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TG-FTIR study on thermal degradation in air of some new diazoaminoderivatives

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Abstract The study on the thermal behavior of some new diazoaminoderivatives was aimed to follow the structure-thermal stability-degradation mechanism correlation by means of the TG-FTIR technique and formation enthalpies. The TG-DTG-DTA curves reveal the thermal degradation in air (30–900 °C) to show two ranges as a function of temperature (time), where the gaseous species resulting by degradation are eliminated: the first, an endothermic one which is identical to that under nitrogen atmosphere and the second, an exothermal one. As made evident by the identification of the individual gaseous species by their characteristic absorbances as well as those obtained by TG-FTIR the compounds C_2H_2, $H_2C=NH$, SO_2, NH_3, CO_2, H_2O, HCl are eliminated in the first domain while CO_2, SO_2, H_2O in the second, which afforded the advancement of the most probable degradation mechanism.

Keywords Diazoaminoderivatives · Degradation mechanism · TG-FTIR · Thermal degradation · Thermal stability

Introduction

The diazoaminoderivatives are known to belong to a group of compounds of a particularly high biological potential [1] included in the class of the phenoxy-alkancarboxylic acids sulphonamides [2–5]. The sulphonamidic group has a favorable influence on their selectivity that results in various activities as growing stimulators, herbicides, fungicides, acaricides and also as pharmaceuticals [6–10].

In recent years the researches on the obtaining of new biologically active compounds have also been extended over the sulphonamidated aryloxyalkyl carboxylic derivatives with potential antitumor, antioxidant or anti-inflammatory actions, with applications in treatments of the ulcer, convulsions, diabetes [11–16]; apart from this, the potential herbicide action deserves mention [17–20].

Going further with our studies in this field, regarding the thermal behavior under nitrogen atmosphere of some new complexes of the sulphonamidated aryloxyalkyl-carboxylic derivatives with transitional metals [21], diazoaminoderivatives [22], azomethines [23], in the present paper the structure-thermal stability–degradation mechanism correlation was followed with four new diazoaminoderivatives by means of TG-FTIR analysis and enthalpies of formation, namely: 1-(3-{2-[4-(aminosulphonyl)-2-methylphenoxy]ethyl}triaz-1-enyl)-2-naphthol **(a)**, 1-(3-{2-[2-(aminosulphonyl)-4-chloro-phenoxy]ethyl}triaz-1-enyl)-2-naphthol **(b)**, 1-(3-{2-[2-(aminosulphonyl)-2-chloro-phenoxy]ethyl}triaz-1-enyl)-2-naphthol **(c)**, 1-(3-{2-[2-(aminosulphonyl)-2-chloro-phenoxy]ethyl}triaz-1-enyl)-1-Schaffer acid **(d).**

The analysis of the TG-DTG-DTA curves reveals the thermal degradation in air (30–900 °C) to show two domains as a function of temperature (time): an endothermic one which is identical to that for the degradation in nitrogen atmosphere [22] and an exothermal one, these

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domains including also the release of the gaseous species resulting by degradation. The identification of the individual gaseous species by their characteristic absorbances [24] obtained by TG-FTIR revealed the compounds C_2H_2, $H_2C = NH$, SO_2, NH_3, CO_2, H_2O, HCl to be released within the endothermic domain and CO_2, SO_2, H_2O within the exothermal domain.

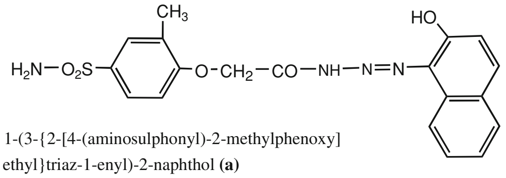
The obtained results were indicative of a good correlation of the chemical structure with the thermal stability resulting from the initial degradation temperatures from DTG and from the formation enthalpies estimated from the combustion enthalpies [25] and also with the degradation mechanism on the basis of the TG-FTIR analysis [26–30].

Since the TG-DTG-DTA thermal methods applied along with FTIR [31] or with mass spectroscopy [32] were found to be very efficient for completing the thermal analysis data in studying the degradation mechanisms [33] we made use of them in the present paper and the obtained results afforded the elucidation of the structure-thermal stability-degradation mechanism correlations for the compounds under study.

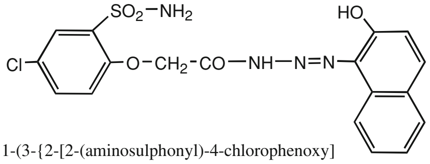
By taking the practical significance of these compounds as biologically active products into account the present paper makes evident the temperature range proper for their stability, use and storage. At the same time, the degradation mechanism affords useful information regarding the environmental impact when the initial degradation temperature is exceeded by the processing processes.

Experimental

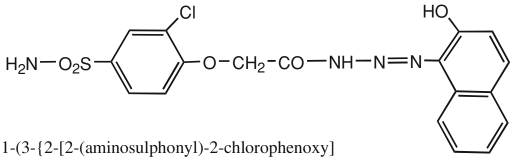
The chemical structure of the compounds under study, their formulae, IUPAC names, molecular weights and melting points measured by the Boetius method are given in Scheme 1.



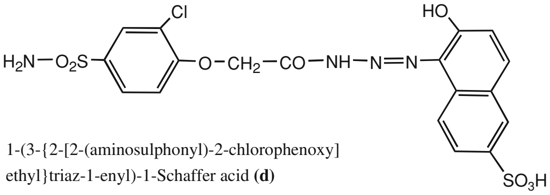
1-(3-{2-[4-(aminosulphonyl)-2-methylphenoxy] ethyl}triaz-1-enyl)-2-naphthol **(a)**
Chemical formula: $C_{19}H_{18}N_4O_5S$
Molecular weight: 414
Melting point: 130-132°C



1-(3-{2-[2-(aminosulphonyl)-4-chlorophenoxy] ethyl}triaz-1-enyl)-2-naphthol **(b)**
Chemical formula: $C_{18}H_{15}N_4O_5SCl$
Molecular weight: 434.5
Melting point: 110-115°C



1-(3-{2-[2-(aminosulphonyl)-2-chlorophenoxy] ethyl}triaz-1-enyl)-2-naphthol **(c)**
Chemical formula: $C_{18}H_{15}N_4O_5SCl$
Molecular weight: 434.5
Melting point: 168-170°C



1-(3-{2-[2-(aminosulphonyl)-2-chlorophenoxy] ethyl}triaz-1-enyl)-1-Schaffer acid **(d)**
Chemical formula: $C_{18}H_{15}N_4O_8S_2Cl$
Molecular weight: 514.5
Melting point: 195-198 °C

Scheme 1 Samples under study (structure, molecular weight and melting points)

Methods

TG-FTIR

The TG-FTIR analyzer consists of a TG/DTA Diamond (Perkin Elmer) thermo-balance and a FTIR spectrometer, Spectrum 100 (Perkin Elmer), provided with a TG-FTIR (Perkin Elmer) gas transfer accessory and a gas cell of 100 mm length and KBr windows, heated at 150 °C. The FTIR spectra were recorded within the 700–4,000 cm^{-1} range at a resolution of 4 cm^{-1} and scanning rate of 200 cm/s, a single spectrum being recorded every 15 s by means of the Spectrum Time Bose Perkin Elmer program. A G7 gas analyzer (Dominic Hunter) supplies the dry air (pearl point: –50 °C) entering the TG/DTA analyzer at a flow rate of 100 mL/min as well as the nitrogen for purging the analysis room of the FTIR spectrophotometer. The analysis was run with 10 mg sample placed into a platinum crucible, at a heating rate of 10 K/min within the 30–900 °C temperature range.

The gaseous species resulting by the thermal degradation of the samples were identified by means of the standard IR spectra.

Berthelot calorimeter

The formation enthalpies of the samples were estimated from the combustion enthalpies [25] by using a Berthelot calorimeter (calorimetric bomb), the VEB (AB) Apparatus bau Potsdam–Bahelsherg (Germany) model. The temperature variation during the sample combustion was measured by means of a Bekman thermometer of a 0.01 °C precision. The calorimeter standardization was made by the combustion of a known amount of benzoic acid $\left(\Delta_{c}H^{o}_{298} = 3.2257 \times 10^{6}\,\mathrm{J/mol}\right)$. The sample amount submitted to this analysis was of ≈ 0.2 g, burned iron thread ≈ 0.005 g, $\Delta T = 0.383–0.964$ °C, C = calorimeter constant, 6,755 J/mol °C.

Results and discussion

The TG, DTG and DTA curves obtained in air with the compounds under study are given in Fig. 1A–D.

The TG, DTG, DTA curves reveal that the thermal degradation mechanism under the recording conditions is complex and characteristic of every sample showing with every compound two degradation domains according to the thermal nature of the degradation processes, the first between 130–420 °C being predominantly slightly endothermic and the second, strongly exothermal, between 420–620 °C.

Within the endothermic domain the degradation processes in air and in nitrogen atmospheres are noticed to be similar [23].

Within the endothermic domain the compounds **a, b, c** are degraded into three stages and the compound **d** into two stages which are not very well separated. In the exothermal domain the DTG and DTA curves are not symmetrical and show inflexion points which suggests a stepwise degradation in a domain where the molecular remainders of the first domain are burned.

With every sample the endothermic degradation domain is preceded by an endothermic process where the sample weight remains constant corresponding to the sample melting point peak [34].

The characteristic temperatures from TG-DTG-DTA and the weight losses of the stages of the endothermic and exothermal domains are given in Table 1.

The melting points from DTA are noticed to be in agreement to those measured by the Boetius method (Scheme 1).

The initial degradation temperatures from TG-DTG (Table 1) are indicative of the following stability series of the samples:

d > c > b > a

The thermal stability order resulting from the initial degradation temperatures is noticed to be identical to that coming from the formation enthalpies connected with the thermodynamic stability [25].

The experimental enthalpies of combustion and of formation estimated by means of the calorimetric bomb are given in Table 2.

As can be noticed the higher number of the carbon atoms in the molecule the stronger exothermal combustion is. This conclusion seems to be realistic since the $\Delta_{f}H^{o}_{298,CO2}$ term in the relationship for combustion enthalpy

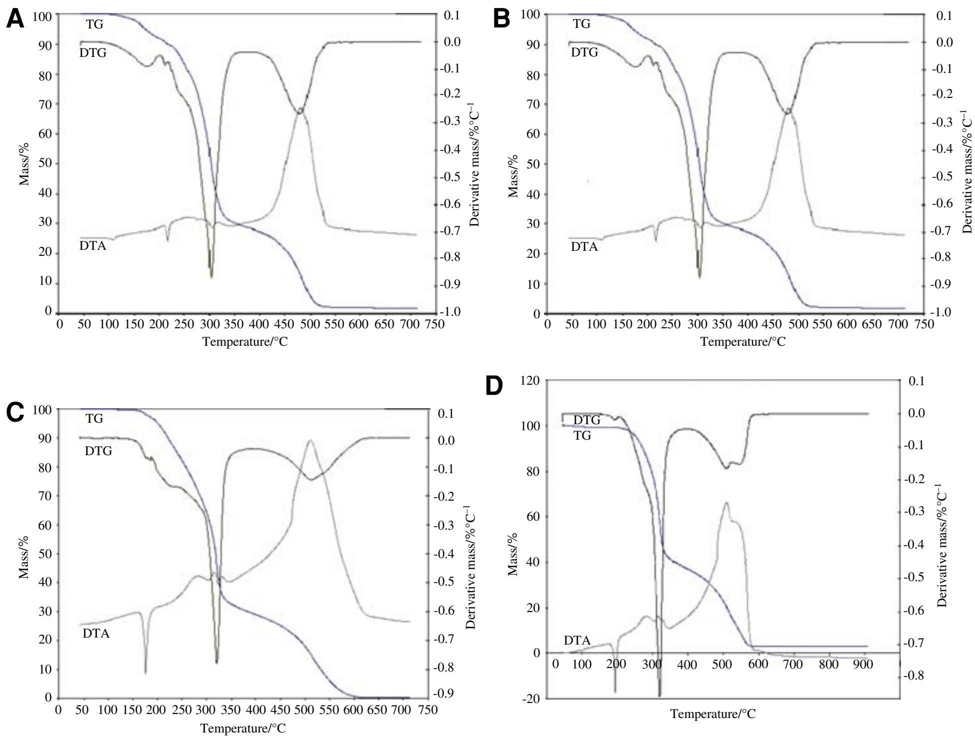


Fig. 1 **A** TG, DTG and DTA curves of the compound **a**. **B** TG, DTG and DTA curves of the compound **b**. **C** TG, DTG and DTA curves of the compound **c**. **D** TG, DTG and DTA curves of the compound **d**

Table 1 Characteristic temperatures from TG-DTG-DTA and weight losses in every stage

Thermal method	Temperature range	Stage	Characteristic temperatures	Sample			
TG-DTG	100–400 °C	I		**a**	**b**	**c**	**d**
			T_i°C	106.0	110.0	140.0	–
			T_m°C	175.0	178.3	176.6	–
			T_f °C	180.0	205.0	201.0	–
			W_∞ (%)	5	8.4	5.03	–
		II	T_i^{o}C	180.0	205	201.0	209.4
			T_m°C	230.0	241.6	226.6	–
			T_f^{o}C	275.0	265.0	270.0	282.0
			W_∞ (%)	24.02	23.02	32	20
		III	T_i°C	275.0	265.0	270.0	282.0
			T_m°C	340.0	303.3	320.0	320.5
			T_f°C	376.6	375.0	357.0	399.8
			W_∞ (%)	35.99	38.81	31.1	34.01
	420–620 °C	IV	T_i°C	376.6	375.0	357.0	399.8
			T_m°C	525.0	483.3	515.0	514.0
			T_f°C	540.0	538.3	620.0	590.0
			W_∞ (%)	31,21	27,86	31,65	40
	Residue (%)			4,65	1,87	0	1,47
DTA	110–330 °C (endothermic domain)		$T_{melting}$ °C	139.4	112.7	176.3	193.7
		I	T_m°C	177.6	174.1	198.7	–
		II	T_m°C	228.9	219.2	231.6	254.8
		III	T_m°C	326.7	308.3	324.8	325.7
	420–620 °C (exothermal domain)	IV	T_m°C	526.9	484.3	514.0	511.8

T_i initial thermal degradation temperature, T_m temperature at the maximum degradation rate, T_f final thermal degradation temperature, W_∞% weight loss (%)

Table 2 Combustion and formation enthalpies

Nr.	Sample	M (g/mol)	$\Delta_c H^o_{298}$ J/mol	$\Delta_f H^o_{298}$ J/mol
a	$C_{19}H_{18}N_4O_5S$	414.0	$-6.42 \cdot 10^6$	$-4.07 \cdot 10^6$
b	$C_{18}H_{15}N_4O_5SCl$	434.5	$-6.09 \cdot 10^6$	$-4.58 \cdot 10^6$
c	$C_{18}H_{15}N_4O_5SCl$	434.5	$-5.93 \cdot 10^6$	$-4.69 \cdot 10^6$
d	$C_{18}H_{15}N_4O_8S_2Cl$	514.5	$-4.31 \cdot 10^6$	$-5.71 \cdot 10^6$

calculation has a higher weight [25], the CO_2 being the most stable chemical compound involved in the combustion reaction. The values of the formation enthalpies of the samples can be ordered as given below which is indicative of a good agreement with the values resulting from T_i:

$$\Delta_f H^o_{298,d} < \Delta_f H^o_{298,c} < \Delta_f H^o_{298,b} < \Delta_f H^o_{298,a}$$

The data in Table 1 show that the temperatures of the maximum degradation rate, $Tm°C_I$, within the endothermic domain of the first stage are of the same values for the samples **a, b, c**, from DTG and coincide with those from DTA (174–178 °C) excepting for the sample **d** which does not show thermal degradation within this temperature domain. This finding is indicative of the same degradation reaction of the samples **a, b, c** in this stage.

The things are quite different with the degradation stages II and III of the samples **a, b, c** where $Tm°C_{II}$ and $Tm°C_{III}$ varies between 219–241 °C and 308–356 °C, respectively, and the stages are not very well separated.

In the exothermal domain, the $Tm°C_{IV}$ values of the samples **a, b, c, d** are close ($\approx$520 °C) which would suggest the same exothermal burning reaction.

By means of the FTIR analysis of the gases resulting from the thermal degradation in air and making use of the standard IR spectra [24] six gaseous species of small molecules were identified with the sample **a** (C_2H_2, $H_2C = NH$, CO_2, SO_2, H_2O, NH_3) and seven with the samples **b, c, d** (C_2H_2, $H_2C = NH$, CO_2, SO_2, H_2O, NH_3, HCl).

The nitrogen released within the endothermic domain at the degradation beginning and made evident by TG-DTG cannot be identified by FTIR since the degradation in the air with a 70% nitrogen content does not allow its identification. For the first stage of the endothermic domain (Table 1) W_{exp}% is in agreement with $W_{theoretical}$% estimated for nitrogen elimination. Thus, with the sample **a** $W_{exp} = 5\%$ and $W_{theoretical} = 6\%$.

The Fig. 2 presents the Gram–Schmidt curve and the evolution curves of the gaseous species released by the

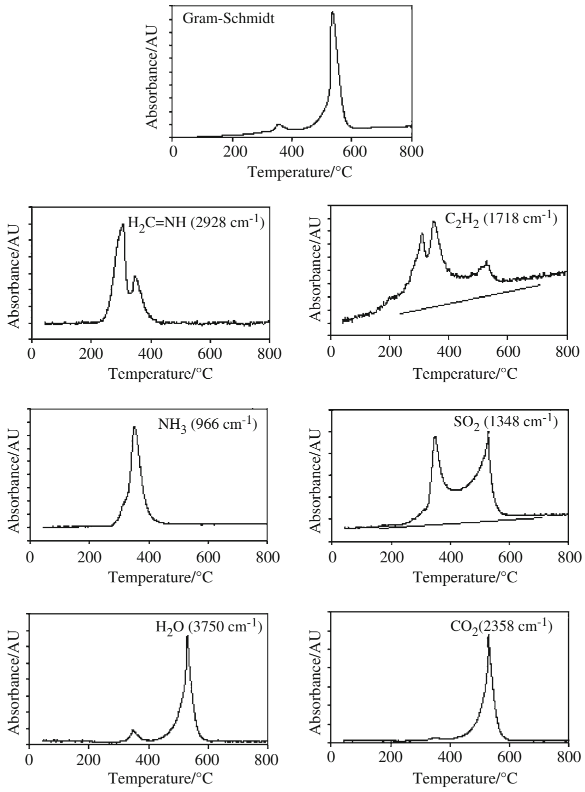


Fig. 2 The Gram–Schmidt curve and the evolution curves of the gaseous species evolved by thermal degradation of the sample (**a**) in air

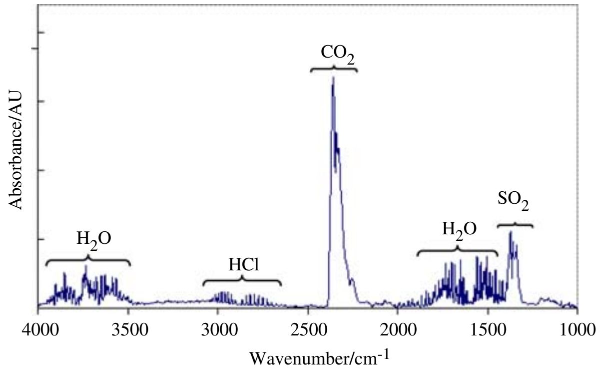


Fig. 3 FTIR spectrum of the gaseous species released by the thermal degradation of the sample (**d**) in air (time 35.1 min)

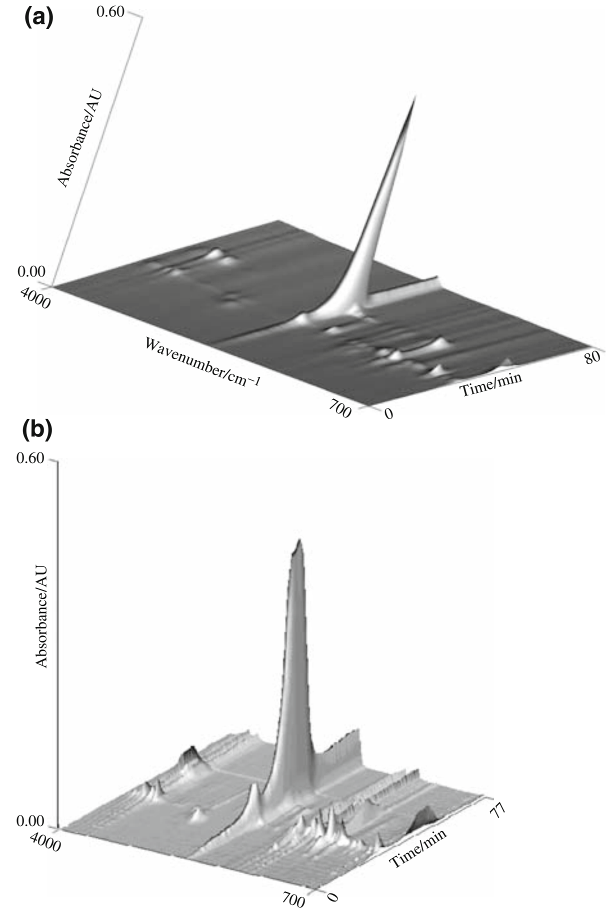


Fig. 4 3DFTIR spectra obtained for the thermal degradation of the samples (**a**) and (**d**) in air

Table 3 Theoretical and experimental weight losses in the endothermic and exothermal domains

Sample	Endothermic domain (%)		Exothermal domain (%)	
	W_t	W_{exp}	W_t + residue	W_{exp} + residue
a	63.28	65.01	36.72	34.99
b	68.69	70.23	31.31	29.77
c	68.69	68.13	31.31	31.87
d	58.01	57.74	41.99	42.26

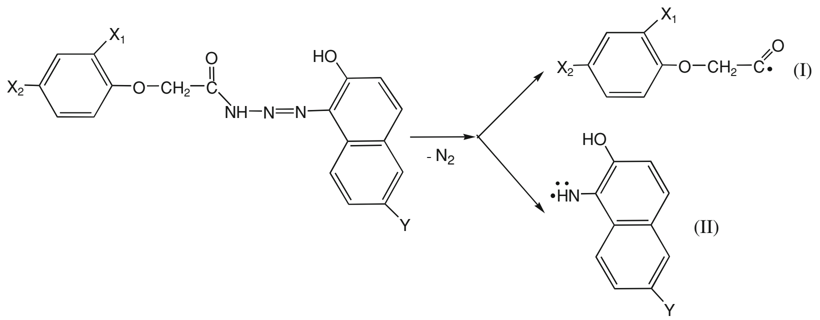
thermal degradation of the sample **a** in air which indicates the gaseous components to be eliminated into two temperature ranges corresponding to those given by TG-DTG-DTA. Thus, within the first range of 150–420 °C, coinciding with the endothermic domain in TG-DTG-DTA (C_2H_2, H_2C = NH, CO_2, SO_2, H_2O, NH_3) are eliminated while within the second, the exothermal domain between 420–600 °C (CO_2, SO_2, H_2O) are eliminated.

The samples **b, c, d** show a similar behavior by thermal degradation with one difference only, that the HCl is eliminated predominantly at the end of the endothermic domain where C_2H_2 and H_2C = NH have previously been eliminated. This fact can clearly be seen in Fig. 3 presenting the FTIR spectrum of the gases evolved by sample **d** degradation in air (time 35.1 min).

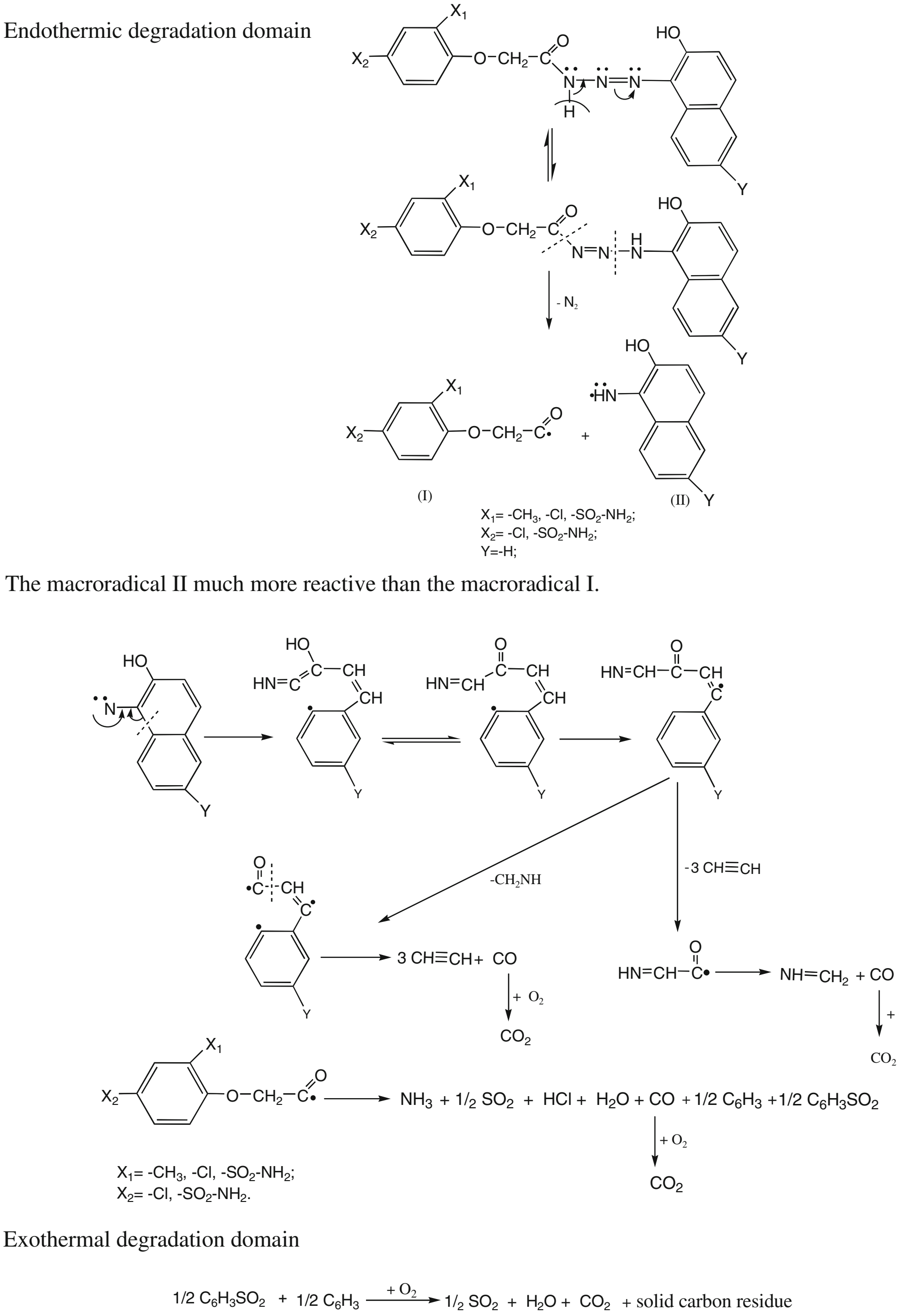
These findings would also result from Fig. 4a and d where the 3DFTIR spectrum of the gases released by thermal degradation of the samples **a** and **d** in air is depicted.

The correlation of the TG-DTG analysis data with FTIR measurements by means of the weight losses (W% exp.)

Scheme 2 The first decomposition of samples under study with nitrogen elimination and with the formation of two radical fragments



Scheme 3 The most probable mechanism of the thermal degradation in air under the condition of the TG analysis


Endothermic degradation domain

X_1= -CH_3, -Cl, -SO_2-NH_2;
X_2= -Cl, -SO_2-NH_2;
Y=-H;

The macroradical II much more reactive than the macroradical I.

X_1= -CH_3, -Cl, -SO_2-NH_2;
X_2= -Cl, -SO_2-NH_2.

Exothermal degradation domain

$$1/2\ C_6H_3SO_2 + 1/2\ C_6H_3 \xrightarrow{+O_2} 1/2\ SO_2 + H_2O + CO_2 + \text{solid carbon residue}$$

within a domain and of the released gaseous components (W% theoretical) gave the data in Table 3.

A good agreement is noticed which affords the advancement of the most probable degradation mechanism of the samples **a, b, c, d** in air. The thermal stability order of the compounds under study and assessments regarding the degradation mechanism in air are well sustained by their chemical structure and also by the general thermal behavior of the azoderivatives.

Thus, the azoderivatives are unstable compounds which are easily decomposed with nitrogen elimination [1] and that is why some of them can be used as radical initiators [35]. In case of these new azoderivatives we are right to admit the nitrogen molecule as being responsible for the easiest possible fragmentation.

Thus, under the assumption that the azoderivatives under study (Scheme 1) are decomposed with nitrogen elimination at the beginning two radical fragments are formed (Scheme 2):

An examination of the sample structures would indicate the samples **a, b, c** to give the same radical fragment, II-a (y = H) unlike the sample **d** giving the radical fragment II-b (y = SO_3H). The SO_3H group in II-b has a strong electron-withdrawing effect which strengths more the bond with –N = N–, the compound **d** is thus much more stable than **a, b, c** and the nitrogen is eliminated at temperatures higher than with the samples **a, b, c**, in agreement with the results coming from TG-DTG and $\Delta_f H^o_{298}$.

As regards the stability of the other three diazoaminoderivatives, **a, b, c,** the samples **b** and **c** are noticed to have the same substitutes, Cl and SO_2NH_2, differing only in their positions on the aromatic ring while the substitutes in the sample **a** structure are the CH_3 and SO_2–NH_2 groups.

Since an electron-releasing effect would result in an increased C–S bond order, hence in a rather difficult SO_2 elimination and thus an increased thermal stability of the compound the following ascertainments can be advanced: the both CH_3 and Cl groups show electron-releasing effects by +I and +E effects, respectively. Since the +E effect of the Cl atom is stronger than the +I of the CH_3 group the diazoaminoderivative **a** will be less stable (easier elimination of SO_2) than **b** and **c.**

Among the derivatives **b** and **c,** the derivative **b** is less stable due to the steric hindrance coming from the two ortho substitutes, the –SO_2NH_2 and –O–CH_2–NH–R groups. Consequently, the following order of the thermal stabilities can be given on the basis of the chemical structures which is in agreement with that resulting from the TG-DTG analysis and formation enthalpies:

d > c > b > a

Based on the TG-FTIR analysis and on the chemical structures of the compounds under study, the most probable degradation mechanism of the thermal degradation in air under the condition of the TG analysis was advanced (Scheme 3).

Conclusions

The study on the structure-thermostability-degradation mechanism correlation performed in air with four new aminoderivatives (Scheme 1) by means of TG-FTIR and formation enthalpies afforded the following conclusions:

- The TG-DTG-DTA analysis made evident a complex and specific thermal degradation mechanism in air (10 K/min) of the compounds under study, with two distinct temperature domains: an endothermic one which is identical to that under nitrogen atmosphere and the other exothermal.
- The TG-DTG-DTA characteristic temperatures indicate that the melting temperatures from DTA are in a good agreement with those measured by the Boetius method and the initial degradation temperatures are indicative of the following series:

  d > c > b > a
- The domains of degradation of the samples resulting from TG-DTG-DTA are also to be found in the grouping of the spectra of the gaseous species released during degradation.
- Based on the identification of the gaseous species eliminated by thermal degradation within the endothermic (C_2H_2, $H_2C = NH$, CO_2, SO_2, H_2O, NH_3) and exothermal (CO_2, SO_2, H_2O) domains the prevailing thermal degradation mechanism was advanced.
- The correlation between structure-thermostability-degradation mechanism brings useful information both on the temperature domain within which these compounds can be used and stored as well as on the environmental impact of the resulting gaseous products when the T_i temperature is exceeded during processing the products retaining these compounds.

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