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Review

Applications of evolved gas analysis
Part 2: EGA by mass spectrometry

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Abstract

The analytical applications of the evolved gas analysis (EGA) performed by mass spectrometry, for the period extending from 2001 to 2004, are collected in this review. By this technique, the nature of volatile products released by a substance subjected to a controlled temperature program is on-line determined, with the possibility to prove a supposed reaction, either under isothermal or under heating conditions.

Keywords: EGA; MS; Evolved gas analysis; Mass spectrometry

Contents

1. Introduction

Evolved gas analysis (EGA) is the most useful technique to determine the nature and amount of volatile products formed during the thermal degradation of materials.

Two approaches are generally used for EGA, simultaneous analysis and combined analysis. In the simultaneous analysis approach, two methods are employed to examine the materials at the same time, such as pyrolysis or TG–FTIR and pyrolysis or TG–MS on-line analysis, in which decomposition products that evolve from pyrolyzed materials can be monitored simultaneously. The combined analysis technique, on the other hand, employs more than one sample for each instrument, and real time analysis is not possible. Intermittent or batch mode refers

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to trapping evolved gaseous species at a low temperature or in an absorption chamber and the trapped products are then introduced into a detector for identification, such as a TG–GC–MS or pyrolysis–GC–MS system. This last mode allows the investigator to optimize the detector parameters to make the best choice for different samples.

EGA–FTIR and EGA–MS hyphenated techniques applications have been previously reported [1–5].

This paper collects the applications of the evolved gas analysis performed by mass spectrometry for the period extending from 2001 to 2004.

Many examples are reported from the literature, and often the references are generally obtained from the journals that specialize in thermal analysis. However, the number of publications on hyphenated techniques continues to grow in areas of specialized applications; as a consequence, it is not unusual for an article on the topic to appear in an unfamiliar journal or a trade-specific publication. The problem is that unless the terminology relating to the specifics of the hyphenated technique is present in the published keywords, the articles may be difficult to locate. As a result, certain important articles may have been overlooked, and the authors apologize for such inadvertent omissions.

2. 2001 applications

2.1. Polymers and inorganics

In 2001, the formation of species when $BaSnO_3$ is exposed to SO_2 under a variety of conditions was reported and discussed by Hodjati et al., who demonstrated that direct contact with 25 ppm SO_2 during 15 cycles of Lean–Burn operation leads to the complete loss of NO_x absorption capacity owing to the formation of stable and irreversible bulk-like sulfates identified by FTIR (990, 1060, 1125 and 1190 cm^{-1}) and TGA–MS [6].

By TG–MS spectra, the effects of temperature and time of reductive dechlorination of $RuCl_3$ to remove negative-effected chlorine were investigated [7].

Dynamic and isothermal thermogravimetry/mass spectrometry (TG–MS) method in an inert environment was applied for the characterization of weakly basic ion exchanger prepared from water-soluble starch, cross-linked with epichlorohydrin in the presence of NH_4OH [8].

Solid state temperature-programmed desorption profiles by EGA–MS spectra of a starting material provided the 25% probability of endohedral T at C_{60} formation at 882 °C [9].

Thermal stability of $Y(thd)_3$ has been studied by EGA–MS, and the decomposition of the complex has been observed at 450–490 °C in an inert atmosphere (but already by 400 °C in the presence of oxygen) [10].

Bart reviewed the applicability of various flash pyrolysis methods (Py–GC, Py–MS and Py–GC–MS) for the qualitative and quantitative analysis of additives in polymeric materials [11].

The oxidative degradation of electrochemically synthesized *p*-toluene sulfonic acid-doped polypyrrole was studied by the application of FTIR evolved gas analysis and direct insertion probe pyrolysis mass spectrometry [12].

A pyrolysis–GC–MS study was presented by Blazsó on two pairs of hindered amine light stabilizers (HALS) oligomers and polymers, also in the presence of PVC, in order to observe the possible effect of this hardly avoidable plastic waste component on the production of volatiles from HALS compounds [13].

Graft polymerization of acrylonitrile onto cellulosic filter paper competing with the homopolymerization by mutual irradiation technique was studied in the presence of homopolymer suppressors by Py–GC–MS [14].

The effect of wood, cellulose, lignin and activated charcoal on the thermal decomposition of polystyrene (PS) and polyethylene (PE) has been studied in order to investigate the thermal behavior of these materials occurring in municipal waste [15]. Thermogravimetry–mass spectrometry and pyrolysis–gas chromatography–mass spectrometry revealed that these materials had a similar influence on PS and PE thermal decomposition under slow and fast heating conditions, respectively [16].

Three different apparatus, a thermobalance, a pyroprobe and a laboratory furnace, coupled with a GC–MS apparatus, were used to study the kinetics of decomposition and to identify volatile and semi-volatile organic compounds generated by the thermal degradation reactions of a polyurethane in an inert atmosphere [17].

The kinetics of the thermal decomposition of polytetrafluoroethylene (PTFE) have been studied by thermogravimetry coupled to mass spectrometry [18]. A TGA–MS study on the pyrolysis of polyamide 6 under catalytic conditions and its application to reutilization of carpets was proposed by Bockhorn et al. [19], while aliphatic and aromatic polyamides (polyamide-12, polyamide-6,6 and poly(1,4-phenylene terephthalamide) (Kevlar)) with poly(vinyl chloride) (PVC) were examined by pyrolysis–gas chromatography–mass spectrometry [20].

The thermal degradation of polystyrene produced by nitroxide-controlled radical polymerization [21] and the effect of several solvents on the polymerization of acrylonitrile [22] were studied by Py–GC–MS hyphenated technique. The thermal degradation of polyamide 6, polyacrylonitrile and of a polyurethane rigid foam was studied using two different thermal analyzers with coupled techniques for the evolved gas analysis (TA–MS and TA–FTIR) and two combustion devices [23].

The use of tetramethylammonium hydroxide to perform the “thermochemolysis”, followed by GC–MS analysis, has gained more attention in the analysis of polymers, especially biopolymers, and Schwarzinger et al. applied this technique for the analysis of various cellulose esters [24].

The thermal degradation of poly(ether-imide) (PEI) was studied through a combination of thermogravimetric analysis and stepwise pyrolysis–gas chromatography/mass spectrometry (Py–GC–MS) techniques with consecutive heating of the samples at fixed temperature intervals to achieve narrow temperature pyrolysis conditions [25]. Novel PMR polyimides (TMBZ-15) based on substituted benzidines were characterized and compared to the state-of-the-art PMR-15 by evolved gas analysis (either TG–FTIR or Py–GC–MS) to particularly verify the pathway of polyamide degradation [26,27]. Both TG–FTIR and Py–GC–MS evolved gas analysis were also applied to study the thermal degradation behavior of epoxy resin blended with propyl ester phosphazene [28].

Thermal degradation of poly(arylene-sulfone)s was studied by the combination of thermogravimetric analysis/mass spectrometry with pyrolysis/gas chromatography/mass spectrometry techniques [29].

TG–FTIR and TG–DTA–MS studies were proposed to investigate the properties of *cis*-1,4-polybutadiene both in air and in inert atmosphere [30].

Conducting polymer coatings on ABS were prepared by dipping them into *m*-cresol solutions containing polyaniline (PANI), poly(*o*-toluidine) (POT) and poly(*o*-anisidine) (PoAN) protonated with dioctyl hydrogen phosphate (DiOHP) in the presence and absence of polystyrene and characterized by TG–MS, SEM and X-ray diffraction [31].

Thin plasma polymer films were deposited from a mixture of dichloro(methyl) phenylsilane (DCMPS) vapor and gaseous hydrogen in an rf (13.56 MHz) capacitive coupling deposition system on pieces of silicon wafers and the thermal stability and decomposition of the plasma polymer with increasing temperature were characterized using thermogravimetry together with mass spectrometry [32].

The preparation of mixed crystals $Ba_{1-x}Sr_xTiO_3$ $(0 \leq x \leq 1)$ by an acetate-based sol–gel method was monitored by TG–DTA, TG–MS and XRD with respect to the reaction paths and the quality of the products [33].

The temperature-programmed decomposition (TPD) of thorium oxalate hexahydrate was studied by TGA–MS evolved gas analysis by Dash et al. [34].

TGA–MS and other techniques were employed to characterize the alluaudite-type manganese arsenates of Tl, Cs, Na and K and to determine the influence of the cation size [35].

Detailed thermoanalytical experiments were carried out to reveal the oxidative and reductive processes in the Bi_2TeO_5/Bi_2TeO_6 system [36].

Poly(ε-caprolactam) and its copolymers with ε-caprolactone were studied by thermogravimetric analysis followed by FTIR–EGA and MS–EGA to determine the structure of the copolymers [37].

2.2. Other applications

Principal component analysis (PCA) and variance diagram (VarDia) technique have been used for curve resolution in time-resolved mass spectrometry data. A set of simulated data corresponding to an unresolved gas chromatography–mass spectrometry or thermogravimetry–mass spectrometry peak and a set of real unresolved GC–MS data consisting of four compounds were used for evaluating the PCA–ContVarDia method [38].

To clarify the relation between organic sulfur distribution in coal and the emission of sulfur-containing gases, six Japanese standard coals, three Argonne premium coals and one Chinese coal were pyrolyzed using a thermogravimetric analyzer–mass spectrometer and a Curie-point pyrolyzer [39].

By hyphenated TG–MS, a comparison of different methods for predicting coal devolatilization kinetics was proposed by Arenillas et al. [40].

Micro-pyrolysis ovens coupled to GC–MS have yielded abundant information about the structure and the characteristics of the distillates obtained from selected Spanish oil shales [41].

In order to determine the decomposition products and the kinetics of the thermal decomposition, the pyrolysis and the combustion of polycoated cartons were studied by coupled thermogravimetry–mass spectrometry [42].

The structural characterization of a set of kerogen concentrates belonging to the three well differentiated conventional main types (I, II and III) has been undertaken by pyrolysis–gas chromatography–mass spectrometry both in the absence and presence of tetramethylammonium hydroxide (TMAH) [43].

Fabbri described the use of pyrolysis–gas chromatography–mass spectrometry to study the environmental pollution caused by synthetic polymers in the particular case of the Ravenna Lagoon [44].

Since a well-characterized pyrolytic lignin can be of interest for commercial applications such as adhesives in wood-based panel industry, the ratios of hydroxyphenyl-, guaiacyl- and syringyl-units (H-, G- and S-units) were obtained from pyrolysis gas chromatography–mass spectrometry (pyroprobe–GC–MS) and were compared with milled wood lignin (MWL) data [45]. Using Py–GC–MS it was also found that the composition of wheat lignin was modified by *P. eryngii* causing a decrease of S:G ratio in the residual lignin [46–49] and it was elucidated whether the chemical modifications produced by streptomycetes on wheat straw are related with the lignocellulose degrading enzymes produced by the microorganism on such substrate [50].

Lichens were analyzed by Py–GC–MS to investigate their potential use as stress monitors by examining changes in their chemistry under various environmental conditions [51].

Pyrolysis–gas chromatography–mass spectrometry has also been proved to be a well suited technique for identification of pictorial organic materials because of its response specificity and the possibility of obtaining structural information from very small samples without the need of any preliminary treatment. Scalarone et al. reported a study on the application of Py–GC–MS to the analysis of artificially aged linseed oil [52]. Nakamura et al. described the analysis of waterborne paints by gas chromatography–mass spectrometry with a temperature-programmable pyrolyzer, and the EGA profile suggested the optimal thermal desorption conditions for solvents and additives and the subsequent optimal pyrolysis temperature for the remaining polymeric material [53].

Evolved gas analysis by TGA-GC–MS, combined with volume-expansion measurements, was used to determine the composition of feed gases as they evolved during the progressing conversion of feed to glass in a study to determine the effect of feed melting, temperature history and minor component addition on spinel crystallization in high-level waste glass [54].

Giron proposed a critical review on the main features in the investigation of polymorphic behavior of pharmaceuticals and emphasizes the role of combined thermoanalytical techniques for the proper interpretation of complex problems [55].

Price et al. described the localized evolved gas analysis by micro-thermal analysis, a technique that employs a scanning probe microscope fitted with a miniature resistive heater/thermometer to obtain images of the surface of materials and then perform localized thermoanalytical measurements [56].

An off-line application was reported by Aas et al. where the gas collection is done by inserting sample tubes filled with a suitable adsorbent into the exhaust from the TG instrument [57].

A TG–DTA/GC–MS coupled apparatus was employed to clarify the thermal decomposition of asphaltenes, mainly responsible for the formation of coke in petroleum processing, since the phenomena involved are not yet clearly understood [58].

The chemical–physical decomposition processes that occur in a brake pad heated to 1000 °C have been studied. This temperature can be reached when a brake pad is applied. Thermogravimetry and differential thermal analysis were used in combination with evolved gas analysis, and image analysis using a scanning electron microscope [59].

Capillary and skimmer interfaces in EGA–MS were compared by the analysis of the carbon substances (impurities in ceramic raw materials) remaining in the hydrolysis products of aluminum alkoxides [60].

Thermal analysis of Israeli phosphorites by TG–DTA methods complemented with titrimetric and thermogas chromatographic determination of the evolved gases was performed in an oxygen and inert gas flow, to determine the amounts and the temperature intervals of H_2O, CO_2 and SO_2 evolvement [61].

Xie and Pan published a review on the application of the evolved gas analysis for the thermal characterization of materials [62].

Quantitative quadrupole mass spectrometry of (Ti, W)C-based cemented carbonitrides was carried out in order to monitor the evolution of the gas species as a function of time and temperature during vacuum sintering [63].

The thermal decomposition of four frothing agents used in the flotation of gold bearing sulfide minerals was investigated, both alone and when adsorbed on activated carbon. Thermal desorption–pyrolysis–gas chromatography–mass spectrometry (TD–Py–GC–MS) was used to determine the gases evolved from the thermal decomposition [64].

The possibility to understand the relationship between the molecular structure and the thermal stability (decomposition temperature, rate and degradation products) of the organic modification of the layered silicate was proposed by a study with thermoanalytical techniques combined with infrared spectroscopy and mass spectrometry (TGA–FTIR–MS) [65].

Flammability hazards and mechanisms were analyzed for milligram-scale samples of polymers by integrating the use of a new TGA/DSC/GC–MS technique, the PCFC microcalorimeter developed by Lyon, and computational quantum chemistry [66].

Samples of three-dimensional microporous zinc phosphates with fully connected framework with faujasite and chiral zincophosphate structures were characterized by TGA–MS evolved gas analysis [67].

3. 2002 applications

3.1. Polymers and inorganics

A university level laboratory experiment utilizing Py–GC–MS of polystyrene and ABS polymer was reported as a teaching tool [68].

In a study published by Hwu et al., modern thermal analysis techniques combined with infrared spectroscopy and mass spectrometry were used to obtain information on the thermal stability and degradation products of organic modified clay [69].

The curing chemistry involved in the polyimide formation was studied in real time with thermogravimetry/Fourier transform infrared (FTIR)/mass spectrometry evolved gas analysis techniques. The off-gas reaction products identified by FTIR and MS essentially confirmed the literature polyimide curing mechanisms [70].

The thermal decomposition of poly[diethyl 2-(methacryloyloxy)ethyl phosphate] (PDMP) by free-radical polymerization was studied by the combination of pyrolysis–gas chromatography/mass spectrometry with thermogravimetric analysis/mass spectrometry, in which the former identified directly the compositions of the evolved gases and the latter could further obtain real-time evolution curves of pyrolysates [71].

Jin et al. and Huang et al. investigated the thermal degradation mechanism of high-performance polyimide with norbornene ends using Py–GC–MS [72,73].

In a study of organic material in fine atmospheric particulates using Py–GC–MS, Fabbri et al. pyrolyzed poly(styrene-*co*-isoprene), polylimonene and polypinene to identify markers for these organic materials [74].

In the thermal analysis of acrylonitrile polymerization and cyclization in the presence of *N*,*N*-dimethyl formamide, Aviles et al. identified occluded molecules of DMF and several compounds containing nitrogen that were produced during the thermal degradation [75].

The combination of Py–GC–MS and TGA was employed by Nishida et al. to study the initial thermal degradation process for poly(*p*-dioxanone) [76].

TGA–MS and Py–GC–MS were used by Jakab and Blazsó to study the effect of carbon black on the thermal decomposition of vinyl polymers in an inert atmosphere [77].

Focarete et al. used TGA–MS along with several other analytical techniques to study the solid-state properties of polylactide, poly(lactide-*co*-glycolide) and blends of these polymers with atactic poly(3-hydroxybutyrate) [78].

Boeker et al. used TGA and thermal desorption mass spectrometry to study the thermal degradation of block copolymers to which perfluoroester, fluorinated urethane or fluorinated carbonate side chains had been attached to the polymer pendant hydroxy groups [79].

Fazlioglu and Hacaloglu studied the thermal decomposition of glycidyl azide polymer using the direct insertion probe inlet [80].

Badawy and Dessouki were able to determine that the pyrolysis products from polymer produced by radiation polymerization of acrylonitrile in a viscous system with styrene at ambient temperature corresponded to oligonitriles with styrene end groups [81].

Cox et al. studied the thermal degradation of ethylene–methyl acrylate copolymers and blends of this copolymer with polyethylene or poly(methyl acrylate) homopolymers to determine the feasibility of quantifying the monomer composition of the copolymer [82].

Because of its use in the solid-phase extraction of aluminium, a poly(styrene-*co*-divinylbenzene) commercial resin (Amberlite XAD-4) was modified, and the different steps of the modification were characterized by Py–GC–MS hyphenated techniques [83].

The thermal decomposition chemistry of nine different polycyanurates was studied by thermogravimetry and analysis of the gases evolved during pyrolysis using infrared spectroscopy and gas chromatography–mass spectrometry [84].

Zn(II)–Cr(III)–layered double hydroxides (LDHs) containing sulfated or sulfonated surfactants as the interlamellar anion were studied by a set of analysis methods, included TG–MS; from the results obtained it was possible to conclude that, in an air atmosphere, the anions decomposed by a partial combustion, leading to the formation of sulfide [85]. ZnS was studied because of its flame retardant effect in plasticized poly(vinyl chloride) materials, and a detailed analysis of the decomposition products was obtained using both TG–IR and TG–MS [86].

Mullens et al. reported how the combination of TGA–evolved gas analysis (TGA–EGA) (i.e. mass spectrometry and Fourier transform infrared spectroscopy on-line coupled to TGA) gives important informations for an identification of the intermediates that are formed during the thermal decomposition of promising multimetal oxide ceramic materials, such as superconducting cuprates and ferroelectric materials [87].

Thermal decomposition of cerium(III) acetate hydrate, $Ce(CH_3CO_2)_3\cdot1.5H_2O$, to cerium(IV) oxide, CeO_2, in helium has been successfully investigated by sample-controlled thermogravimetry combined with evolved gas analysis by mass spectrometry (SCTG–MS) [88]. The thermal behavior of four new zinc(II) aliphatic carboxylate complexes was studied by thermal analysis and the gaseous intermediates and products of the thermal decomposition were determined by MS–EGA [89].

The thermal decomposition of some aminotetrazoles has been studied by thermoanalytical techniques, including evolved gas analysis to determine the decomposition mechanism [90].

The thermal decomposition of borazane BH_3NH_3 in the temperature range up to 500 K has been studied by differential scanning calorimetry (DSC) and thermogravimetry combined with the FTIR spectroscopic and mass spectrometric analysis of the gas phase [91].

Sol–gel derived biodegradable SiO_2 gel fibers, with incorporated an active component, dexmedetomidine hydrochloride, have been prepared and characterized by Raman spectroscopy, SEM, ^{29}Si MAS NMR and TG–MS, respectively [92].

The thermochemical behavior of betaine and betaine monohydrate was investigated under two degradation conditions, in air and nitrogen flows, and the evolved gas was analyzed by both the combined TG–FTIR system and by gas chromatography using mass-selective detection (Py–GC–MSD) [93].

The solid state thermal behavior of cobalt(III) thiocyanate complexes has been studied using thermoanalytical techniques, infrared spectroscopy and pyrolysis coupled to both infrared and mass spectrometry, Py/FTIR and Py–MS, to describe the decarbonylation of the equatorial amide ligand [94].

Scandium dipivaloyl methanate, as a volatile precursor for thin film deposition, was investigated by TGA–MS [95].

The degradation mechanism of some aromatic polyethers containing flexible spacers was proposed by Hurduc et al. by using mass spectrometry as evolved gas analysis [96].

By GC–MS and TGA–FTIR techniques, the thermal decomposition processes in polybenzoxazine model dimmers was investigated: the 2,4-dimethylphenol-based benzoxazine dimers degraded into smaller and highly volatile compounds, leaving no char at the end of degradation, while the *p*-cresol-based benzoxazine dimers degraded into smaller and highly volatile products as well, nevertheless some of which are able to undergo cross-linking and aromatization processes and form char [97].

The thermal degradation of two different thermoplastic polyurethane elastomers (TPU) based on 4,4′-MDI was studied using two different thermal analyzers with combined techniques for the evolved gas analysis, and two combustion devices [98].

The thermoanalytical characterization of hydride and phenol groups chemisorbed at the surface of Aerosil 300 was reported by Braun et al. by the TGA–MS coupled analysis [99].

The thermal behavior of the epoxy and polyester powder coatings was studied by using thermogravimetry/differential thermal analysis coupled with gas chromatography/mass spectrometry technique for the identification of the degradation products [100].

3.2. *Other applications*

Factor analysis was used for extracting informations out of the mixture mass spectra recorded in a thermogravimetric–mass spectrometric analysis. Principal component analysis and a special diagram, the contour variance diagram (ContVarDia), were used for performing the factor analysis [101].

The combustion of pyrolyzed sewage sludges has also been studied, related to the possibility of combining combustion with pyrolysis when trying to valorize sludges by producing active carbon. From the burning probe data it has been possible to observe that a rapid devolatilization occurs during sludge combustion compared to coal and previously pyrolyzed sludge [102].

The non-oxidative thermal degradation chemistry of alkyl and aryl quaternary phosphonium-modified montmorillonites (P-MMT) was examined using TGA combined with pyrolysis–GC–MS [103].

Turel, in his review, cited the evolved gas analysis among the techniques to study the interactions of metal ions with quinolone antibacterial agents [104].

The use of a mass spectrometer linked to the TG gave additional data about the devolatilization process [105], yielding information about the evolution of different volatile products and about possible chemical reactions occurring during the formation of mesophase spherules from a low-temperature coal tar pitch under carbonization conditions [106] and about an advanced demineralization procedure on the combustion characteristics of coal [107].

An investigation involving spectroscopy and TGA–MS was reported by Van Craenenbroeck et al. to prove the activity of Au–V-based catalysts in the catalytic removal of diesel soot particulates [108].

Giron et al. reported the solid state characterizations of pharmaceutical hydrates [109] and collected the applications of thermal analysis, including EGA by mass spectrometry, in the pharmaceutical industry [110], while Kök reviewed the applications of thermal analysis in fossil fuel science [111].

TG–MS and TG–FTIR evolved gas analysis techniques were applied for an unambiguous thermal analysis of intumescent coatings [112].

4. 2003 applications

4.1. Polymers and inorganics

The complex interactions of Pd and O_2 in catalytic oxidation reactors have been characterized by a detailed mechanism which captures the bulk and surface hysteresis of Pd oxidation/reduction, as observed in TGA–MS experiments [113].

The temperature-programmed decomposition of $C_{60}Br_{24}$ and $C_{60}Br_6$ in the temperature range 323–523 K revealed multiple decomposition routes using evolved gas analysis and mass spectrometry (EGA–MS) [114].

Complexes with pyrazole-based ligands, studied by DSC and TG–MS, have been characterized and a low stability intermediate was observed in the thermal decomposition [115].

Magnesium alanate was investigated by thermogravimetry and mass spectrometry of the evolved gas to evacuate its use for a reversible hydrogen storage. Thermal analysis showed a decomposition with a release of hydrogen proceeding in two major steps [116].

Pyrolysis followed by on-line mass spectrometry has been applied for the analytical characterization of acrylic acid polymers [117], of poly-pyrrole–ethylvinylether polymers [118], of the monomers ratios in random and block copolymers of ethylene and propylene [119], of flame-retarded high-impact polystyrene [120], diterpenic resins [121], of model uronic acids of pectin and hemicelluloses [122], of the effect of tetramethylammonium hydroxide on the slow pyrolysis of cellulose [123], of biomatrix materials [124], of the quantification of paper additives [125], of quaternary hydroxides as alkylating reagents for aquatic fulvis acid [126], of chlorinated natural rubber [127] and of alkyne-containing polystyrenes [128]. Flame-retardant rubber was also characterized by high-resolution thermogravimetry coupled to mass spectrometry [129].

The thermal degradation kinetics of poly(3-hydroxybutyrate) (PHB) and poly(3-hydroxybutyrate-*co*-3-hydroxyvalerate) [poly(HB–HV)] under nitrogen was studied by both TG–FTIR and Py–GC–MS [130].

Hibshman et al. studied the gas separation effects of annealing polyimide–organosilicate hybrid membranes by coupled TGA–MS evolved gas analysis [131].

Mn(II), Co(II), Ni(II) and Cu(II) complexes with 4,4′-bipyridine and dichloroacetates were characterized by TGA–MS coupled techniques [132]. Evolved gas analysis allowed the characterization of the thermal decomposition of Cr(III) nitrate nanohydrate [133] and the phase transitions and thermal effects occurring during annealing in air of material with general formula CrO_x with $x \geq 2.4$ [134].

Thermal analysis–mass spectrometry, by means of a home-assembled instrumental interfaces, was performed in order to quantify the organic component present in a homogeneous TiO_2 gel sample and its release during pyrolysis [135,136].

The decomposition pathway of extremely nitrogen-rich tetrazine was investigated by Loebbecke et al. by either a TG/mass spectrometer system (Balzer Thermostar) or a self-constructed fast infrared spectroscopic EGA equipment based on a heatable optical cell adapted to a rapid scan FTIR spectrometer (Nicolet 60 SX) which allows an on-line monitoring of the gas phase [137].

Py–GC–MSD allowed to study the formation of the main degradation compounds from arabinose, xylose, mannose and arabitinol during the pyrolysis process [138].

The thermal decomposition behavior of poly(propylene carbonate)s (PPC)s synthesized with varying molecular weights was studied at various pyrolysis temperatures by the combination of pyrolysis–gas chromatography/mass spectrometry and thermogravimetric analysis/infrared spectrometry (TG–IR) techniques [139].

TG–MS and TG–GC–MS were employed to evaluate the thermal degradation characteristics of isomeric poly(styrene)s [140,141].

Dakka showed the role of the oxidative environment [142] and of the particle size [143] in the degradation of poly(methyl methacrylate) by coupled TG–DTA–MS. Di Maggio et al. studied the physico-chemical characteristics of hybrid polymers obtained by 2-hydroxyethyl(metacrylate) and alkoxides of zirconium [144].

Mass spectrometry EGA and X-ray diffraction measurements have been used to study the reaction between uranyl nitrate hexahydrate and strontium nitrate. The results confirmed the absence of a direct interaction between the two compounds [145].

Dichlorophenoxy-acetic and propionic acid complexes with Ni(II) and Co(II) were studied by Ptaszynski and Zwolinska [146], 2,2′-bipyridine and trichloroacetates complexes with Mn(II), Co(II) and Ni(II) were characterized by Czakis-Sulikowska and Czylkowska [147], zinc di-hydroxybenzoate complexes with imidazoles and its derivatives were studied by Olczak-Kobza et al. [148], complexes with pyrazol-based ligands were described by Mészáros Szecsenyi et al. [149], new polymorphic copper(I) coordination compounds with 2-ethylpyrazine were studied by Näther et al. [150], while metallic iron-organic coordination compounds were characterized by Lu et al. by the coupled TG–DSC–MS technique [151].

Hardy et al. proposed a study of the decomposition of an aqueous metal-chelate gel precursor for $(Bi, La)_4Ti_3O_{12}$ by means of TGA–FTIR, TGA–MS and HT-DRIFT [152].

Sonochemical synthesis followed by supercritical drying was used to prepare MgO nanoparticles from $Mg(OCH_3)_2$ and $Mg(OC_2H_5)_2$. Their physico-chemical properties were examined by evolved gas analysis [153].

The thermal behavior of Ni–Mn layered double hydroxide and the characterization of formed oxides was studied by a TG–DTA–MS hyphenated system [154].

The thermal decomposition of zinc acetate dihydrate $Zn(CH_3CO_2)_2{\cdot}2H_2O$ in some humidity-controlled atmospheres has been successfully investigated by novel thermal analyses, which are sample-controlled thermogravimetry and thermogravimetry combined with evolved gas analysis using mass spectrometry [155]. Copper nitrate thermal decomposition was investigated at reduced pressures by Morozov et al. [156].

A combination of differential scanning calorimetry and high-resolution DTG coupled to a gas evolution mass spectrometer has been used to study the thermal activation of copper oxide based upon the copper hydrocalcite $Cu_xZn_{6-x}Al_2(OH)_{16}(CO_3)$ tetra hydrated [157].

The thermal change due to the formation of indium oxide thin films fabricated by a dip-coating process using indium 2-ethylhexanoate monohydroxide was investigated by TG–DTA–MS [158].

Ceria- and titania-supported chromia catalysts were prepared by wet impregnation technique and their thermal stability as well as redox behavior was examined by mass spectrometry evolved gas analysis [159].

4.2. Other applications

A mixture of carbon compounds was analyzed by temperature-programmed combustion tests under 20% oxygen in argon in a TGA linked to a mass spectrometer (TGA–MS) to compare the reactivity of coal and synthetic coal models [160].

Oxygen reduction catalysts for PME fuel cells on the basis of carbon-supported, heat-treated iron phenanthroline complexes have been investigated with thermogravimetry coupled to mass spectrometry, showing that the catalyst precursor is completely embedded into the carbon [161].

Chemometric methods were used for extracting information out of the mixture mass spectra recorded in a thermogravimetric–mass spectrometric analysis. Principal component analysis and discriminant rotation (DR) were applied for studying the effects of fire retardants on the gases evolved during thermal degradation of cellulose [162].

Pyrolysis followed by on-line mass spectrometry has been applied for the analytical characterization of different types of speck impurities present in bleached eucalypt kraft pulps [163], of low rank perhydrous coals [164] and of solvent and thermal extractable material from perhydrous vitrinites [165]. Smieja-Krol reported the analysis of organic matter from Witwatersrand Basin, South Africa, by Oxyreactive Thermal Analysis (OTA) and pyrolysis–gas chromatography–mass spectrometry [166].

Low-temperature pyrolysis is evaluated as a possible technique for the disposal of chromated copper arsenate treated wood waste. Theoretical and experimental studies, performed in order to gain more insight in the mechanism of arsenic release, are brought together in order to present the current state of knowledge [167].

Hyphenated techniques of thermal analysis (including TGA–MS) were proposed by Turcotte et al. for the thermal analysis of black powder [168] and by Dell'Abate et al. for the characterization of soil humic substances [169].

Thermal analysis coupled with mass spectrometry (TA–MS) was applied to the pyrolysis of Chinese coals with different ranks, with a total of 13 investigated Chinese coals [170].

Hatton and Southward described the optimization of the connection between TA–MS systems by a hot zone inlet system and in situ probe both of which provide fast response, zero dead volume sampling directly to the MS [171].

By TGA–MS, Thomas et al. characterized the oxidation products of pyrite [172], while both TGA–MS and TGA–FTIR allowed the thermal characterization of toresemide [173].

The thermal behavior in air of two Al nanopowders, Alss and Alsstef, a Teflon coated version of Alss, was determined using the coupled TG–DTA–FTIR–MS technique [174].

The dehydration of synthetic and natural vivianite $((Fe^{2+})_3(PO_4)_2{\cdot}8H_2O)$ were determined using a high-resolution thermal analyzer coupled to a mass spectrometer [175].

A combination of high-resolution thermogravimetric analysis (HRTGA) coupled to a gas evolution mass spectrometer combined with infrared emission spectroscopy has been used to study the thermal decomposition of two Australian hydrotalcites, carrboydite and hydrohonessite [176].

The thermal stability and reactivity of adsorbed pyridine on sulfated zirconia and Pt/sulfated zirconia were studied by temperature-programmed desorption (TPD) coupled with infrared spectroscopic and mass spectrometric analyses [177].

5. 2004 applications

5.1. Polymers and inorganics

DSC–TG–MS and spectroscopic techniques were used to characterize the coated nanoscale Cu/SiC_p composite particles [178].

CsF supported by α- and γ-alumina was characterized by DTG–DTA coupled with mass spectral analysis of the gases evolved by the solid [179].

Bismuth vanadate, nominally $BiVO_4$, is used as a yellow pigment in the ceramic and plastic industries. It can be prepared by heating stoichiometric quantities of Bi and V precursors, but brighter colours are obtained by an aqueous preparation route. Since either a zircon- or scheelite-structured phase may be obtained, thermal analysis, coupled with effluent gas analysis of the heated zircon-structured solid, disclosed complex phase transitions in the range 200–450 °C [180].

The thermal and biological properties of diopside, $CaMgSi_2O_6$, prepared by a sol–gel process using a metal alkoxide and metal salts were investigated by TGA–MS for its applicability as a biomaterial [181,182].

A series of segmented polyurethanes based on 4,4′-diphenylmethane diisocyanate (MDI), polyoxypropylenediol (POPD) and low-molecular chain extenders (1,2-propanediol (PD) or

3-chloro-1,2-propanediol (CPD)), obtained by one-step polyaddition, was investigated by thermogravimetric analysis coupled with Fourier transform infrared spectroscopy or mass spectrometry [183].

The thermal degradation of the P(CL-*block*-PDX) block copolymers was investigated by TGA simultaneously coupled to a FTIR spectrometer and a mass spectrometer for evolved gas analysis [184]. The flame retardant mechanisms of red phosphorus, magnesium hydroxide and red phosphorus combined with magnesium hydroxide were studied in high impact polystyrene by means of thermogravimetry coupled with Fourier transform infrared spectroscopy and mass spectrometry [185].

Studies by TGA–MS reported new polymeric precursors suitable to form boron containing silicon oxycarbide glass [186], the effect of humidity on the thermal decomposition of terephtalate polyester [187], the thermochemical properties of 18-crown-6 ether complexes with aralkylammonium perchlorates [188], of cobalt and nickel lactate and bypyridine-lactato complexes [189] and the thermal and thermo-oxidative degradation of engineering thermoplastics and their life estimation [190].

To circumvent the detrimental effect of the lower thermal stability of alkyl ammonium-treated montmorillonite, a series of alkyl-imidazolium molten salts were prepared and characterized by elemental analysis, thermogravimetry and thermal desorption mass spectroscopy (TDMS) [191]. Copper was adsorbed onto Ca-exchanged montmorillonite (Cheto clay) under basic conditions, and differential thermogravimetric analysis (DTGA) combined with mass spectroscopy was employed as the principal technology to study the distributions and structures of adsorbed copper species on Cheto clay [192]. EGA by mass spectrometry was also employed to study the thermal decomposition of syngenite, $K_2Ca(SO_4)_2 \cdot H_2O$, formed during the treatment of liquid manure [193].

Tin-doped indium oxide transparent conducting films, fabricated on silicon substrates by a dip coating process, were characterized by temperature-programmed desorption or thermal desorption spectroscopy (TDS) in high vacuum; gas evolution from the ITO film mainly consisted of water vapor [194]. By TG–DTA and EGA–MS techniques, new compounds synthesized by the decomposition of metal nitrate–tartarate complex [195] and hydrothermally synthesized powders of $BaTiO_3$ and its Fe- or Nd-doped analogues [196] were characterized by mass spectrometry evolved gas analysis.

The mixed-valence cluster $[Cu_{20}Se_{13}(PEt_3)_{12}]$, synthesized and characterized by TGA–MS evolved gas analysis, showed that in addition to the labile phosphane ligands, selenium atoms are cleaved as $SePEt_3$ during the consolidation process [197].

Ytterbium nitrate was used as a parent compound for the formation of Yb_2O_3 at up to 800 °C in atmosphere of air, and the thermal processes occurring during the decomposition course were monitored by means of TG–GC–MS coupled techniques [198].

The gaseous products of the TG decomposition of $[Mg(NH_3)_6](NO_3)_2$, $[Ni(NH_3)_6](NO_3)_2$ and $[Ni(ND_3)_6](NO_3)_2$ were on-line identified by a quadruple mass spectrometer (QMS) [199].

A new hydroxylammonium fluorogermanate has been synthesized and its thermal decomposition was studied by thermal analysis coupled to mass spectrometry [200].

TGA coupled to MS studies suggested that volatile, titanium hexanuclear species may be formed between 423 and 553 K in the decomposition of a new titanium hexanuclear oxo carboxylato alkoxide complex [201] and described the pyrolysis behavior of sol–gel derived TiO_2 powders [202,203]. Commercially available sols of amorphous TiO_2 and anatase particle of nano-size, precipitated and dried carefully, have been studied by simultaneous thermogravimetric and differential thermal analysis (TG–DTA) coupled with on-line evolved gas analysis methods in flowing air and helium atmosphere in order to check and model pyrolytic processes taking place at elevate temperature ranges of anatase film formations by spray pyrolysis deposition (SPD) [204].

The thermal characterization of strontium-tetrahydrogen-hexaoxoperiodate-trihydrate, $Sr(H_4IO_6)_2 \cdot 3H_2O$, showed that the dehydration and the decomposition of the compound takes place via an unknown metaperiodate $Sr(IO_4)_2$, results at 265 °C in the formation of $Sr(IO_3)_2$ in an exothermic reaction and finally $Sr_5(IO_6)_2$ is formed in a disproportionation reaction [205]. The conditions under which compounds of the commercial herbicides with lead(II) and cadmium(II) are formed and the results of the examination of their properties were discussed [206].

By evolved gas analysis, hydrogen evolution from borohydride reduced electroless nickel deposits was monitored [207], the properties of four new mixed ligand complexes prepared by the reaction of title metal dichloroacetates and 2,4′-bipyridine [208] and of lanthanide hexacyanochromate(III) [209], chromite [210] and hexamethylenetetramine [211] complexes were shown. Comparative studies (TG–FTIR and TG–DTA–MS) were reported for the thermal decomposition of ammonium paratungstate tetrahydrate in air [212,213], thiourea-rich complexes of tin chloride [214], cadmium acetate [215] and crystals of unknown origin, crystallizing spontaneously from ethylenediamine on standing [216].

Evolved gas analysis by pyrolysis coupled to mass spectrometry was applied for the characterization of red phosphorus [217]. Kamruddin et al. showed the properties of nanocrystalline ceria by several techniques, including TG-EGA–MS [218], and the surface nitridation of pure Ti and Cr was carried out by exposing them to ammonia atmosphere at optimum temperatures in a thermogravimetric analyzer–mass spectrometer system [219].

High-resolution thermogravimetric analysis combined with evolved gas mass spectrometry allowed to study the natural magnesium oxalate dihydrate, glushinskite [220].

Balek et al. described the results of a study of the preparation of ruthenia-based catalytic materials by heating their precursors [221].

New solid complexes of a herbicide known as dicamba (3,6-dichloro-2-methoxybenzoic acid) with Pb(II), Cd(II), Cu(II) and Hg(II) were characterized by TG–DTA–MS on-line EGA [222].

Dakka showed the effect of heating rate on the rate of production of evolved gases for poly(methyl methacrylate) [223].

A coupled TG–MS system was used to analyze the principal volatile thermal decomposition and fragmentation products of d-metal complexes with 4,4′-BPY and propionates under dynamic air and argon atmosphere [224].

The formation mechanism of thermally prepared IrO_2/SnO_2 [225] and IrO_2/Ta_2O_5 thin films [226] has been investigated under in situ conditions by thermogravimetry combined with mass spectrometry.

5.2. Other applications

A thermogravimetry–gas chromatography–mass spectrometry (TG–GC–MS) coupling was developed in order to analyze complex gaseous mixtures which are evolved during degradation process of materials. A self-contained interface has been built and tested with different compounds as hydrocarbon products, copolymers and gaseous mixtures from urban landfill [227].

The results of the use of mass spectrometry together with a thermogravimetric analysis system, used to study the thermal conversion processes of two kinds of sewage sludge under different atmospheres simulating pyrolysis, gasification and combustion, indicated that gasification is the best process for some kinds of sludge while a co-combustion process is more suitable for other [228].

The influence of thermal process for indium hydroxoformate, used as one of the precursor material of ITO transparent conducting films, has been successfully investigated in some controlled atmospheres by unique thermal analyses equipped with a humidity generator, which are thermogravimetry–differential thermal analysis, thermogravimetry in conjunction with evolved gas analysis using mass spectrometry [229].

The design, the abilities and a characteristic application of an in-house made interface for combining thermogravimetry with mass spectrometry were presented by Pappa et al. [230].

Éhen et al. proposed the thermal characterization of hair by TG–MS combined thermoanalytical technique [231].

The thermal behavior of BisGMA/TEGDMA-based dental resins of various degrees of conversion was examined using differential scanning calorimetry, thermogravimetric–gas chromatography mass spectrometry and thermogravimetric–mass spectrometry [232].

Monitoring the products of the thermal fragmentation of parent β-cyclodextrin inclusion complexes of Thymol and Lippa sidoides Cham essential oil extract, applying TG–MS combined technique, provided evidence concerning the inclusion complex formation, and besides, gave selective signal to follow the decomposition of the cyclodextrin inclusion complexes [233].

The effect of liquid-phase oxidation on the texture and surface properties of carbon nanofibers has been studied using spectroscopic and TGA–MS techniques to show that the most predominant effect that gives rise to these textural modifications is the opening of the inner tubes of the fibers [234].

Four different artificial aerosol particles as well as one standard environmental aerosol particle (NIST SRM 1649a) were investigated by TG–MS [235].

Functionalized SH-MCM-41 materials have been prepared via grafting and co-condensation and a detailed thermogravimetric–mass spectrometry study of SH-MCM-41 and the correspondent acid form SO_3H-MCM-41 materials has been carried out to show the influence of the oxidation procedure in the final sulfur species [236].

A new poly(fullerene oxide) thin film material has been fabricated by thermal activation and electron bombardment on hexanitro[60]fullerene film deposited on a Au substrate, all under vacuum conditions, and the reaction products in the polymerization process are analyzed by TGA–MS and LDI–MS techniques [237].

Giron et al. summarize in their article the different steps needed for a proper design and monitoring of the solid-state in pharmaceutical industry in order to fulfill the requirements of the guideline dealing with polymorphism of the International Conference of Harmonization [238].

High-resolution thermogravimetry coupled to evolved gas–mass spectrometry showed that the release of carbon dioxide, but not of water, indicates that the moolooite is the anhydrous copper(II) oxalate as compared to the synthetic compound which is the dihydrate [239].

Influences of the controlled decomposition rate and controlled residual pressure on the thermal decomposition process of synthetic bayerite were investigated by applying a method of controlled rate evolved gas detection (CREGD) [240].

The results of TG and TG–MS measurements on a co-continuous nanostructure composed of phenolic resin rich phase and PMMA rich phase, obtained in the curing process of miscible blends of phenolic resin and poly(methyl-methacrylate) suggested that, whether or not the nanoporous structures are retained, is related to the main products of the thermal decomposition of each phenolic resin [241].

The raw Yima (YM) and Datong (DT) coal, their demineralized (YM-ash and DT-ash) and de-pyrite (YM-p and DT-p) coals were pyrolyzed in a fluidized-bed reactor to examine the sulfur removal efficiency. The effect of process parameters such as temperature, residence time and atmosphere was investigated by TGA–MS [242]. Different ignition behavior was observed for several coals studied; the sub-bituminous and low volatile bituminous coals ignited heterogeneously, while homogeneous ignition occurred for the high volatile bituminous coal [243].

The thermal decomposition behavior of Cyagard, a fire retardant additive, was studied by GC–MS evolved gas analysis coupled to a thermogravimetric analyzer [244]. The effects of three retardants on the thermal decomposition of wood-derived rayon fiber have been investigated by thermogravimetry–mass spectrometry analysis [245].

The complex reactions of biomass pyrolysis and the evolution of different volatile species can be well represented by a Distributed Activation Energy Model (DAEM) and the model required kinetic parameters as inputs were obtained from TG–FTIR or TG–MS analysis of the sample [246]. TG–FTIR and TG–MS were together employed to study the thermal degradation of PVC cable insulation [247] and of dichlorobis-thiourea-zinc [248] and dichloro-thiourea-tin compounds [249].

A simple method to estimate the fuel ratio as a combustion index of char obtained from the pyrolysis of municipal waste by a waste disposal facility was developed, based on the data for the pyrolysis of cellulose, polyethylene, polypropylene, polystyrene and poly(vinyl chloride); pyrolysis reactions involved were explained by thermal analysis (TG–MS) [250]. Carbon nanofiber-supported platinum catalysts were studied to establish the influence of the support surface composition on their performance in the liquid-phase hydrogenation of cinnamaldehyde; by acid–base titration, XPS and TGA–MS, the number of oxygen-containing surface groups was determined in oxidized carbon nanofiber samples and in platinum catalysts [251]. Titania-supported vanadia catalysts were prepared by a wet impregnation technique and their thermal behavior, morphology as well as redox properties, were examined by thermal analysis methods like differential scanning calorimetry and temperature-programmed–evolved gas analysis with mass spectroscopy (EGA–MS) [252].

Evolved gas analysis by pyrolysis coupled to mass spectrometry was applied for the characterization of products that may interfere with the forensic analysis of fire debris [253], of the gas phase of synthetic lubricants [254], of tobacco ingredients [255], of vegetable oils from Macuba fruit [256] and of representative samples of acrylic emulsion paints for artists [257].

Thermogravimetry, in situ diffuse reflectance Fourier transform infrared spectroscopy (DRIFT) and on-line mass spectrometry were combined to study the effect of O-methylation and O-acetylation on the hydrogen bonds (HBs) in coal [258]. The characterization of model compounds and a synthetic coal by TG–MS/FTIR to represent the pyrolysis behavior of coal was proposed by Arenillas et al. [259]. Ding and Frost reported a study of copper adsorption on montmorillonites using thermal analysis methods [260].

Three bio-fuels with or without additives and their fly ash samples were characterized using simultaneous thermogravimetry–differential thermal analysis–Fourier transform infrared spectrometry–mass spectrometry [261].

The thermal decomposition of natural iowaite [262] and whewellite [263] was studied by using a combination of thermogravimetry and evolved gas mass spectrometry. The thermal decomposition of the ammonium-exchanged vermiculite was shown to take place in three steps, with a strong correlation between the mass loss of ammonia and water, thus suggesting a simultaneous release [264].

High-resolution thermogravimetric analysis coupled with a gas evolution mass spectrometer was used to study the decomposition of Peisleyite, a hydrated hydroxylated multianion mineral [265], and the decomposition of metazeunerite [266], and both showed a thermal decomposition in five stages.

The thermal stability of fluorinated silica gels at various fluorine contents was investigated by coupling thermogravimetric measurements with mass spectrometric and gas chromatographic analyses of the evolved gaseous species [267].

Thermal analysis of non-isothermal and isothermal oxidation of the Tallium carbonitride solid solution powder was performed using simultaneous TG–DTA–MS technique [268].

A combination of high-resolution thermogravimetric analysis coupled to a gas evolution mass spectrometer combined with infrared emission spectroscopy has been used to study the thermal decomposition of synthetic hydrotalcites honessite and mountkeithite and the cationic mixtures of the two minerals [269].

The identification and quantification of decomposition products of tetrabromobisphenol A (TBBA) was based on GC–MS on-line coupled to a thermogravimetric analyzer [270].

The efficiency of fining agents is determined by the temperature range in which fining gas is released, and the evolution of fining gas can be quantitatively measured by means of evolved gas analysis using on-line coupled mass spectrometry [271].

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