents: methyl alcohol, acetone and butyl acetate were also analytical-grade and purchased from Chempur. Active carbon (35–50 mesh) from Merck was used for the adsorption of analytes during preparation samples for the GC-MS analyses.

Thermo-oxidative decomposition experiments

Polyester fabrics were decomposed in the air atmosphere under isothermal conditions at 600ºC. The experiments were carried out in a flow tubular furnace. The samples of fabrics (~0.1 g) in the ceramic boat were placed in the furnace for 20 min, with the air flow kept at 0.030 m³/h. The delivered air was cleaned by passing through

Table 1. Characteristic of tested polyester fabrics

Sample	Thickness [mm]	Mass per unit area [g/m²]	Colour	Application
Fabric 1	0.90	413	beige	upholstery
Fabric 2	0.46	198	pale-yellow	curtains
Fabric 3	0.28	127	pale-yellow	curtains

the filters with molecular sieves 4A and silica gel. During experiments, the emitted solid decomposition products were trapped on a fiberglass filter placed at the outlet of the tube. The emitted volatile compounds were collected in a 0.0127 m³ – volume hermetic glass container, after previous evacuation of air using a vacuum pump. The gas samples were taken for the analysis after 30 min. stabilization at the room temperature. The apparatus used for the studies was shown schematically in Figure 1. The experiments were repeated 3 times for each sample of fabric and the obtained results were averaged.
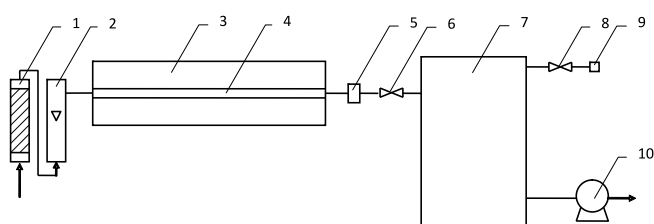

Figure 1. Apparatus used for the decomposition of polyester fabrics in thermo-oxidative conditions: (1) air-cleaning filter with silica gel and molecular sieves, (2) flowmeter, (3) flow tubular furnace, (4) ceramic tube, (5) fiberglass filter, (6) needle valve, (7) vacuum glass container, (8) glass cock, (9) septum, (10) vacuum pump

Analytical methods

The analyses of the compounds emitted during thermo-oxidative decomposition of polyester fabrics were performed mainly using gas chromatography (GC) and high performance liquid chromatography (HPLC) methods. Using of gas chromatographs with different detectors (MS, FID, TCD) and HPLC systems with UV detector enabled the identification and quantification of both volatile and solid decomposition products. The basis of the identification was comparison of retention times and mass spectra of the analytes with the respective data obtained for standards. The quantitative analyses were performed using external calibration method.

The GC-MS analyses were performed using an Agilent Technologies 6890N gas chromatograph equipped with the 5973 Network Mass Selective Detector. The analytes were separated on HP-5MSI capillary column (30 m x 0.25 mm I.D. x 0.25 μm film thickness). Mass spectra

were obtained using electron impact ionisation at 70 eV. In order to perform GC-MS analyses of volatile compounds from glass container, the analytes were adsorbed on active carbon and then extracted with two different solvents (acetone or butyl acetate). Those two solvents of different volatility enabled the analysis of both very volatile compounds, eluted before butyl acetate, and others, eluted after acetone. Temperature program applied for analysis of volatile compounds was started from 50°C (hold for 5 min) to 280°C (hold for 10 min), with the ramp rate of 10°C min⁻¹. The solid decomposition products before analysis were dissolved in 25 cm³ of methanol and separated using another program: 60°C (hold for 3 min) to 300°C (hold for 13 min), with the ramp rate of 10°C min⁻¹. The retention times and normalized mass spectra of volatile and solid decomposition products of tested fabrics analyzed by GC-MS method are presented in Table 2 and Table 3, respectively.

The quantitative analyses of emitted volatiles were carried out using GC-FID chromatograph (Chrom 5) with packed columns (A: Chromosorb 102, 80–100 mesh; B: SE 30 (10%) + Carbowax 20M (0.3%) on a Chromosorb W NAW, 60–80 mesh). Another gas chromatograph (ELPO N-504) with a TCD detector and column packed with silica gel (50–100 mesh) was used for carbon dioxide analyses. The analyses of carbon monoxide were carried out using a Combustion Analyser (KANE 400). Formaldehyde after absorption in redistilled water was analyzed by colorimetric method as a complex with chromotropic acid, using SPEKOL 11 spectrophotometer.

The high performance liquid chromatography (HPLC) method was used to quantitative analyses of solid compounds emitted during decomposition of tested fabrics, based on external calibration method. In HPLC chromatographs (LC-5b) reversed phase systems were used, consisted on non-polar stationary phase SEPARON SGX C-18 (7 μm) and two different mobile phases: methanol/water/acetic buffer pH = 3.6 (50 : 30 : 20) and methanol/water (80 : 20). The mobile phases were selected taking into consideration the nature of the analytes. The first mobile phase enabled analysis of the aromatic carboxylic acids; the addition of acetic buffer prevented their dissociation and enabled a good resolution. The second eluent was proper to perform the analysis of non-polar components of the samples – polycyclic aro-

Table 2. GC-MS data of the volatile compounds emitted from tested polyester fabrics

Compound	RT [min]	Fragmentation pattern at 70 eV [mass / charge (relative intensity)]
Acetaldehyde	1.36	44(M⁺, 77), 43(42), 42(14), 41(7), 29(100), 28(59), 27(7), 26(10)
Propionaldehyde	1.49	58(M⁺, 84), 57(25), 31(8), 29(100), 28(68), 27(51), 26(17)
Benzene	2.06	78(M⁺, 100), 77(23), 52(14), 51(14), 50(12), 39(7)
Toluene	3.26	92(M⁺, 70), 91(100), 65(15), 63(13), 51(10), 39(18), 27(7)
Ethylbenzene	5.66	106(M⁺, 38), 91(100), 78(8), 77(10), 65(11), 51(12), 39(9), 27(6)
Styrene	6.60	104(M⁺, 100), 103(44), 78(43), 77(20), 63(9), 52(8), 51(26), 50(15), 39(11), 27(8)
Benzaldehyde	8.68	106(M⁺, 100), 105(95), 78(18), 77(81), 74(10), 52(8), 51(31), 50(19), 39(5), 29(5)
Acetophenone	10.72	120(M⁺, 45), 106(10), 105(100), 77(76), 51(17), 43(6)
Vinyl benzoate	12.11	148(M⁺, 1), 106(11), 105(100), 77(65), 51(21), 50(9)

RT – retention time, M⁺ – molecular ion

Table 3. GC-MS data of the solid decomposition products of tested polyester fabrics

Compound	RT [min]	Fragmentation pattern at 70 eV [mass / charge (relative intensity)]
Benzoic acid	9.50	122(M^+, 89), 105(100), 77(62), 74(7), 51(23), 50(13)
4-Methylbenzoic acid	10.95	136(M^+, 86), 119(63), 92(15), 91(100), 65(19), 63(10), 51(7)
Biphenyl	12.58	155(M^++1, 12), 154(M^+, 100), 153(38), 152(25), 151(7), 76(8)
4-Vinylbenzoic acid	12.60	149(M^++1, 12), 148(M^+, 100), 131(89), 103 (39), 102(12), 77(29), 51(14), 50(8)
1,4-Diacetylbenzene	13.51	162(M^+, 24), 148(12), 147(100), 119(17), 91(25), 77(10), 76(8), 44(20), 43(16)
4-Acetylbenzoic acid	14.63	164(M^+, 16), 150(12), 149(100), 121(24), 75(6), 65(20), 51(6), 50(6), 43(8)
Monovinyl terephthalate	15.31	192(M^+, 1), 150(9), 149(100), 121(18), 76(7), 65(17), 50(5)
Terephthalic acid	17.40	166(M^+, 68), 149(100), 121(26), 76(8), 75(8), 74(8), 65(23), 50(9), 44(9)
Phenantrene	17.45	179(M^++1, 15), 178(M^+, 100), 177(11), 176(18), 152(8), 151(7), 89(8), 88(6), 76(6)
p-Terphenyl	21.40	231(M^++1, 20), 230(M^+,100), 229(9), 228(14), 227(5), 226(7), 202(6), 152(5), 115(7)

RT – retention time, M^+ – molecular ion.

matic hydrocarbons. The monitoring wavelength of UV detector was 254 nm.

RESULTS AND DISCUSSION

The thermo-oxidative decomposition process of tested polyester fabrics at 600°C resulted in the complete or almost complete sample destruction (weight losses in the range of 99.8–100%) was accompanied by the emission of complex mixtures of volatile and solid compounds. The amounts of compounds emitted from tested polyester fabrics were related to the 1 g of the samples and collected in Table 4 and Table 5.

The emission quantities were averaged from three separate experiments and standard deviations (s) of mean values were calculated. Obtained results indicate that applied method for determination of compounds emitted during thermo-oxidative decomposition of polyester fabrics is precise. For the highest levels of emission (e.g. carbon oxides, benzoic and terephthalic acids) standard deviations of emission were below 5% of average values. For substances emitted from the tested materials in smaller quantities standard deviation values were higher but in most cases did not exceed 10% of average values. Expanded uncertainty (U) of measurement results was calculated at 0.05 confidence level. This parameter defines a range of values within which the actual value exists with defined probability and is calculated from formula:

$$U = k \frac{s}{\sqrt{n}} \tag{1}$$

where: k – coverage factor (for confidence level 0.05 k = 2), s – standard deviation, n – number of measurements.

Calculated values of expanded uncertainty were rounded to two decimal places or to two significant digits (for low values). The corresponding average values of emission and standard deviations were rounded with the same precision.

Comparing the results obtained for the tested fabrics it is possible to notice some differences in the levels

Table 4. Emission of volatile compounds during thermo-oxidative decomposition of polyester fabrics at 600°C

Compound	Fabric 1			Fabric 2			Fabric 3		
	emission [mg/g]	s	U	emission [mg/g]	s	U	emission [mg/g]	s	U
Carbon monoxide	95.66	3.48	4.02	90.07	4.17	4.82	94.57	3.47	4.01
Carbon dioxide	874.88	33.72	38.94	891.95	12.91	14.91	1024.27	23.48	27.11
Formaldehyde	1.760	0.068	0.079	2.160	0.009	0.010	1.010	0.046	0.053
Acetaldehyde	22.82	0.26	0.30	17.06	0.76	0.88	21.70	0.98	1.13
Propionaldehyde	2.50	0.24	0.28	2.133	0.025	0.029	2.34	0.22	0.25
Benzene	15.43	0.72	0.83	17.35	0.79	0.91	25.35	0.16	0.18
Toluene	1.73	0.13	0.15	1.96	0.13	0.15	1.84	0.09	0.10
Ethylbenzene	0.573	0.052	0.060	0.697	0.074	0.085	0.806	0.055	0.064
Styrene	1.64	0.16	0.18	1.84	0.13	0.15	2.30	0.21	0.24
Benzaldehyde	0.91	0.09	0.10	0.80	0.09	0.10	1.58	0.16	0.18
Acetophenone	7.22	0.74	0.85	13.62	1.11	1.28	20.09	2.05	2.37
Vinyl benzoate	9.99	0.77	0.89	18.67	1.56	1.80	22.55	1.83	2.11

s – standard deviation (n = 3), U – expanded uncertainty (α = 0.05, k = 2).

Table 5. Emission of solid compounds during thermo-oxidative decomposition of polyester fabrics at 600°C

Compound	Fabric 1			Fabric 2			Fabric 3		
	emission [mg/g]	s	U	emission [mg/g]	s	U	emission [mg/g]	s	U
Benzoic acid	37.32	1.29	1.49	34.42	1.25	1.44	24.47	1.18	1.36
4-Methylbezoic acid	3.13	0.31	0.36	3.17	0.10	0.12	2.62	0.14	0.16
Biphenyl	0.217	0.022	0.025	0.318	0.017	0.020	0.225	0.034	0.039
4-Vinylbenzoic acid	7.49	0.53	0.61	8.45	0.89	1.03	4.73	0.26	0.30
1,4-Diacetylbenzene	0.486	0.035	0.040	0.84	0.09	0.10	0.476	0.026	0.030
4-Acetylbenzoic acid	16.83	1.20	1.39	19.48	1.53	1.77	9.43	0.51	0.59
Monovinyl terephthalate	29.19	0.59	0.68	32.91	1.39	1.61	21.60	0.97	1.12
Terephthalic acid	103.81	4.99	5.76	119.34	5.40	6.24	78.28	2.35	2.71
Phenantrene	0.297	0.018	0.021	0.408	0.025	0.029	0.213	0.013	0.015
p-Terphenyl	0.403	0.026	0.030	0.629	0.072	0.083	0.647	0.073	0.084

s – standard deviation (n = 3), U – expanded uncertainty (α = 0.05, k = 2).

of the particular emitted compounds and in the ratios between the volatiles and solids fractions. Fabric 3, with the lowest mass per unit area, decomposed forming the highest emission of carbon dioxide and other volatiles (benzene, acetophenone, vinyl benzoate). The emission of solid compounds, especially terephthalic acid, monovinyl terephthalate, benzoic acid and its derivatives were smaller than obtained for the two other fabrics. The percentage ratio of volatiles and solids fractions was in this case about 90 : 10, whereas for fabrics 1 and 2 – about 83 : 17. The total emissions, summarized and shown in Figure 2, are significantly higher than mass of the samples. This fact indicate the participation of atmospheric oxygen in formation of some oxygen-containing compounds.

The total emissions expressed in relation to area of the sample [g/m^2] are shown in Figure 3. Those values decrease with the decreasing of the thickness of the tested fabrics, as expected. The total emission from 1 m^2 of upholstery fabric was higher than 500 g/m^2, whereas emissions from fabrics for curtains were 2 and 3-times smaller.

For the emitted compounds the percentage share in total emission was calculated and collected in Table 6. The profiles of decomposition products for all tested fabrics were similar. The principal fraction consisted of carbon dioxide (70–75%) and carbon monoxide (7–8%). Besides carbon oxides, the main volatile compounds were benzene, acetaldehyde, vinyl benzoate and acetophenone. Some other aromatic hydrocarbons (toluene, ethylbenzene, styrene) and aldehydes (formaldehyde, propionaldehyde, benzaldehyde) were also detected. The emitted solids consisted mainly of aromatic carboxylic acids and esters, among which terephthalic acid, benzoic acid and monovinyl terephthalate took the greatest part. Small amounts of *p*-substituted derivatives of benzoic
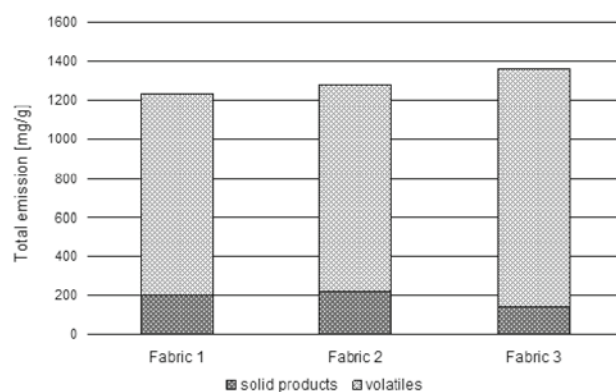

Figure 2. Total emissions of decomposition products from 1 g of polyester fabrics at 600°C
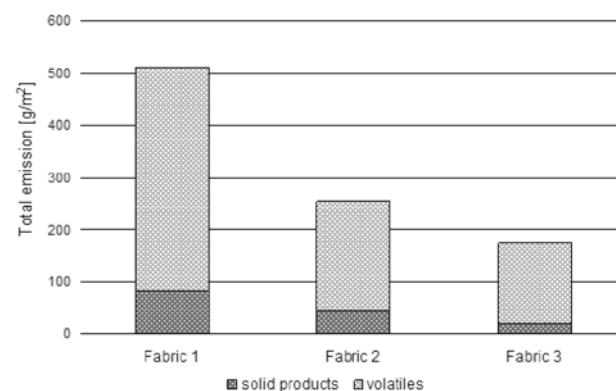

Figure 3. Total emissions of decomposition products from 1 m^2 of polyester fabrics at 600°C

acid and of polycyclic aromatic hydrocarbons (biphenyl, phenantrene, *p*-terphenyl) were also found.

Taking into consideration the kind of emitted compounds it is possible to confirm, that the basic polymer used in all tested fabrics is poly(ethylene terephthalate),

Table 6. The profiles of decomposition products of the tested fabrics

Group of compounds	Percentage of emission [%]		
Compound	fabric 1	fabric 2	fabric 3
Carbon oxides	78.63	76.83	82.20
Carbon monoxide	7.75	7.05	6.95
Carbon dioxide	70.88	69.78	75.25
Aldehydes	2.26	1.73	1.95
Formaldehyde	0.14	0.17	0.07
Acetaldehyde	1.85	1.33	1.59
Propionaldehyde	0.20	0.17	0.17
Benzaldehyde	0.07	0.06	0.12
Ketones	0.62	1.14	1.51
Acetophenone	0.58	1.07	1.48
1,4-Diacetylbenzene	0.04	0.07	0.03
Carboxylic acids	13.65	14.46	8.78
Benzoic acid	3.02	2.69	1.80
4-Methylbezoic acid	0.25	0.25	0.19
4-Vinylbenzoic acid	0.61	0.66	0.35
4-Acetylbenzoic acid	1.36	1.52	0.69
Terephthalic acid	8.41	9.34	5.75
Esters	3.17	4.03	3.25
Vinyl benzoate	0.81	1.46	1.66
Monovinyl terephthalate	2.36	2.57	1.59
Aromatic hydrocarbons	1.57	1.70	2.23
Benzene	1.25	1.36	1.86
Toluene	0.14	0.15	0.14
Ethylbenzene	0.05	0.05	0.06
Styrene	0.13	0.14	0.17
Polycyclic aromatic hydrocarbons	0.07	0.10	0.09
Biphenyl	0.02	0.02	0.02
Phenantrene	0.02	0.03	0.02
p-Terphenyl	0.03	0.05	0.05

which in the same conditions decomposes into similar products[20, 21]. Decomposition of polyesters at high temperatures in the presence of oxygene is very complex and involves both thermal and thermo-oxidative processes. In general it is believed that thermal degradation is initiated by random scission of the polymer chain at the ester linkage, whereas thermo-oxidation starts by the formation of hydroperoxide at the methylene group[10, 11]. Both processes result in formation of a vinyl ester and carboxyl end groups. Newly formed end groups decompose by secondary reactions, e.g. decarboxylation and hydrogen transfer, leading to a wide variety of compounds. The original hydroxyl end groups of PET decompose producing acetaldehyde, which is also formed in several different paths including transesterification of the end groups. Scheme of PET decomposition with formation of the main products is presented in Figure 4. Oxidation of the particular compounds is additional source of the carbon oxides emission.

CONCLUSIONS

The conditions applied during experiments can be related to burning of polyester fabrics. During fire people staying indoors can be exposed to the mixtures of compounds emitted from burning curtains, furniture, tablecloths or other polyester-containing elements. The obtained results indicate that the decomposition products of polyester fabrics contain some compounds of very high

toxicity, like carbon monoxide, aromatic hydrocarbons (including cancerogenic benzene) and polycyclic aromatic hydrocarbons. The presence of aldehydes, acetophenone and vinyl benzoate may cause eye and mucous membrane irritations. Large amounts of solid particles formed intensive smoke emission can also reduce visibility and hinder orientation during fire.

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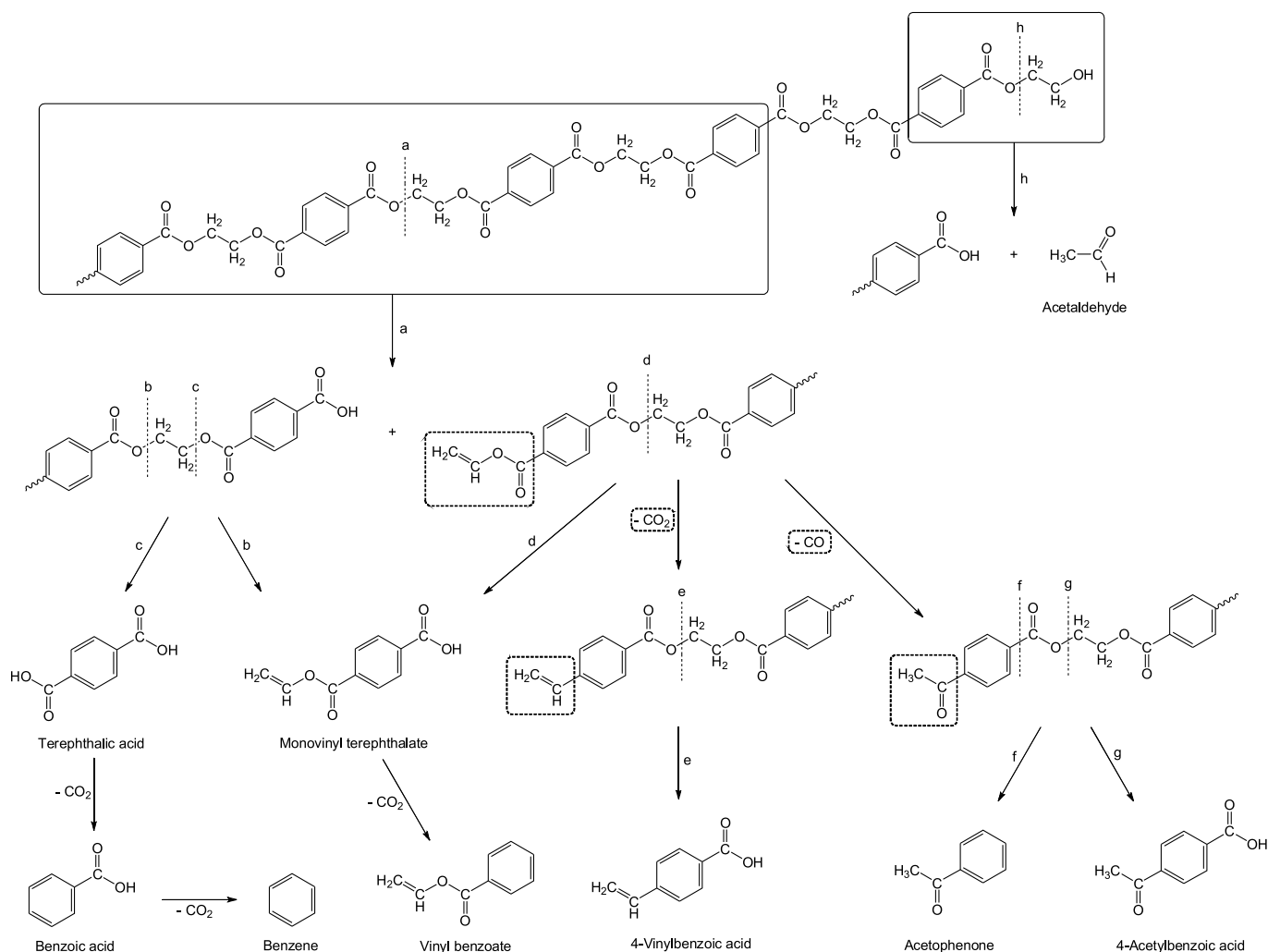
Figure 4. Formation of the main decomposition products of poly(ethylene terephthalate)

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