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Microwave-assisted polymerization process: A way to design new, high molecular weight poly(arylimidazole)s

Edouard Chauveau, Catherine Marestin*, Vincent Martin, Régis Mercier

Laboratoire des Matériaux Organiques à Propriétés Spécifiques, UMR5041 CNRS – Université de Savoie, Chemin du Canal 69 360 Solaize, France

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ABSTRACT

In this paper, the microwave-assisted synthesis of high molecular weight poly(arylimidazole)s is described. These polymers were obtained by a one-pot polycondensation reaction involving a bis-(α-diketone), an aromatic dialdehyde and ammonium acetate. Depending on the dialdehyde monomer, different poly(arylimidazole)s structures were synthesized. Structural characterization (^{1}H and ^{13}C NMR), thermal properties (T_g, thermal stability) as well as a molecular weight determination of the polymers are reported.

1. Introduction

2,4,5-Triaryl imidazole-containing compounds obtained from a one-pot condensation reaction often possess interesting biological and pharmacological activities. As specific properties should be expected from the resulting polymers, the introduction of imidazole units in macromolecular chains has attracted the attention of chemists for long time. One of the pioneering work related to this subject was reported in 1967. Krieg and Manecke [1] described the condensation of aromatic dialdehydes with bis(α-diketone) in the presence of ammonia. However, only low molecular weight polymers were obtained. Despite a lot of work related to the synthesis of triaryl imidazole molecular compounds (using different catalysts [2–8], in the presence of ionic liquids [9], under microwave irradiation [10–12]), to the best of our knowledge, no further improvements have been yet reported concerning the synthesis of poly(arylimidazole)s. One probable reason is the complex control of the polycondensation reaction as it involves three different reactants. In this respect, it is worth mentioning that most polymers obtained by step-growth polymerization (for example poly(ester)s, poly(imide)s, poly(amide)s, poly(benzimidazole)s) are synthesized by condensation reactions between two difunctional monomers. To date, no example of linear aromatic and heterocyclic polymers obtained by condensation of three or more monomers has been yet described.

In order to circumvent the synthetic difficulties associated with the formation of triaryl imidazole ring during the polymerization reaction, Akutsu et al. [13] have designed a specific monomer (4-4′-2-phenyl-(4,5-imidazole)dibenzoic acid) containing the desired heterocyclic group which was then involved in the synthesis of poly(amide)s by a conventional polycondensation reaction. Following a similar approach, Hergenrother and Connell [14] have synthesized poly(arylene ether imidazole)s by aromatic nucleophilic substitution, also using a triaryl imidazole-containing monomer.

In this article, our recent advances on the synthesis of poly(arylimidazole)s bearing the imidazole ring in the main chain are reported. Whereas conventional thermal polymerization conditions were found to be ineffective to get high molecular weight polymers, a microwave irradiation process enabled to do it.

2. Experimental section

2.1. Materials

All chemicals were of reagent grade and used as received from commercial sources (unless otherwise mentioned).

2.2. Characterization methods

Thermogravimetric analyses (TGA) were performed on a TA Instruments model TGQ50 thermogravimetric analyzer, under air, both in isothermal or dynamic mode. In dynamic mode, experiments were achieved from 25 to 500 °C at 10 °C/min. Monomer

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* Corresponding author. Tel.:+33 4 78 02 35 42; fax: +33 4 78 02 77 38.
E-mail address: marestin@lmops.cnrs.fr (C. Marestin).

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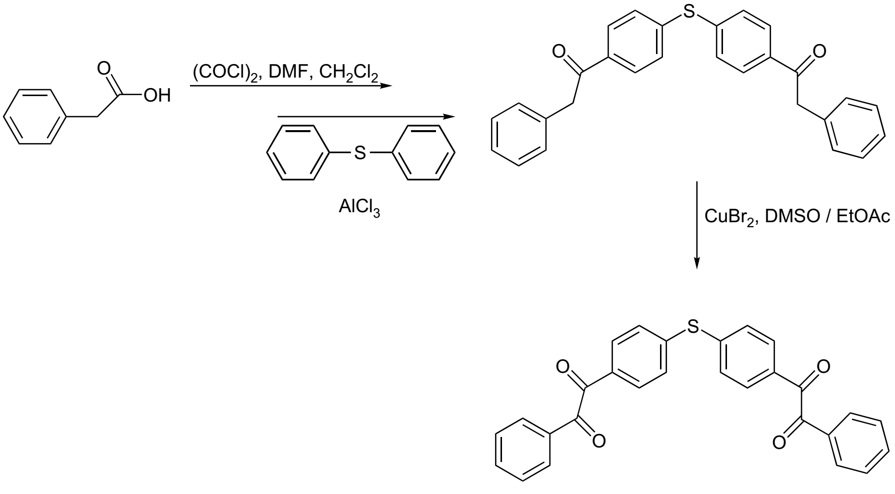

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Fig. 1. Bis(α-diketones) **1** monomer synthesis.

NMR spectra were recorded either on a Bruker 200 MHz or 250 MHz spectrometer. Polymer NMR spectra were recorded on a Bruker 400 MHz spectrometer. Deuterated dimethylsulfoxide (DMSO-d_6) and deuterated chloroform ($CDCl_3$) were used as the solvents. Tetramethylsilane (TMS) was used as the chemical shift reference. The polymer glass transition temperatures (T_g) were determined from Mettler-Toledo DSC822e measurements. Analysis were performed under nitrogen, at a heating rate of 5 °C/min. Reported values were obtained from a second heat scan. T_g was calculated using the midpoint method. Molecular weights were determined by size exclusion chromatography on a system equipped with a Waters 515HPLC pump, a differential refractive index detector (Wyatt optilab -rEX 25 °C and 658 nm), a viscosity detector (Wyatt viscostar) and a laser light scattering detector (Wyatt Minidawn TREOS-658 nm). DMF containing 0.05 M LiBr was used as an eluent. SEC analyses were performed on two PL gel Mixed D columns thermostated at 70 °C. Absolute molecular weights were determined taking into account the measured d_n/d_c of the corresponding polymers. d_n/d_c was measured in the same conditions (temperature, wavelength) as those used for the SEC analysis. "Equivalent polystyrene" mean molecular weights (denoted $M_{n(PS)}$ and $M_{w(PS)}$) were also determined by using a polystyrene calibration curve.

2.3. Synthesis of monomers

*4,4′-Bis(phenylglyoxylyl)diphenylsulfide (**1**).* 10 g (72 mmol) of phenylacetic acid were suspended in 40 ml of dichloromethane containing few drops of DMF. 14 g (108 mmol) of oxalyl chloride were added under vigorous agitation. After 2 h reaction at room temperature, the temperature was increased and the reaction mixture was refluxed for 1 h. After completion of the reaction, the excess of oxalyl chloride was distilled off and the resulting product was further involved in the following Friedel–Crafts reaction.

In a 250 ml three-necked round bottom flask equipped with a magnetic stirrer, a nitrogen inlet, a thermometer and an acid trap were introduced 6.4 g (34 mmol) of diphenylsulphide, 9.37 g (68.8 mmol) of aluminum chloride and 45 ml of dichloromethane. The temperature of the reaction mixture was maintained at −5 °C and the acyl chloride previously prepared was added drop wise. After complete addition, the medium was stirred for 2 h at 0 °C and for two more hours at room temperature. Upon completion of the reaction (as witnessed by TLC), the medium was poured into deionized water. The resulting yellowish powder was then collected by filtration, washed with water and acetone and dried at 70 °C under vacuum. The pure diketone was isolated with 95% yield. ^1H NMR (DMSO-d_6, 294 K): δ (ppm) 7.99 (d, 4H, $J = 7.5$ Hz), 7.45 (d, 4H, $J = 8$ Hz), 7.28–7.23 (*m*, 10H), 4.31 (*s*, 4H). ^{13}C NMR (DMSO-d_6, 294 K): δ (ppm) 196.7, 139.9 (C_Q), 135.7 (C_Q), 134.9 (C_Q), 130.5, 129.5, 129.4, 128.2, 126.4, 44.7.

11.68 g (27 mmol) of the aforementioned diketone and 24.7 g (110 mmol) of copper (II) bromide were suspended in 66 ml of a (50/50-v/v) DMSO/ethyl acetate mixture and refluxed for 24 h. The system was cooled to room temperature and poured into 350 ml of deionized water. The organic product was extracted with 350 ml of ethyl acetate. The organic phase was thoroughly washed with water in order to remove all copper salts. The yellow solid obtained after evaporation of the ethyl acetate was dissolved in dichloromethane. The white and insoluble dimethylsulfide–CuBr complex formed during the reaction was eliminated by filtration. The organic phase was finally washed with water, dried over sodium sulphate and concentrated in vacuo. The crude product was purified by crystallization in acetonitrile, giving a bright yellow

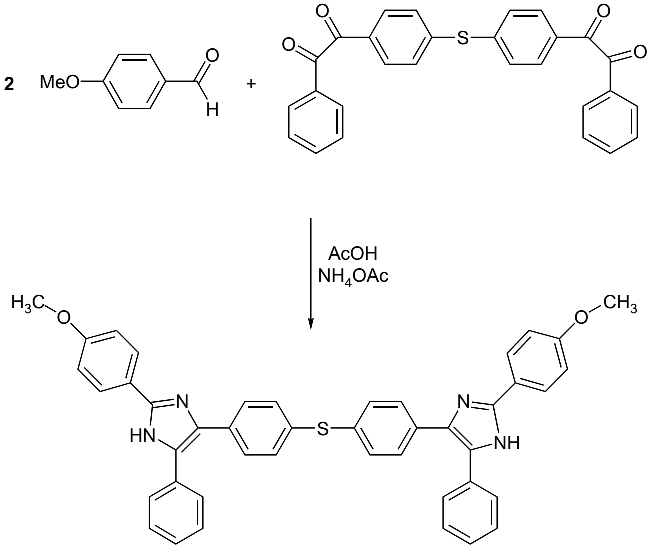

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Fig. 2. Bis(arylimidazole) model compound synthesis.

polymer grade monomer with 88% yield. ^{1}H NMR ($CDCl_3$, 294 K): δ (ppm) 7.96 (d, 4H, *J* = 7.2 Hz), 7.92 (d, 4H, *J* = 8.7 Hz), 7.66 (*t*, 2H, *J* = 7.2 Hz), 7.52 (*t*, 4H, *J* = 7.7 Hz), 7.45 (d, 4H, *J* = 8.5 Hz). ^{13}C NMR ($CDCl_3$, 294 K): δ (ppm) 194.0, 193.2, 143.0 (C_Q), 135.1, 132.8 (C_Q), 131.8 (C_Q), 130.8, 130.7, 129.9, 129.1.

Terephtaldehyde ***2a***, commercially available, was purified by the following procedure. The dialdehyde was dissolved in ethyl acetate and washed with a basic aqueous solution. The organic phase was concentrated under reduced pressure, leading to a pure monomer.

*4,4′-Bisformyl-diphenylether (**2b**).* A 250 ml three-necked round bottom flask equipped with a condenser, a nitrogen inlet and a magnetic stirrer was charged with 12 g (98.2 mmol) of 4-hydroxybenzaldehyde, 12.44 g (98.2 mmol) of 4-fluorobenzaldehyde, 10.86 g (78.6 mmol) of potassium carbonate and 57 ml of DMAC. The reaction mixture was heated at 150 °C for 6 h. After cooling, the medium was poured into 300 ml of deionized water. The resulting precipitate was filtrated and thoroughly washed with water. Finally, any traces of 4-fluorobenzaldehyde were removed by slightly washing the product with petroleum ether. A polymer grade monomer was obtained with 70% yield. ^{1}H NMR ($CDCl_3$, 294 K): δ (ppm) 9.96 (*s*, 2H,), 7.91 (d, 4H, *J* = 8 Hz), 7.17 (d, 4H, *J* = 8 Hz). ^{13}C NMR ($CDCl_3$, 294 K): δ (ppm) 190.6, 160.9 (C_Q), 132.5 (C_Q), 132.0, 119.3.

2c–**2f** dialdehyde monomers were obtained by aromatic nucleophilic substitution involving 4-fluorobenzaldehyde and various biphenol compounds, according to the procedure detailed for the synthesis of compound **2c**.

*1,4-Bis(4-formylphenoxy)benzene (**2c**).* In a 250 ml three-necked round bottom flask equipped with a nitrogen inlet, a condenser and magnetic stirrer, 15 g (136 mmol) of hydroquinone, 37.95 g (299 mmol) of 4-fluorobenzaldehyde and 22.59 g (163 mmol) of potassium carbonate were suspended in 120 ml of DMAC. The reaction mixture was heated at 150 °C for 6 h. After completion of the reaction, the medium was cooled to room temperature. The crude dialdehyde compound was isolated by filtration as red crystals which were successively washed with 300 ml of water and 200 ml of methanol. A polymer grade monomer was obtained with 75% yield, after crystallisation in 1,2-dichloroethane. ^{1}H NMR ($CDCl_3$, 294 K): δ (ppm) 9.91 (*s*, 2H,), 7.86 (d, 4H, *J* = 8 Hz), 7.12 (*s*, 4H), 7.08 (d, 4H, *J* = 8 Hz). ^{13}C NMR ($CDCl_3$, 294 K): δ (ppm) 190.6, 163.0 (C_Q), 151.9 (C_Q), 131.8, 131.5 (C_Q), 121.9, 117.5.

*4,4′-Bis(4-formylphenoxy)biphenyl (**2d**).* The crude product was washed with methanol and petroleum ether. A polymer grade monomer was obtained with 82% yield without further purification. ^{1}H NMR (DMSO-d_6, 360 K): δ (ppm) 9.95 (*s*, 2H,), 7.93 (d, 4H, *J* = 8 Hz), 7.74 (d, 4H, *J* = 8 Hz), 7.21 (d, 4H, *J* = 8 Hz), 7.18 (d, 4H, *J* = 8 Hz). ^{13}C NMR (DMSO-d_6, 360 K): δ (ppm) 191.5, 162.3 (C_Q), 155.0 (C_Q), 136.1 (C_Q), 131.8 (C_Q).

*2,2-Di[4-(4-formylphenoxy)-phenyl]propane (**2e**).* Pure monomer was obtained with 45% yield after crystallization in acetone. ^{1}H NMR ($CDCl_3$, 294 K): δ (ppm) 9.91 (*s*, 2H,), 7.84 (d, 4H, *J* = 8 Hz), 7.3 (d, 4H, *J* = 8 Hz), 7.07 (d, 4H, *J* = 8 Hz), 7.01 (d, 4H, *J* = 8 Hz). ^{13}C NMR ($CDCl_3$, 294 K): δ (ppm) 190.7, 163.2 (C_Q), 152.9 (C_Q), 147.1 (C_Q), 131.9, 131.2 (C_Q), 128.5, 119.9, 117.5, 42.4 (C_Q), 31.0.

*2,2-Di[4-(4-formylphenoxy)-phenyl]-1,1,1,3,3,3-hexafluoropropane (**2f**).* A high purity monomer was obtained with 40% yield after crystallization in isopropanol. ^{1}H NMR ($CDCl_3$, 294 K): δ (ppm) 9.94 (*s*, 2H,), 7.89 (d, 4H, *J* = 8 Hz), 7.44 (d, 4H, *J* = 8 Hz), 7.14 (d, 4H, *J* = 8 Hz), 7.08 (d, 4H, 8 Hz). ^{13}C NMR ($CDCl_3$, 294 K): δ (ppm) 190.7, 161.8 (C_Q), 156.1 (C_Q), 132.0, 129.1 (C_Q), 124.1 (*q*, *J* = 286 Hz), 119.2, 118.6, 63.9 (sept, *J* = 25.6 Hz).

2.4. Microwave-assisted synthesis

Reactions performed under microwave radiations were achieved in a multi-mode Milestone microwave oven. 90 ml heavy-walled, high pressure Teflon reactors equipped with a magnetic stirrer, a pressure sensor and an optical fiber probe dipping in the reaction mixture as temperature sensor were used. For safety reasons, in all microwave-assisted experiments, the upper operating temperature and upper operating pressure were respectively set to 160 °C and 25 bars. Whenever one of these parameters was reached, the magnetron's power was automatically shut down and further regulated.

Bis(arylimidazole) model compound synthesis. 1.3 g (2.9 mmol) of bis(α-diketone) **1** 0.84 g (6 mmol) of anisaldehyde and 4.54 g (57.7 mmol) of ammonium acetate were suspended in 12 ml of acetic acid. The Teflon reactor was hermetically closed and submitted to microwave irradiation (500 W) for 10 min. The system was cooled until complete depressurization of the reactor. The resulting homogeneous medium was poured into 150 ml of deionized water. The bis(arylimidazole) model compound was

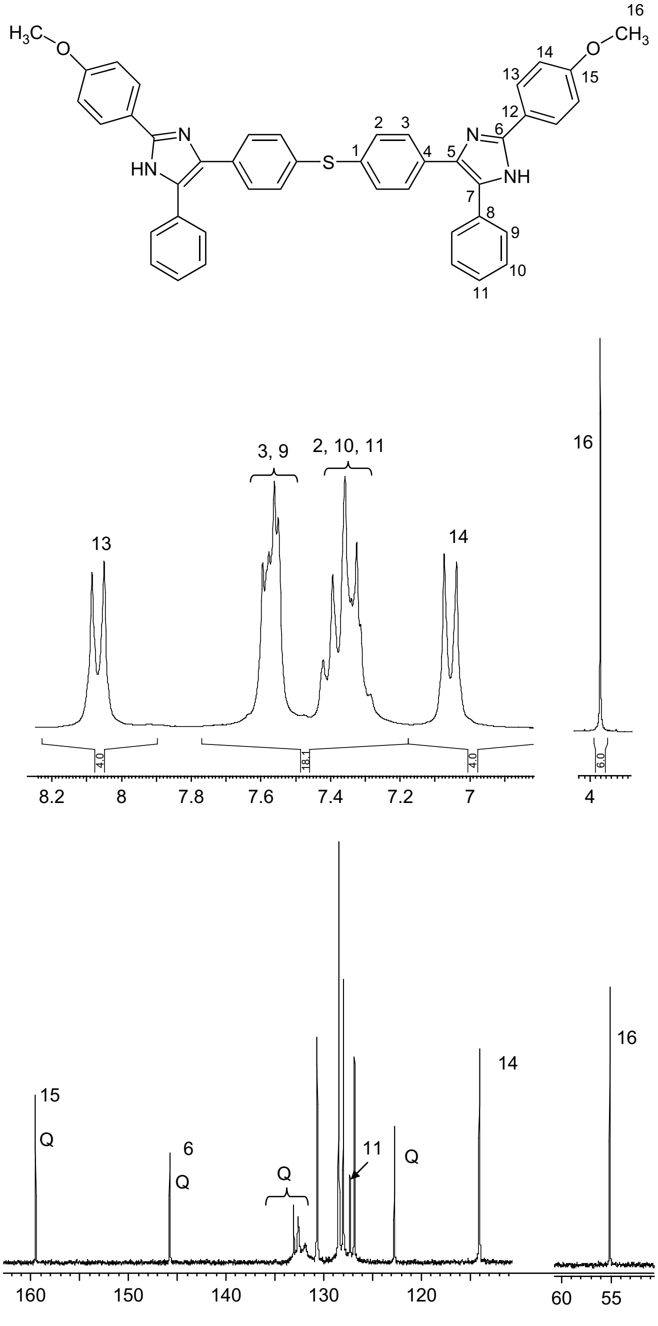

Fig. 3. ^{1}H and ^{13}C NMR spectra (DMSO-d_6) of the bis(imidazole) model.

isolated as a white solid which was washed with a small amount of cold isopropanol to remove any traces of residual anisaldehyde. The resulting compound was thoroughly dried at 80 °C under vacuum.

Microwave-assisted poly(arylimidazole)s synthesis. All microwave-assisted poly(arylimidazole)s synthesis were performed according to the following general polymerization procedure: An equimolar amount (3.33 mmol) of bis(α-diketone) **1** and dialdehyde **2a**–**2f** monomers, 66.6 mmol of ammonium acetate, 5 ml of acetic acid and few ml of NMP (in order to reach a 15 wt% solid content) were introduced in a high-pressure Teflon reactor and submitted to microwave irradiations (500 W) for 15 min. Once the reaction medium was cooled to room temperature, it was poured into water to precipitate the polymer which was then isolated by filtration. The polymer was chopped into pieces, washed successively with water and methanol, and dried under vacuum.

2.5. Film preparation

Thin dense films were prepared by casting 20 wt% polymer solution in NMP onto clean glass plates. After the solvent evaporation (12 h at 50 °C, 1 h at 80 °C, 1 h at 120 °C, 1 h at 150 °C and 1 h at 180 °C), the films were peeled off by immersion in methanol.

3. Results and discussion

3.1. Synthesis of monomers

Monomer **1** was obtained through a multi-step synthesis. Fig. 1 illustrates the general synthetic procedure. First, the phenylacetic acid is allowed to react with oxalyl chloride [15] to form its carboxylic chloride derivative. This reagent is further involved in a Friedel–Crafts reaction on diphenylsulfide. The oxidation of the resulting bisbenzyl compound is then performed in the presence of cupric bromide and DMSO, in ethyl acetate. The resulting bis(α-diketone) product is obtained as a polymer grade monomer with a 84% overall yield.

On the other hand, series of dialdehyde compounds were synthesized from fluorobenzaldehyde and different bisphenol, via a nucleophilic substitution.

3.2. Bis(arylimidazole) model compound and poly(arylimidazole)s synthesis

From the bis(α-diketone) monomer and anisaldehyde, a bis(arylimidazole) model compound **2** was synthesized, in the presence of ammonium acetate (Fig. 2).

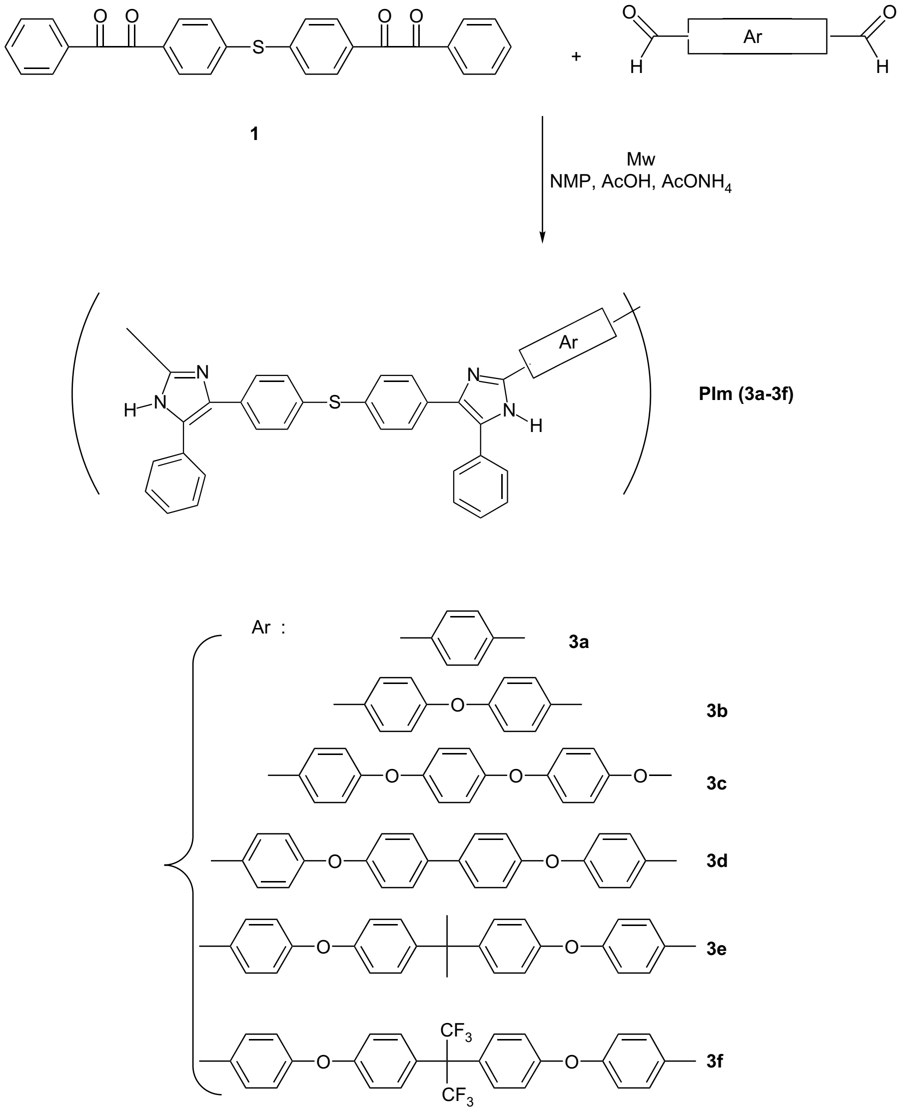

Fig. 4. Microwave-assisted synthesis of poly(arylimidazole)s.

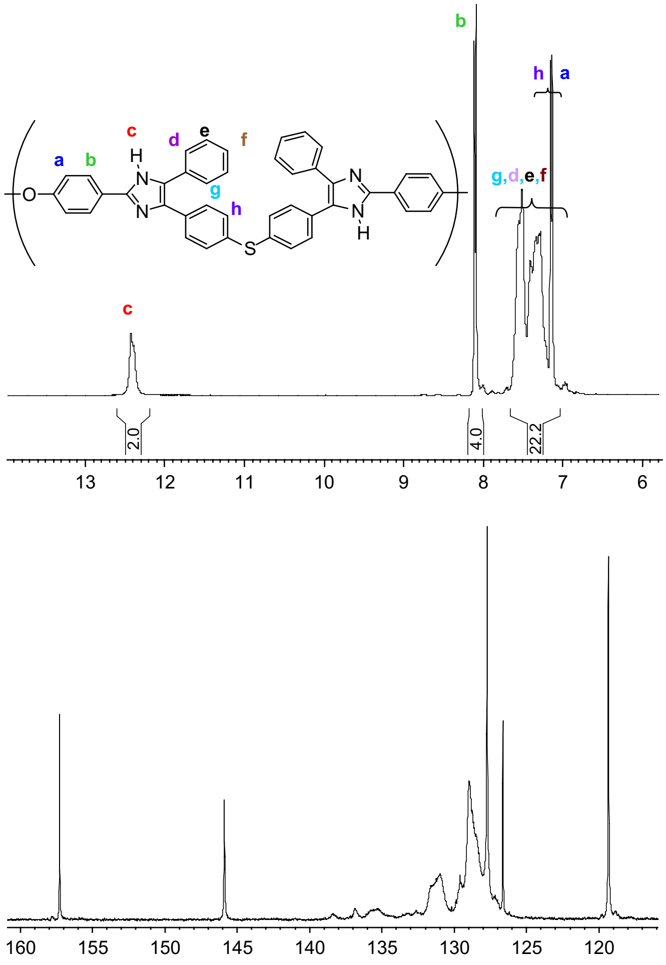

Fig. 5. ^{1}H and ^{13}C NMR spectra of PIm **3b** in (DMSO-d_6).

In an abundant dedicated literature,[16–22] microwave-assisted processes are generally considered as very attractive because of the short reaction time and the good reaction reactivities in comparison with conventional thermal treatment. As witnessed by the tremendous interest of pharmaceutical groups in such a technique, microwave-assisted synthesis appears particularly suitable for the elaboration of aromatic and heterocyclic compounds.

Therefore, the imidazole-containing model compound was performed under microwave radiation. For this purpose, all reagents were introduced simultaneously in a high-pressure Teflon reactor and submitted to microwaves (500 W) for 15 min. In such conditions, very high temperatures were reached quasi-instantly leading to ammonium acetate dissociation and give free ammonia. It should be noted that whereas high-pressures could be recorded (up to 10 bars) at the very beginning, the evolution of the reaction could be followed by a pressure decrease associated to ammonia consumption. The resulting compound was fully characterized by NMR (Fig. 3).

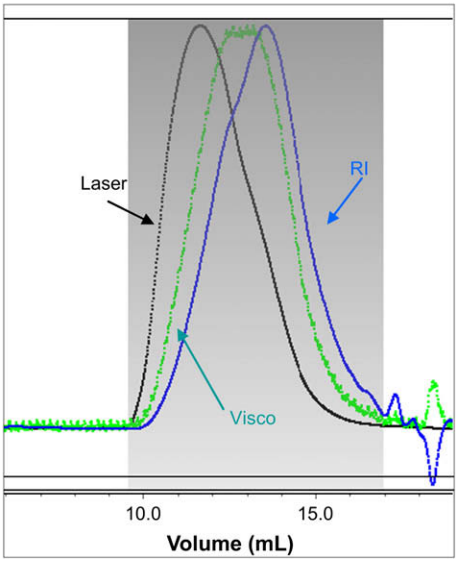

Fig. 6. SEC chromatogram of a poly(arylimidazole).

The ^{1}H NMR spectrum is in good accordance with the expected structure. The presence of the two methoxy groups is characterized by the singlet at 3.8 ppm. Further evidence to support the model compound structure concerns a "AA'-BB'" system, which is typical for a para-substituted aromatic ring. While a first doublet (7.05 ppm, $J = 8.75$ Hz) is present upfield, due to the electron-donating effect of the methoxy groups, the other doublet (8.07 ppm, $J = 8.75$ Hz) appears downfield, because of the electron-withdrawing effect of the imidazole ring.

^{13}C NMR spectrum confirms the model compound identification.

A series of polymers were synthesized using bis(α-diketone) **1** and the different dialdehyde monomers **2a**–**2f** (Fig. 4). Whereas dialdehyde **2a** and **2d** leads to insoluble polymers, the more flexible ether-containing dialdehyde monomers allowed to obtain readily soluble polymers in common organic solvents (NMP, DMSO, DMF,…).

Numerous attempts to synthesize poly(arylimidazole)s with the aforementioned structures by thermal conventional procedures have been performed as well. Different experimental conditions were investigated. Some experiments were done at atmospheric pressure in the presence of various catalysts (Yb(Otf)$_3$ or NiCl$_2$), according to the procedures described by Wang et al. [3] and Heravi et al. [5] for the synthesis of imidazole organic compounds. Some polymerization reactions were also performed without any catalyst, but under pressure. In this case, a wide range of polymerization times were investigated (from 4 to 18 h reactions). However, whatever the conditions, only insoluble materials and useless oligomers were isolated.

From these results, it appears that the synthesis of high molecular weight poly(arylimidazole)s can only be achieved under microwave irradiation. To our opinion, the most meaningful

Table 1
Poly(arylimidazole)s characterization

Polymer sctructure	T_g (°C)	$T_{\Delta 5\%}$ (°C)	Δm% After a 48 h treatment	M_n (g/mol)	M_w (g/mol)	IP	M_{nPS} (g/mol)	M_{WPS} (g/mol)	IP
PIm **3a**	295	413	/	[a]	[a]	[a]	[a]	[a]	[a]
PIm **3b**	278	429	<1% @270 °C	–	–	–	–	–	–
PIm **3c**	258	427	<1% @250 °C	–	–	–	–	–	–
PIm **3d**	228	443	<1% @216 °C	[a]	[a]	[a]	[a]	[a]	[a]
PIm **3e**	237	429	<1% @229 °C	16 700	39 800	2.4	44 400	94 200	2.1
PIm **3f**	239	439	<1% @231 °C	9 600	17 000	1.8	26 500	50 900	1.9

[a] Insoluble; -poorly soluble in DMF/LiBr; $T_{\Delta 5\%}$: 5% weight loss temperature.

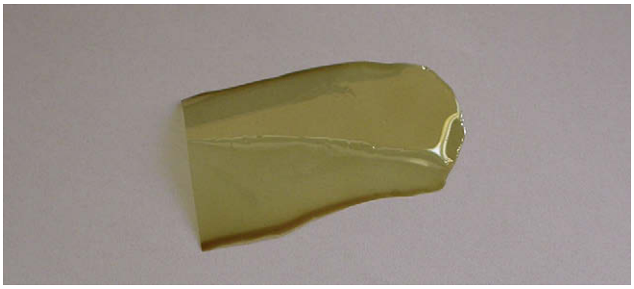

Fig. 7. Thin tough film of a poly(arylimidazole) obtained from solution casting method.

hypothesis which could account for that is related to the specific thermal profile obtained under microwave irradiation (very high temperatures reached quasi-instantly, no wall effects). As a matter of fact, we can reasonably think that in such conditions, the formation of side reactions is precluded. On the other hand, in thermal conventional conditions, the formation of side reactions (even in extremely low proportions) is supposed to lead to the formation of chain ends and result in the synthesis of low molecular weight oligomers.

3.3. *Characterization of poly(arylimidazole)s*

The ^{1}H and ^{13}C NMR characterizations of the polymers obtained under microwave irradiation are consistent with the expected macromolecular structure. As an example, the NMR spectra of polymer PIm **3b** are reported on Fig. 5.

Depending on the monomer structure, the glass transition temperature of poly(arylimidazole)s range from 237 to 295 °C. The thermal stability under air of these polymers was also investigated by TGA analysis. The results are reported in Table 1. All polymers can withstand drastic thermal conditions as they have a 5% weight loss temperature around 427–443 °C. Moreover, they are unaffected by a prolonged treatment (48 h) performed at a temperature slightly below their glass transition temperatures and under air.

“Polystyrene equivalent” molecular weights were determined using a polystyrene calibration curve. Moreover, in order to obtain more accurate molecular weights, specific refractive Index Increments (d_n/d_c) were determined for each polymer. Values obtained are 0.261 and 0.217 respectively, for PIm **3e** and PIm **3f**. Taking these values into account, absolute average molecular weights were determined thanks to a triple detection (RI, Viscosimeter, MALLS) system. Fig. 6 shows an example of a poly(arylimidazole) SEC chromatogram. DMF/LiBr was used as an eluent.

SEC data obtained for PIm **3b** and PIm **3c** are not reported because the chromatograms shape suggests the presence of some aggregates (probably related to a poor solubility of these structures in DMF/LiBr).

All poly(arylimidazole)s above mentioned could be cast into transparent thin tough films (Fig. 7) suggesting that the molecular weights are quite high.

Recent advances in microwave-assisted polymer synthesis or polymer modification [23–27] already underlined the high potential of this technique. However, all reported examples concern macromolecular structures which can be synthesized as well by conventional thermal way. In this work, it was shown that thanks to microwave irradiation, not only very short reaction times (15 min) were required to obtained high molecular weight polymers, but also new macromolecular structures (high molecular weight poly(arylimidazole)s bearing imidazole rings in their main backbone) can be envisioned.

4. Conclusion

In this work, the polycondensation of a bis(α-diketone) monomer with different aromatic dialdehydes, in the presence of ammonium acetate was investigated. Whereas conventional thermal polymerization conditions only afforded the formation of oligomers and insoluble materials, high molecular weight polymers were successfully synthesized under microwave irradiation. By varying the nature of the aromatic dialdehydes, different poly(arylimidazole)s structures were obtained. These polymers have a high T_g and display a good thermal stability. It was also shown that self-supporting films can be obtained from these polymers. These results highlight the great potential of microwave-assisted processes for the synthesis of new and innovative aromatic and heterocyclic polymers. Works are in progress to further investigate the properties of these polymers.

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