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Pyrolysis gas chromatography of synthetic polymers – a bibliography

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Abstract

An extensive bibliography on the pyrolysis gas chromatography of synthetic polymers is presented. The analysis of homopolymers, copolymers, of microstructure and end groups are included. References concerning pyrolytic alkylation and reactive degradation are included as separate tabulations. © 1998 Elsevier Science B.V. All rights reserved.

Keywords: Pyrolysis gas chromatography; Polymers

The analysis of polymers has been of importance for many decades. The analysis of many groups of materials were revolutionised by the introduction of the instrumental chromatographic techniques, typical being in the biological area where with lipids several thousand publications appeared in a few years. The application of gas chromatography to polymers was restricted due to their high molecular mass and low volatility. The molecular mass of some polymers was reduced and the volatility of the products increased by chemical degradation. In some cases, i.e. alkyd resins, the traditional methods of chemical cleavage were followed [1] with subsequent separation of the reaction products by gas chromatography [2–4] rather than by gravimetry. The Zeisel Reaction has also been applied to polymers, the use of gas chromatography allowing separation of the individual alkyl halides. The Zeisel Reaction was first

reported with acrylic resins [5] in 1958. Other early applications of chemical degradation of polymers included the acid hydrolysis of polyurethanes by Schroder [6] in 1962, degradation of polyethers by Nadeau and Williams [7] in 1963, acidic degradation of polysiloxanes by Heylmum and Piloula [8] in 1964, and acidic hydrolysis of polyamides by Anton [9] in 1968.

Prechromatographic reaction of polymers was given impetus by the work of Siggia and his many coworkers [10], who conducted the degradation reaction in a modified furnace pyrolyser attached to a gas chromatograph where the volatile reaction products were determined. The work of Siggia has been extended by Haken and various coworkers [11] who used the same reagent with reaction external to a gas chromatograph in a micro reactor. It was possible to achieve complete analysis of all of the reaction products either as liberated or as appropriate derivatives after prior chemical reaction.

The major use of gas chromatography with poly-

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Table 1
Qualitative or fingerprint pyrolysis of polymer and copolymer systems

Material	Reference
Acrylamide in emulsions	[35]
Acrylic acid in emulsions	[36]
Acrylic acid acrylic copolymers	[37]
Acrylic fibres	[38–40]
Acrylic lacquers (automotive paints)	[41–56]
Alkyd and acrylic architectural paints	[42,50,53,57–70]
Amino resins	[71]
Aramids	[72–74]
Aliphatic polyamides	[75]
Aromatic polyamides	[76]
Aryl-alicyclic polyimides	[77]
Chloromethyl substituted polystyrene	[78]
Chemically modified polystyrene	[79]
Chlorinated polystyrene	[80]
Chlorinated polyvinyl chloride	[81]
Chlorine containing polymers	[82]
Coumarone indene resin	[83,84]
Diol modified epoxy resin	[85]
EPDM rubber	[86–89]
Epoxy powder coatings	[90]
Epoxy resin	[91–120]
Epoxy urethane	[121]
Fibre epoxy composites	[103]
Fibre phenolic/epoxy composite	[104]
Fibre polycarbonate composite	[104]
Fibres various systematic characterisation	[38]
Flame retardant finishes	[122,123]
Fumaric acid in emulsion polymers	[124]
Functionally terminated polybutadiene	[125]
Fluorine polymers	[126,127]
Furfuraldehyde resin	[128]
Furfuryl alcohol resin	[129]
Hercosett 125	[130]
ion-exchange resins	[131–137]
Itaconic acid in emulsion polymers	[124]
Liquid crystal polyesters	[138–140]
Ketone resins	[141]
Methacrylic acid in emulsions	[36]
Modacrylic fibres	[38,39]
Nitrile rubber	[89,142,143]
Nitrile rubber–polyvinyl chloride	[89]
Nitrocellulose lacquers	[144,145]
Nylon	[40,146–153]
Petroleum resins	[154]
Phenol formaldehyde	[111,155–172]
Phenol formaldehyde–epoxy systems	[173]
Phenol formaldehyde–urethane	[174]
Phosphate esters of bisphenol A	[175]
Pitch resins	[176]
Polyacrylonitrile fibres	[38]
Polyacrylonitrile–butadiene	[177,178]
Polyacrylonitrile–styrene	[179,180]

Table 1. Continued

Material	Reference
Polyacrylamide	[181,182]
Poly(alkylene terephthalates)	[183–186]
Polyalkyl acrylates	[40,187] [188]
Polyamide fibres	[40]
Polyalkyl methacrylates	[40,187–191]
Polyaryl ether sulfones	[192]
Polyacrylic acid	[38,193–195]
Polyacrylonitrile	[126,196,197]
Polyamides	[198–200]
Polybutadiene	[201–210]
Polybutadiene–polyisoprene	[89]
Polybutene	[211–213]
Polybutyl acrylate	[214–216]
Polybutyl methacrylate	[217]
Polycarbonates	[151,218]
Polycarbonate siloxane copolymer	[199]
Polychlorotrifluoro ethylene	[219]
Polychloroprene	[142,220,221]
Polyetherketone	[222]
Polyester	[221,223–226]
Polyester–melanine–acrylic–epoxy paints	[227]
Polyesteramide fibres	[228]
Poly(2,6-dimethyl-1,4-phenylene ether)	[229]
Polyester–polyether elastomers	[230]
Polyether(aromatic–aliphatic)	[231]
Polyethylenechlorosulphonate	[89]
Polyethyl acrylate	[193,232,233]
Polyethylene	[201,213,234–246]
Polyethylene–propylene	[247–250]
Polyethylene terephthalate	[251]
Poly 2-fluorostyrene	[252]
Polyhydroxy methacrylates	[253]
Polyisoprene	[89,201,254–264]
Polyisobutylene	[100,265–267]
Polyisobutylene–polyisoprene	[89]
Polyimide	[268]
Polyimide aromatic Polyether–polyketones	[269]
Polymethyl acrylate	[232,270,271]
Polymethyl methacrylate	[151,163,172,232,272–277]
Polymethyl-2-cyanoacrylate	[278]
Polymethylene	[279,280]
Poly(3-methyl-1-alkenes)	[153]
Poly- α -methyl styrene	[151,281]
Polynaphthalene	[282]
Polyoxymethylene	[283]
Poly(phenyl-as-triazines)	[284]
Polyphosphate esters of bisphenol A	[174]
Polypropylene	[238,285–292]
Polypropyl acrylate	[214]
Polypyrrole	[293]

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Table 1. Continued

Material	Reference
Polyquinone	[294]
Polysiloxanes	[142,295–297]
Polystyrene	[126,163,252,298–310]
Polystyrene–butadiene	[89,258,259,311]
Polystyrene–divinyl benzene	[312,313]
Poly(styrenesulphides)	[314]
Polysulphides	[315–318]
Polyurethane	[319–334]
Polyvinyl chloride	[163,298,335–343]
Polyvinyl chloride–butyl acrylate	[344]
Polyvinyl acetate	[12,298,311]
Polyvinyl acetate–tetrafluoroethylene	[345]
Polyvinyl alcohol	[145,298]
Polyvinyl butyral	[346]
Polyvinylidene chloride	[298,347]
Polyvinylidene fluoride	[209]
Polyvinyl chloride–polyvinylidene chloride	[298]
Polyvinyl propionate	[298]
Polyvinylsilanes	[182]
Polyvinylpyrrolidone	[348]
Resin modified paints	[349]
Silicone alkyd	[68]
Sodium polyacrylate	[350]
Styrene copolymers	[351]
Styrene copolymer sizing agents	[352]
Styrene crosslinked polyesters	[353]
Styrene crosslinked vinyl esters	[353]
Styrene–maleic anhydride copolymer	[354]
Tetrafluoroethylene–hexafluoropropylene	[355–357]
Thermosetting acrylic polymers	[358–360]
Various rubbers	[361]
Vinylidene fluoride–perfluoromethyl vinyl ether	[355]
Vinylpyrrolidone–vinylimidazolium chloride	[362]

Table 2

Quantitative Analysis of Copolymer Systems

Copolymer Systems	Reference
ABS–methyl styrene	[363]
Acrylic acid–ethyl acrylate–ethyl methacrylate	[364]
Acrylic resins–amino resins	[71]
Adipates of ethylene oxide, butanediol, octanediol	[365]
Butadiene–isoprene	[366]
Butylene terephthalate–polycarbonates–SBR–acrylonitrile	[367]
<i>n</i> -Butyl methacrylate–isobutyl methacrylate	[368]
Chlorbutyl rubber–natural rubber	[369]
Chlorbutyl rubber–styrene–butadiene	[370]
Chlorinated polyethylene	[370]
Elastomers	[285,369,371]
Ethyl acrylate–styrene	[372,373]
Ethylene–butene	[237,247]
Ethylene–ethyl acrylate	[374]
Ethylene–chlorotrifluoroethylene	[127]

Table 2. Continued

Copolymer Systems	Reference
Ethylene–isobutylene	[375,376]
Ethylene–methyl methacrylate	[377,378]
Ethylene–methyl acrylate	[377]
Ethylene–propylene	[216,238,247,278,379,380]
Ethylene–butene–hexene–octene	[237,247,381]
Ethylene–styrene	[382]
Ethylene–vinyl acetate	[383–386]
Ethylene–propylene–dicyclopentadiene	[247]
Ethylene oxide–propylene oxide	[265,266]
Ethylene oxide–trioxane	[387]
2-Hydroxyethyl methacrylate–acrylate–methacrylate	[358,388]
Terepolymers	
2-Hydroxymethyl methacrylate–acrylate–methacrylate	[358]
Terepolymers	
2-Hydroxyethyl methacrylate–butyl acrylate–ethyl methacrylate	
Crosslinked with melamine resin	[389–391]
Isoprene–methyl methacrylate	[392]
Isoprene–styrene	[393,394]
Isopropene–vinylxylene	[395]
Isopropene–vinyltoluene	[395]
Kraton 1108	[394]
Melamine–crosslinked acrylics	[389–391]
Methyl acrylate–acrylonitrile	[396,397]
Methyl acrylate–methyl methacrylate	[14,397–399]
Methyl methacrylate–methacrylic acid	[14]
Methyl methacrylate–ethylene glycol dimethacrylate	[14]
Methyl, ethyl, <i>n</i> -propyl, <i>n</i> -butyl, <i>n</i> -pentyl, <i>n</i> -hexyl, 2-ethylhexyl Acrylate, methyl, ethyl, <i>n</i> -propyl, <i>n</i> -butyl, <i>n</i> -pentyl, <i>n</i> -hexyl and 2-ethylhexyl methacrylate copolymers	[400–402]
Natural vulcanizates	[210]
Polyimide (ethers)	[403]
Poly(<i>p</i> -phenylene sulfide–sulfone) block copolymer	[404]
Methyl methacrylate–acrylic acid	[405]
Methyl methacrylate–diallyl phthalate	[406]
Methyl methacrylate–ethyl acrylate	[372,402,407–410]
Methyl methacrylate–ethyl acrylate– <i>N</i> -methylol acrylamide	[408]
Methyl methacrylate– <i>n</i> -butyl acrylate	[72,398,399]
Methyl methacrylate– <i>n</i> -propyl methacrylate	[398,399]
Methyl methacrylate– <i>n</i> -ethyl methacrylate	[14,398,399,411]
Methyl methacrylate– <i>n</i> -butyl methacrylate	[398,399,412,413]
Methyl methacrylate– <i>n</i> -hexyl methacrylate	[399,394]
Methyl methacrylate–isobonyl methacrylate	[412]
Methyl methacrylate–isoprene	[414]
Methyl methacrylate– <i>n</i> -pentyl methacrylate	[399]
Methyl methacrylate–polyethylene terephthalate laminate	[415]
Methyl methacrylate–acrylonitrile	[416]
Methyl methacrylate–vinyl chloride	[417]
Methyl methacrylate–vinylidene chloride	[418]
Methyl methacrylate–ethyl acrylate–2-ethylhexyl methacrylate	[419]
Methyl methacrylate–2-ethylhexyl acrylate–acrylic acid	[412]

(Continued on p. 175)

Table 2. Continued

Copolymer Systems	Reference
Methyl methacrylate–styrene	[216,317,411,417–423]
Methyl methacrylate–styrene–diallyl phthalate	[424]
Methyl methacrylate–styrene–butadiene	[425]
α -Methylstyrene–methyl acrylate	[426]
α -Methylstyrene–methyl methacrylate	[426–428]
α -Methylstyrene–styrene	[427–429]
Mixture of Natural Rubber, Butadiene–Styrene and SBR Rubbers	[430]
Multicomponent acrylic resin	[407,412,431–435]
Nylon 66	[149,150]
Styrene–acrylamide	[436]
Styrene–acrylonitrile	[213,437–447]
Styrene–butadiene	[213,441,448–451]
Styrene–butadiene–acrylonitrile	[210,446,452,453]
Styrene–butadiene–natural rubber–ethylene–propylene	[446,454]
Terepolymer	
Styrene–butyl acrylate	[215,455]
Styrene–butyl methacrylate	[421]
Styrene copolymers–ABS–polycarbonate Blends	[456]
Styrene–dimethylsiloxane block copolymers	[457]
Styrene–divinyl benzene	[113,232,458,459]
Styrene–ethyl acrylate–acrylic acid	[460]
Styrene–2-fluorostyrene	[252]
Styrene–glycidyl methacrylate	[461]
Styrene–maleic anhydride copolymers	[354,462]
Styrene–methacrylate terepolymer	[463]
Styrene–methyl acrylate	[464]
Styrene–polyester	[426]
Styrene–propylene	[465]
Styrene–vinylcyclohexane	[466]
Styrene–vinyl ester	[353]
Styrene– <i>N</i> -vinylpyrrolidone	[467]
SBR–polybutadiene	[433,452,468]
Tetrafluoroethylene–ethylene	[468]
Tetrafluoroethylene–hexafluoropropylene	[127]
Tetrafluoroethylene–perfluoromethyl vinyl ether	[127]
Tetrafluoroethylene–1,1,5-trihydroperfluoroamyl acrylate	[469]
Phenol formaldehyde	[470,471]
Vinyl acetate–vinyl chloride	[472–475]
Vinyl acetate– <i>n</i> -butyl acrylate	[215,476]
Vinyl acetate–butyl maleate	[476]
Vinyl acetate–2-ethylhexyl acrylate	[476]
Vinyl acetate–2-ethylhexyl maleate	[476]
Vinyl acetate–vinyl trifluoroacetate	[477]
Vinylidene chloride–methyl methacrylate	[478]
Vinylidene fluoride–hexafluoropropylene	[127]
Vinylidene fluoride–chlorotrifluoroethylene	[127]
Vinylidene fluoride–tetrafluoroethylenehexafluoropropylene	[127]
Vinylidene fluoride–tetrafluoroethyleneperfluoromethyl vinyl ether	[127]

mers is in association with pyrolysis. The technique with polymers is well established, the first application being in 1954 [12] where the pyrolysis was conducted separately, however in situ pyrolysis soon

followed with the use of a filament device [13,14] little different to that which has subsequently found extensive usage.

The early works used a large sample resulting in

Table 3
Microstructure of polymer and copolymer systems

Material	Reference
Acetylenic polymers	[479]
Hydrogenated acrylonitrile–butadiene	[480]
Ethyl acrylate–butyl methacrylate copolymers	[481]
Acrylonitrile–fluoroacrylate	[482]
Acrylonitrile–1,3-pentadiene	[483]
Poly(di- <i>n</i> -alkylsilylene)	[484]
<i>cis</i> -1,4-Butadiene-isoprene	[366,485]
Chlorinated polyethylene	[370,486]
Chlorostyrene–acrylonitrile	[487]
Chlorostyrene–styrene	[488]
Deuterated styrene	[489]
Ethyl acrylate–butyl methacrylate	[481]
Ethylene–vinylcyclohexane	[490]
Phenol–formaldehyde	[491,492]
Polyaryl ether sulfones	[192]
Poly(butadiene–acrylonitrile–methacrylic acid)	[493]
Poly(di- <i>n</i> -alkylsilylene)	[494]
Polyesteramides	[228]
Polyacrylonitrile	[495]
Polybutadiene	[312,496–500]
Polyethylene	[179,243,244,247,254,279]
	[280,312,501–505]
Polyisobutylene	[202,255,506,507]
Poly α -methyl styrene	[152]
Poly-1,3-pentadiene	[207]
Polypropylene	[202,312,508–515]
Polyethylene–propylene copolymer	[516–521]
Polyethylene–polypropylene–diene rubbers	[169,522]
Polymethyl methacrylate	[523]
Polystyrene	[152,524–527]
Polystyrene–acrylonitrile	[589]
Polystyrene–butadiene	[206,489]
Polystyrene–butyl acrylate	[415]
Polystyrene–divinyl benzene	[528]
Polystyrene–ethyl acrylate–ethyl methacrylate	[529]
Polystyrene–Glycidyl Methacrylate	[460]
Polystyrene–heptyl acrylate	[530]
Polystyrene–maleic anhydride copolymer	[346]
Polystyrene–methyl acrylate	[531]
Polystyrene–methyl methacrylate	[532]
Polystyrene–methyl methacrylate–acrylonitrile	[533]
Polytetrafluoroethylene–ethylene	[468]
Polytetrafluoroethylene–ethylene-hexafluoropropylene	[534]
Polyvinyl chloride	[35,535]
Polyvinylidene chloride–vinyl chloride	[349,536]
Chlorinated polyethylene	[178]

poor heat transfer and the production of non-reproducible results. Early libraries of pyrolysis results either as pyrograms or bar charts are of limited value as the reproducibility is poor and it is frequently difficult to determine which products result from the initial pyrolysis reaction and which are due to subsequent recombination reactions. It has long been recognised that small samples, typically much less than 1 mg, good heat transfer and rapid heating of the pyrolysis element are essential in achieving reproducibility. While capillary columns [15] and mass spectrometric detection [16] had been used for decades, the widespread use of enhanced separation with capillary columns and mass spectrometric and Fourier Transform Infrared Detection have been more recent.

The terms used in pyrolysis have been described [17], while IUPAC recommendations were published in 1993, entitled Nomenclature and Terminology for Analytical Pyrolysis [18]. Pyrolysis gas chromatography has been the subject of several books and has been described in chapters in monographs [19–26] and also in a greater number of reviews. The most recent being that of Wampler [27] which appeared in 1989 covered the period 1980–1989 and included almost 500 references to the application of pyrolysis to many types of compounds, about 80 of the references relating to polymers.

Data bases of references on pyrolysis gas chromatography have been reported by Smith [28] and by Tsuge and Ohtani [29] but being in book form, searching has been restricted considering the large amounts of data involved. Wang [30] has reported a computer based system which considers the mass spectrum of the pyrolysis products and uses the Hypertext Markup Language.

Matheson and her coworkers [31] have recently reported that a computer library is being prepared for the identification of polymers. Such a library may be of considerable value in exploitation of the technique.

The following tables shows some of the synthetic polymer systems that have been examined for various characteristics by the chromatography and pyrolysis based techniques. Table 1 shows qualitative or fingerprint analyses of polymer and copolymer systems, Table 2 shows quantitative of polymer and copolymer systems, Table 3 shows

Table 4

End group analysis of polymer and copolymer systems

Material	Reference
Acetylene polymers	[479]
Polycarbonates	[476]
Polymethyl methacrylate	[532–538]
Polystyrene	[539]
Polystyrene macromonomers	[540–546]

Table 5

Alkali and acid fusion

Polymer	Reference
Aramid	[547]
Epoxy resins	[548]
Liquid crystal polyesters	[549]
Polyacrylamide	[550]
Polyacrylonitrile	[550]
Polyamides (nylons)	[550,551]
Polyamides (dimer)	[552–554]
Polyamides–imides	[555,556]
Polycarbonates	[557]
Polycarboranesiloxanes	[558]
Polyesters	[559,560]
Polyesters (crosslinked with aminoplasts)	[561,562]
Polyhydrazides	[563]
Polyimides	[555]
Polymethacrylates	[564]
Polysiloxanes	[565,566]
Polysulphones	[567]
Polyurethanes (esters)	[568–571]
Polyurethanes (ethers)	[572–574]
Polyvinyl esters	[575]
Silicone polyesters	[576–579]
Silicone polyesters (crosslinked with aminoplasts)	[580]
Vinyl esters	[581]

Table 6

Pyrolysis and alkylation of polymer and copolymer systems

Material	Reference
Alkyd resins	[582,583]
Cyanoacrylates	[582]
Epoxy resins	[582]
Liquid crystal polyesters	[584]
Nylon	[585]
Polyacrylamide	[586]
Polyamides	[587]
Polycarbonate	[588,589]
Rosin adducts	[590–592]
Urea formaldehyde resins	[593]
Varnish (diterpenoid)	[594]

examination for microstructure or sequence distribution of polymer and copolymer systems, Table 4 shows end group analyses of the same systems, Table 5 Table 6 show polymer and copolymer systems that have been examined using reaction chromatography and by simultaneous pyrolysis methylation respectively. Microstructure of Polymers (Table 3) was reviewed in 1995 by Tsuge and Ohtani [32] While the topics shown in Tables 5 and 6 was reviewed by Haken in 1993 [33] and 1996 [34] respectively.

This compilation does not claim to be exhaustive, but includes many references to the important polymer systems.

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