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The electromagnetic properties of poly(p-phenylene-vinylene) derivatives

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We report the studies on the frequency dependence of the complex permittivity ($\varepsilon * = \varepsilon' - i.\varepsilon''$) and of the electromagnetic interference shielding (EMI) of the conducting polymers such as poly(*para*-phenylene-vinylene) (PPV) and poly(2,5-dimethoxy,1,4-phenylene-vinylene) (DMeO-PPV) doped with different dopants. Permittivity measurements from 10 kHz to 10 GHz point out that all conducting polymers doped with H_2SO_4 show a conductivity range of 10^{-1}–10^{+1} S cm^{-1}, but doped DMeO-PPV has conductivity slightly higher than that of PPV. From these experimental data, the reflectivity coefficients of these materials are calculated in a frequency range 3–5 GHz, and have the values up to − 30 dB depending on the frequency, on the thickness of the materials, on the dopant agents and specially on the doping levels of the materials. The potential applications of these materials in EMI shielding are discussed. © 1998 Elsevier Science Ltd. All rights reserved

(Keywords: electromagnetic shielding; microwave; poly(p-phenylene-vinylene))

INTRODUCTION

In recent years, conducting polymers have emerged as a potential class of materials for electromagnetic interference (EMI) shielding and microwave absorbers ([1,2] and references therein,[3–7]). These radiation shielding materials are essential for high operational reliability and long life of electronic equipment, since they reduce or suppress electromagnetic noise. They are also used in military applications, such as in stealth technology. Many types of radar absorbing materials (RAM) are available commercially [8,9]. Yoshino *et al.* [2] were the first authors that reported on the application and characteristics of a conjugated conducting polymer such as polypyrrole in EMI shielding. Similar studies devoted to conducting polymers or their derivatives [2,10–26] are referred to in *Table 1*.

Whether intrinsically conducting or in the composite form, conducting polymers have been the subject of intensive research concerning chemical and electrical, as well as mechanical properties. Microwaves (hyperfrequency range 10^8–10^{11} Hz) have been used in the measurements of complex permittivity to study electrical properties and to elucidate the molecular dynamics of polar and non-polar polymeric materials [27].

Generally, most of conducting polymers are highly sensitive to degradation/oxidation, they are also infusible and insoluble in nature, and this makes processing difficult or impossible. Therefore, many efforts have been made to improve the solubility of conducting polymers. Thus, soluble substituted poly(p-phenylene) [28], poly(p-phenylene ethylene) [29], poly(thiphene) [30,31] and poly(p-arylene vinylene) [32,33] have been prepared. On the other hand, the choice of polymers as materials for application in microwave absorbers and EMI shielding depends on their properties as reported in [23] and references cited therein. For example, polypyrroles for their in-situ synthesis with insulating polymers used as host matrix; polyanilines for their special physico-chemical properties and which can be easily deposited on reinforced textile; and polyalkyl thiophenes for their high solubility and their fusibility.

Compared with other conducting polymers, poly(phenylene-vinylene) (PPV) is a recent polymer, it has not yet been investigated for EMI, though recently, dielectric properties of PPV thin films have been studied [34,35]. However, PPV is the most studied in the recent years because it presents many advantages: it is a high molecular weight polymer obtained via a hydrosoluble precursor [36], it is highly resistant to oxidation and has a high thermal decomposition temperature (up to 500°C under ambient atmosphere) [37]. PPV chains can be oriented [38] and PPV is highly crystalline. It has also excellent inherent mechanical properties [39]. It can be p- or n-doped to give high conductivity [39,40] but still inferior to that of polyacetylene [41,42]. More recently, it has been shown that PPV possesses interesting non-linear optics [43] and electroluminescent properties [44]. Furthermore, the ease of modifying PPV structure to obtain the desired properties (from a powder to a fibre or free-standing films, from insoluble to soluble polymers) offers PPV many potential applications.

On the other hand, in our previous paper [45], we reported a direct treatment of the PPV sulfonium precursor with H_2SO_4 which leads to a H_2SO_4-doped PPV with the same optical properties and slightly higher conductivity compared with PPV obtained by pyrolysis of the precursor and doped with H_2SO_4.

In this paper, we study the electromagnetic shielding of PPV and its derivative DMeO-PPV obtained by different pathways of conversion of their precursors.

EXPERIMENTAL

Products

All chemical products were purchased from Aldrich (except $FeCl_3$ from Fluka) and were used without further purification.

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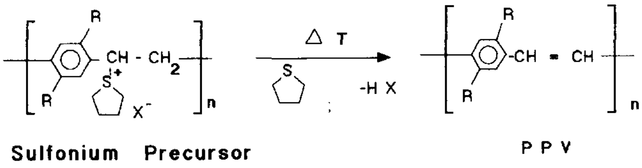
Table 1 Conducting polymers and their potential electromagnetic applications

Polymers	Applications	References
PANi	EMI, ESD	10,5,6,7
PPy (films)	EMI	2
PPy in polyester matrix	EMI, RAM	11,12
PPy on paper, cotton and polyester (Contex®)	RAM	13–18
PPy (films)	EMI	19
PPy: in-situ polymerized on non-conducting polymers		
PPy: powder dispersed in thermoplastics, elastomers	RAM	20–23
PPY, PANi	Welding	24
P-3 Octyl-thiophene/PS,PVC,EVA matrix	EMI	25
PBT (poly[p-phenylene benzo-bis thiazole])	EMI,RAM	26

EMI, electromagnetic interference; RAM, radar absorber materials; ESD, electrostatic dissipation

Synthesis

Poly(p-phenylene-vinylene). PPV was synthesized according to the method described in our previous study [45]. The casting of the precursor allows the production of films of different shapes. The conversion of precursor sulfonium into PPV was carried out by pyrolysis at 150°C for many hours (Scheme I, where R = H), and then PPV films were doped with H_2SO_4 by immersing in the acid. In the chemical conversion, the films of PPV precursor were immersed directly in acids such as H_2SO_4 or HBF_4, and the PPV thus obtained were doped with the corresponding acids during the conversion [45]. All films range in thickness from 30 to 160 μm.



Poly(2,5-dimethoxy-1-4, phenylene-vinylene). The synthesis of DMeO-PPV (R = OCH_3, Scheme I) has been described in [46]. But in this study, the conversion of precursor sulfonium into DMeO-PPV was carried out by different methods. By pyrolysis, the precursor was heated at 200°C for 17 h then doped with concentrated H_2SO_4. By the chemical method, the precursor sulfonium was treated with either concentrated H_2SO_4 or HBF_4 (the DMeO-PPV films thus obtained were doped with the corresponding acid during the conversion). Doping with $FeCl_3$ of DMeO-PPV films was obtained by dedoping the films obtained via the chemical route above by NaOH, and then immersing them in the nitromethane solution of $FeCl_3$. All the DMeO-PPV films have a thickness in the range 45–530 μm. Like PPV films, their thickness are not homogeneous, and the maximum difference of the thickness of one film is about 5%.

Measurements

Measurement of real conductivity and relative permittivity. The conductivity and permittivity are complex quantities:

$\sigma * = \sigma' + j\sigma''$ (σ' is the real part of the conductivity of the sample and σ'' the imaginary component).

$\varepsilon * = \varepsilon' - j\varepsilon''$ (ε' is the real part of the permittivity and ε'' the imaginary component).

On the other hand, σ^* is related to the relative complex permittivity ε^* by the relation: $\sigma * = jw\varepsilon_0\varepsilon*$, where w is the angular frequency of the applied field and ε_0 the vacuum permittivity.

In this paper, the data will be presented using traditional parameters: real conductivity $\sigma = \sigma' = w\varepsilon_0\varepsilon*$ and relative permittivity $\varepsilon = \varepsilon' = \sigma''/w\varepsilon_0$.

The measurements of σ and ε were carried out in the PIOM-ENSCP laboratory (Bordeaux, France) and were performed using the reflectometric technique. All experiments were monitored at room temperature using the pellet-cell on samples (films of thickness from 20 to 600 microns) with a sandwich structure. The σ and ε components were deduced from the measured parallel capacitance. In the frequency range 1 Hz to 10 GHz, the measurements were more than 20 experimental points per decade. At frequency up to 1 GHz, data were obtained by two automatic impedance analysers (HP 4191 and 4192) controlled by a HP 9826 computer. Above 1 GHz, a HP 8510 vectorial network analyser piloted by a HP 300 computer was used. All the details concerning measurements have been detailed in [47,48].

Reflectivity . The reflectivity of these films was calculated by the Laboratory X-RS (Orsay, France) from the experimental data obtained in the study of the permittivity, and the calculations were carried out in the frequency range 3–5 GHz depending on the samples. The limitation in frequency does not allow exploration of the radar absorption of the materials used (particularly at a frequency of 8–12 GHz). It is worth noting that the reflectivity coefficients presented in this paper correspond to the simulation of the properties of our materials. In fact, the signal attenuation of the thick films (millimetres thick) was calculated from the experimental measurements obtained on the thin films (about 10 microns thick) [49].

RESULTS AND DISCUSSION

Complex permittivity

The real part ε' and the imaginary part ε'' of the complex permittivity and the total conductivity σ ($\sigma = w\varepsilon_0\varepsilon''$) of PPV and DMeO-PPV were studied in the frequency range 45 MHz to 2 GHz. As for the total conductivity which derives from ε'' and can be expressed by the relation:

$$\sigma(w, T) = \sigma_{dc}(T) + \sigma_{ac}(W, T)$$

where σ_{dc} is the d.c. conductivity and σ_{ac} is the a.c. conductivity component. The importance of each component depends on the doping level, the sample temperature and the frequency region [50].

From the permittivity measurements, it has been shown that ε'' and ε' of both PPV and DMeO-PPV depend on the frequency, the conversion mode of the precursor as well as on the nature of both the polymer, the dopants and finally on the doping time and doping levels.

Evolution of dielectric parameters of PPV films doped with H_2SO_4 is shown in *Figure 1*: the relative permittivity ε or dielectric constant ε' decreases linearly while the dielectric loss ε'' decreases rapidly to 500 MHz and then slowly. It is worth noting that the precursor polyelectrolyte (assay 1) has a value of ε' which decreases regularly with frequency and is always superior to ε''; the latter becomes

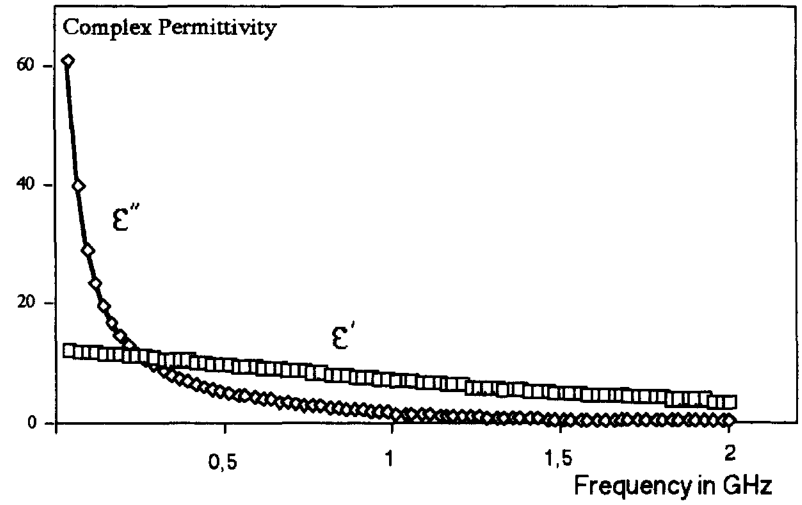


Figure 1 Frequency dependence of the complex permittivity of PPV obtained by pyrolysis of the sulfonium precursor at 150°C for 18 h and doped with H_2SO_4

Table 2 Electromagnetic properties (in frequency range up to 1 GHz) of PPV obtained via different conversion pathways of the sulfonium precursor, and doped with acids

Assays	Conversion and doping	ε^*		Conductivity (S cm^{-1})	Thickness (microns)
		0.1 GHz	(1 GHz)		
1	(Non-treated)	10.4 – *i*2.17	7.07 – *i*0.59	1.7 E-04	40
2	Pyrolysis 150°C 18 h^{-1} H_2SO_4 2 h 30 min	11.6 – *i*23.2	6.96 – *i*1.38	1.5 E-03	30
3	Chemical treat. H_2SO_4 2 h 30 min	Not measured	Not measured	(—)	(—)
4	Precursor deposited on textile fibre and 2 × -stretched. Chemical treat. H_2SO_4 2 h	(−7.3) – *i*250	115 – *i*106	2.1 E-02	160

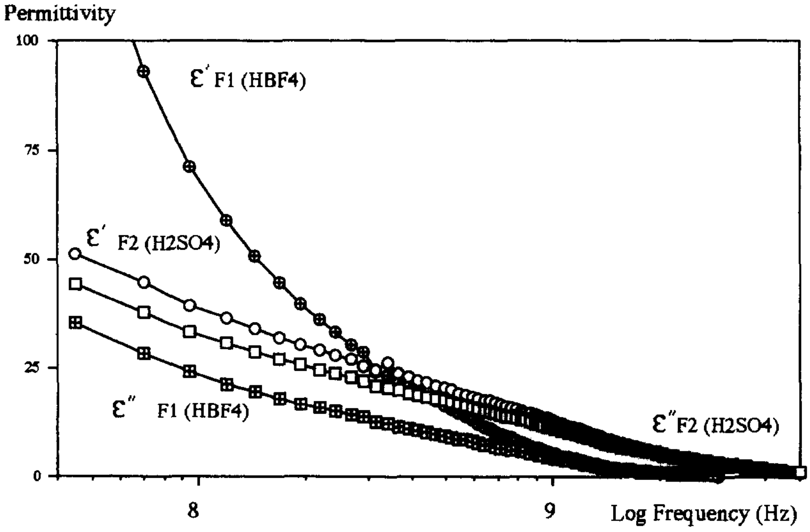


Figure 2 Frequency dependence of the complex permittivity of DMeO-PPV obtained by chemical treatment of the sulfonium precursor with HBF_4, and treated again with H_2SO_4

very small beyond 1 GHz. *Table 2* summarizes, at values of 0.1 GHz and 1 GHz, the electromagnetic properties in function and thickness of the PPV films obtained by different conversion and doping. The conductivity σ shown in this table is the highest value obtained in the studied frequency range.

PPV and DMeO-PPV doped with H_2SO_4 have almost the same conductivity range, though those of DMeO-PPV (assays 6–9) are a little higher than PPV's (assays 2 and 4). This can be explained by the methoxy-group of DMeO-PPV which reduces the ionization potential of the polymer, and therefore facilitates the doping.

The conversion of the DMeO-PPV sulfonium precursor by HBF_4 (assay 10) and doped again with H_2SO_4 (assay 11) leads to a product less conductive than that converted directly by H_2SO_4 (assays 7–9), and this is illustrated in *Figure 2*, which shows that the dielectric loss of DMeO-PPV films doped with HBF_4 is smaller than that of film doped again with H_2SO_4. On the other hand, the conductivity of DMeO-PPV, obtained via conversion by H_2SO_4 and dedoped by reacting with NaOH and doped again with $FeCl_3$, decreases with the doping time as shown in *Figures 3 and 4*. It seems that the dedoping by NaOH leads to a DMeO-PPV more sensitive to the degradation by oxygen, therefore their conductivities decrease with time (assays 14–16 and 18). The exceptionally higher

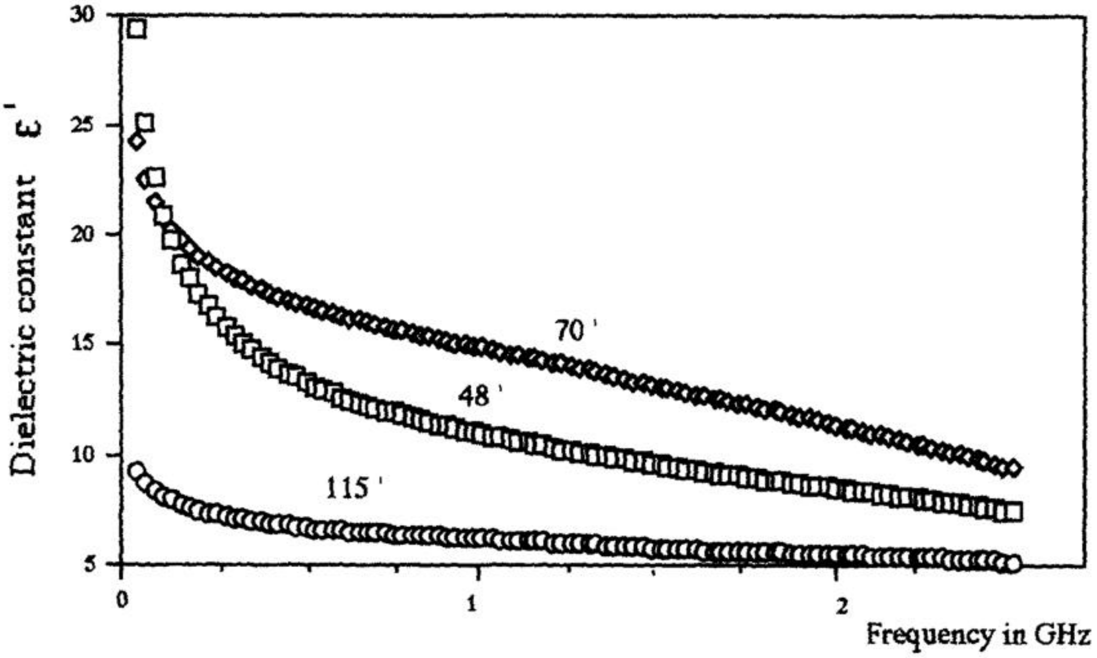


Figure 3 Frequency dependence of the dielectric constant ε' of DMeO-PPV obtained by chemical treatment of the sulfonium precursor by H_2SO_4, then dedoped by NaOH and finally doped with $FeCl_3$

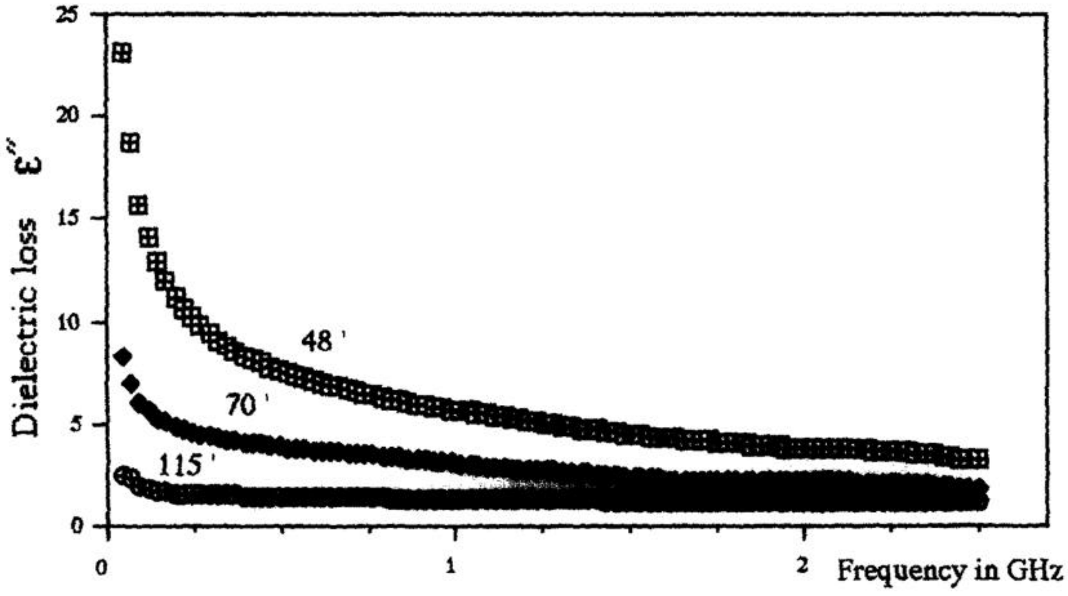


Figure 4 Frequency dependence of the dielectric loss ε'' of DMeO-PPV obtained by chemical treatment of the sulfonium precursor by H_2SO_4, then dedoped by NaOH and finally doped with $FeCl_3$

conductivity (assay 18) may be attributed to the greater thickness of the film as in assays 7 and 8. In general, ε' has values higher than ε''; however, a dispersity cannot be well explained. For example, as shown in *Figure 3*, the sample treated for 70′ has ε' higher than that of the sample treated for 48′, whereas all ε'' decreases regularly with the doping time (*Figure 4*).

It is noteworthy that all the PPV (assays 2 and 4) or DMeO-PPV polymers (assays 6–9) treated with H_2SO_4 are heavily doped, therefore their total conductivities are the predominant σ_{dc} which slightly change with frequency, whereas the DMeO-PPV treated with HBF_4 (assays 10 and 11) or doped with $FeCl_3$ (14–16 and 18) are moderately doped, so their conductivities are essentially σ_{ac} and change with frequency.

Finally, the sulphonium precursors were also investigated. The conductivities of these precursors are essentially ionic in nature, however, even at room temperature and in air these precursors are partially converted into corresponding polymers, so their conductivities shown in *Tables 2 and 3* are considered to be related to the dielectric loss (assays 1 and 5).

Tables 3 and 4 give the highest values of conductivity σ in the frequency range studied and the electromagnetic

Table 3 Electromagnetic properties (in frequency range up to 1 GHz) of DMeO-PPV obtained by different conversion pathways of the sulfonium precursor and doped with different dopants

Assays	Conversion and doping	ε^*		Conductivity ($S\ cm^{-1}$)	Thickness (microns)
		0.1 GHz	1 GHz		
5	(Non-treated)	4.81 − *i*3.65	2.88 − *i*1.22	2.7 E-04	140
6	Pyrolysis: 200°C 17 $h^{-1}H_2SO_4$ 1 h	2253 − *i*3376	153 − *i*(− 79)	1.9 E-01	60
7	Chemical treat.H_2SO_4 30 min	−282 − *i*4441	−251 − *i*445	3,0 E-01	530
8	Chemical treat.H_2SO_4 30 min	225 − *i*389	241 − *i*179	2.8 E-02	90
9	Chemical treat.H_2SO_4	467 − *i*1334	76 − *i*558	9.4 E-02	170
10	Chemical treat.HBF_4 3 h	30.4 − *i*21.2	10.9 − *i*3.99	1.6 E-03	45
11	Chemical treat.HBF_4 3 hH_2SO_4 30 min	36.1 − *i*59	12.5 − *i*4.94	4.0 E-03	60

Table 4 Electromagnetic properties (in frequency range up to 1 GHz) of DMeO-PPV obtained by conversion of the sulfonium precursor by H_2SO_4 and treated with NaOH (to dedope acid) and doped with $FeCl_3$

Assays	Doping time (min)	ε^*		$\sigma(S\ cm^{-1})$	Thickness (microns)
		0.1 GHz	1 GHz		
12	5	Not measured	(—)	(—)	(—)
13	18	Not measured	(—)	(—)	(—)
14	48	20.9 − *i*14.1	10.9 − *i*5.72	1.6 E-03	260
15	70	20.7 − *i*5.68	14.9 − *i*3.10	4.5 E-04	200
16	115	8.08 − *i*1.91	6.19 − *i*1.41	1.5 E-04	300
17	158	Not measured	(—)	(—)	(—)
18	190	27.2 − *i*8.35	19.3 − *i*7.95	6.8 E-04	420

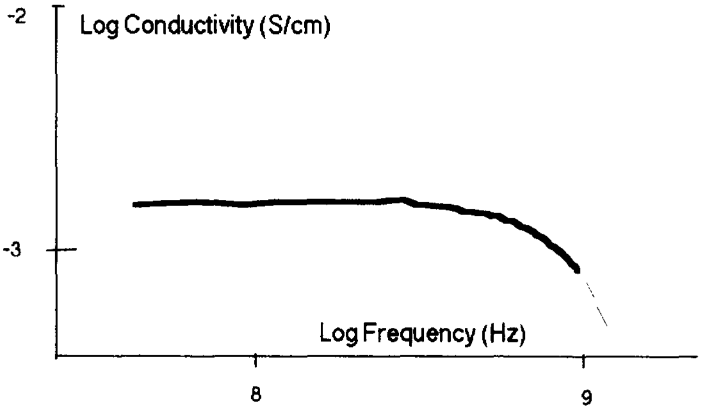


Figure 5 Evolution with frequency of conductivity σ of PPV obtained by pyrolysis of the sulfonium precursor at 150°C for 18 h and doped with H_2SO_4

properties of DMeO-PPV after the conversion mode of the precursor, as a function of thickness of the films, doping at two frequencies: 0.1 GHz and 1 GHz.

The fact that σ has a tendency to decrease at a high frequency range (*Figure 5*) is supposed to be due to the contact effects [50] and/or to the migration of the dopant to the surface of the sample that perturbs the measurement. These effects are observed for both the samples of PPV and DMeO-PPV, and it is difficult to control. Thus, we cannot estimate the error of the measurement, although they can be minimized by evaporating gold on the sample.

Reflectivity simulation

The reflectivity was calculated as described above. Thus the results of reflectivity coefficient R (in decibels, dB) presented as a function of frequency (GHz), or of absorber thickness, reflect the performance of the materials as absorbers for an optimized thickness.

Almost all the PPV and DMeO-PPV samples have displayed a conductivity range required for EMI shielding, because after [23], for a conducting compound, a conductivity in the range 10^{-3}–10^{-1} S m^{-1} can enable an appropriate impedance surface to be obtained, i.e. an absorbing material. Furthermore, to attenuate the electromagnetic wave by an absorber of weak thickness and low reflection coefficient, the material should have at 10 GHz a conductivity of about 10^{-2} S cm^{-1} [1]. And to reach both a.c. and d.c. conductivities at room temperature, light doping levels must be used [50].

Films of PPV and DMeO-PPV give resonant absorption which is characterized by a deep absorbance with narrow

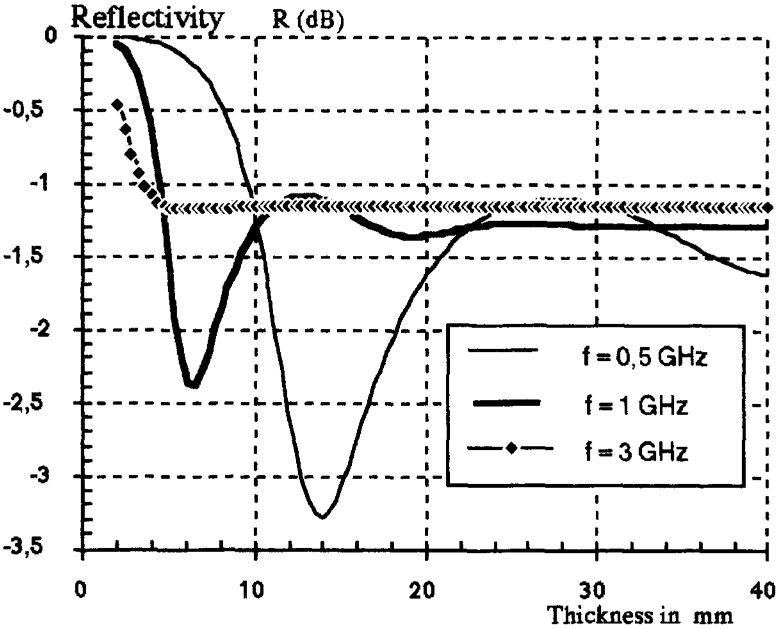


Figure 6 Curves of calculated reflectivity twofold-stretched-PPV obtained by stretching precursor deposited on fibre textile and treated with H_2SO_4

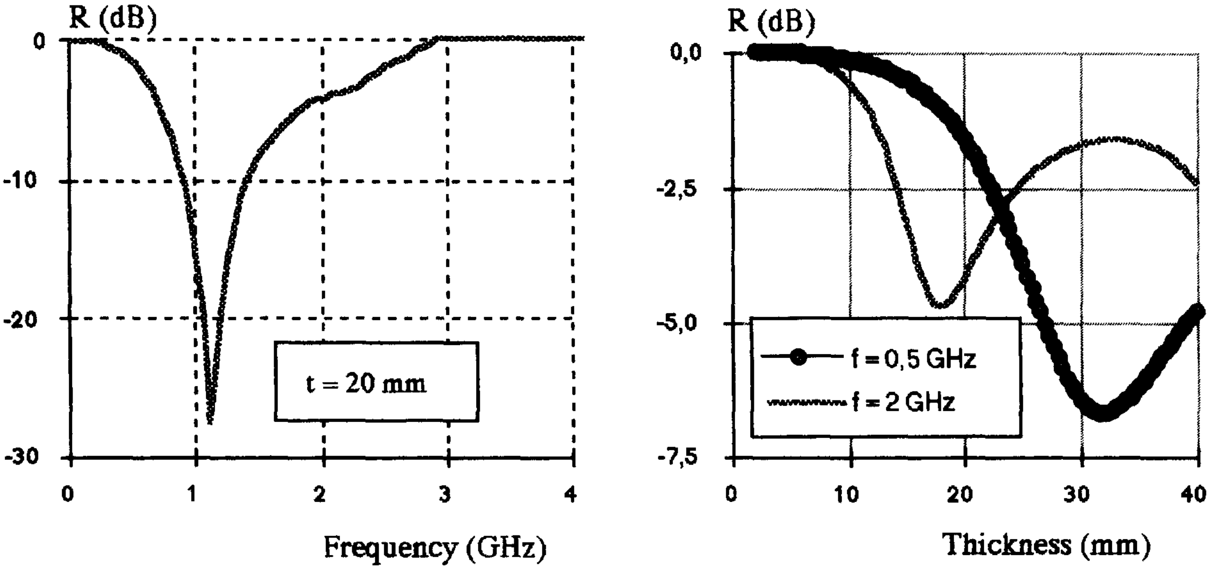


Figure 7 Calculated reflectivity of DMeO-PPV obtained by treatment of the precursor with HBF_4 and treated again with H_2SO_4

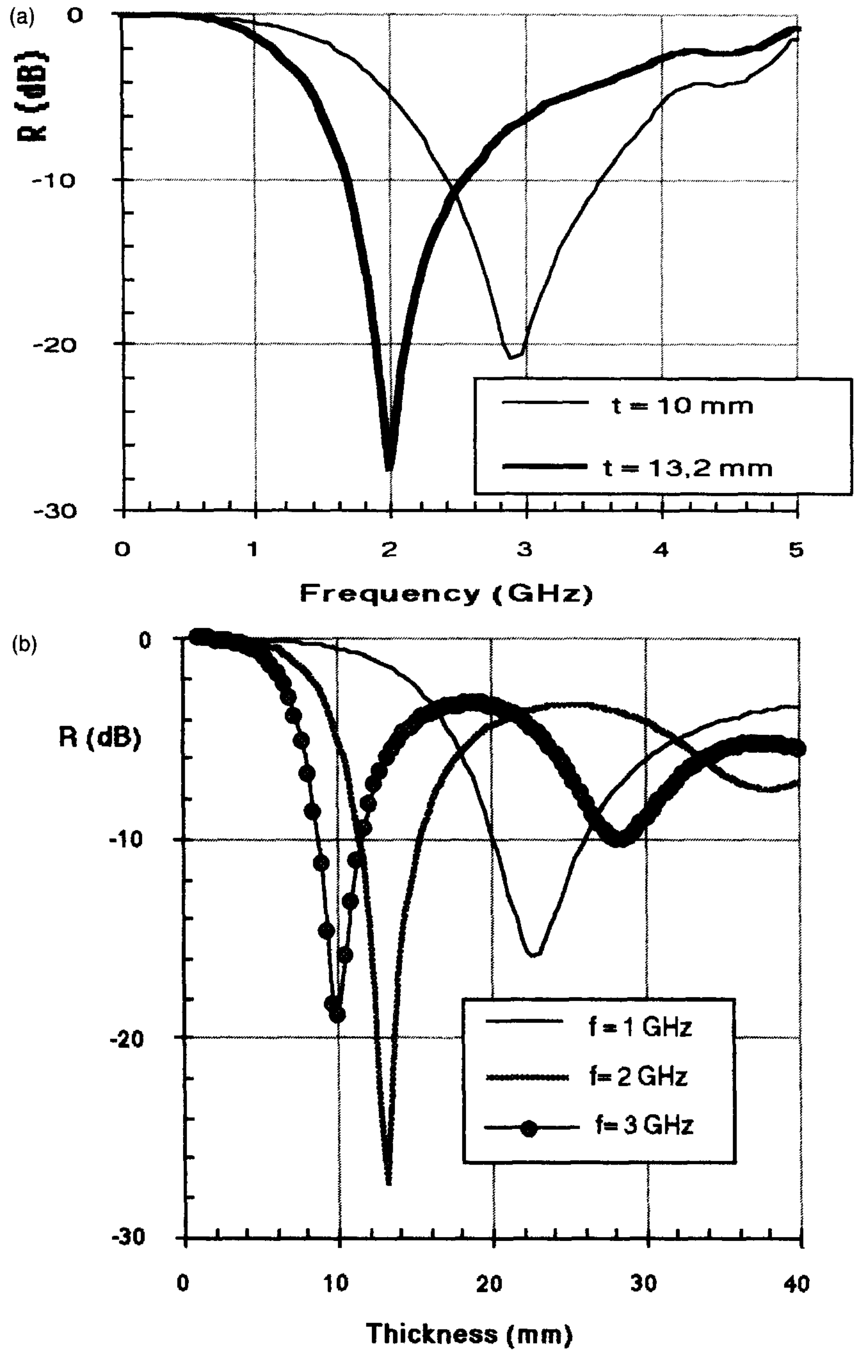


Figure 8 Calculated reflectivity as a function of frequency (a) and of thickness (b) of DMeO-PPV treated with $FeCl_3$ for 48 min

half-width. The deep absorbance can be explained as due to the thickness and the dielectric loss ε'' of the material simultaneously. In fact, the coefficient of reflectivity R (dB) of PPV (for both PPV films or PPV deposited on textile fibre) and DMeO-PPV, all doped with H_2SO_4, is not low (absorbance is not high) because their dielectric loss ε'' is important (*Tables 2 and 3*). *Figure 6* shows the thickness and frequency dependence of R (dB) for PPV deposited on textile fibre. Here, the absorbance is still weak because ε'' remains important.

In contrast, DMeO-PPV obtained by the conversion of precursor using HBF_4 and doped with H_2SO_4 has a more significant attenuation (*Figure 7*). However, all DMeO-PPV samples doped with $FeCl_3$ present a much more important absorbance as illustrated by *Figures 8–10*. It is to be noted that a signal attenuation of -20 dB corresponds to a reflected energy of 1%, or to a dissipation of 99% of energy of incident radiation. It shows clearly that the absorbance depends mostly on the nature of the dopant and the doping level of the materials. The small absorbance of PPV or DMeO-PPV is due to their high doping levels with H_2SO_4 (assays 2, 4 and 6–9) which result in a predominant d.c. conductivity and, for this reason, the material becomes electrically conductive, whereas the high absorbance is ascribed to the lightly doped material which gives a.c. conductivity being appreciated at high frequency.

As observed, only thick samples give an interesting shielding effect, therefore the potential applications of these materials in EMI shielding are compromised. However, since the simulation of reflectivity coefficient cannot be carried out at a frequency higher than 5 GHz, it is expected that PPV and DMeO-PPV can be used as absorber materials

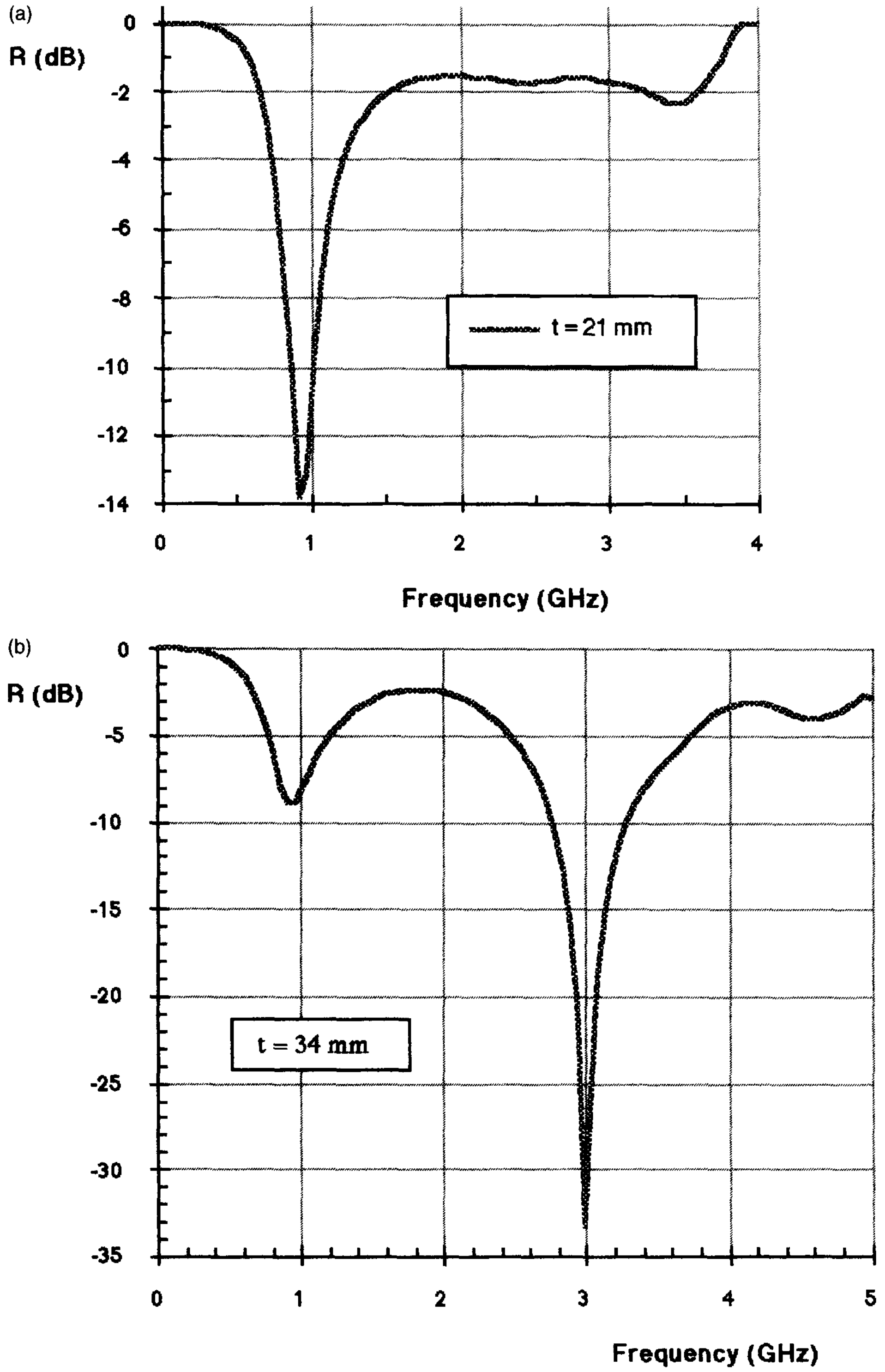


Figure 9 Calculated reflectivity as a function of frequency of DMeO-PPV treated with $FeCl_3$ for 70 min (a) and for 115 min (b)

at a higher frequency range owing to the fact that when the film thickness decreases, the absorption peak is shifted to a higher frequency and its half-height width changes also, as shown by *Figures 6–8a,b* and *Figure 10*, this phenomenon has been observed for other blends [51].

CONCLUSION

The complex permittivity of poly(p-phenylene-vinylene) (PPV) and poly(dimethoxy-phenylene-vinylene) (DMeO-PPV) has been studied in the frequency range 10 kHz–10 GHz. The hyperfrequency absorbance of these materials was calculated from the permittivity data obtained, but the simulation was carried out at a limited frequency range up to 5 GHz.

Although all these polymers have a conductivity satisfactorily required for an electromagnetic absorber, they show a difference in shielding effectiveness in the frequency range studied. The doping agents and the doping levels play a predominant role in the behaviour of the permittivity and the absorbance of both the polymers used. In fact, films heavily doped with H_2SO_4 give high permittivity, and d.c. conductivity which varies slightly with frequency, and thus a very low absorbance. In contrast, DMeO-PPV films lightly doped either with HBF_4 acid or

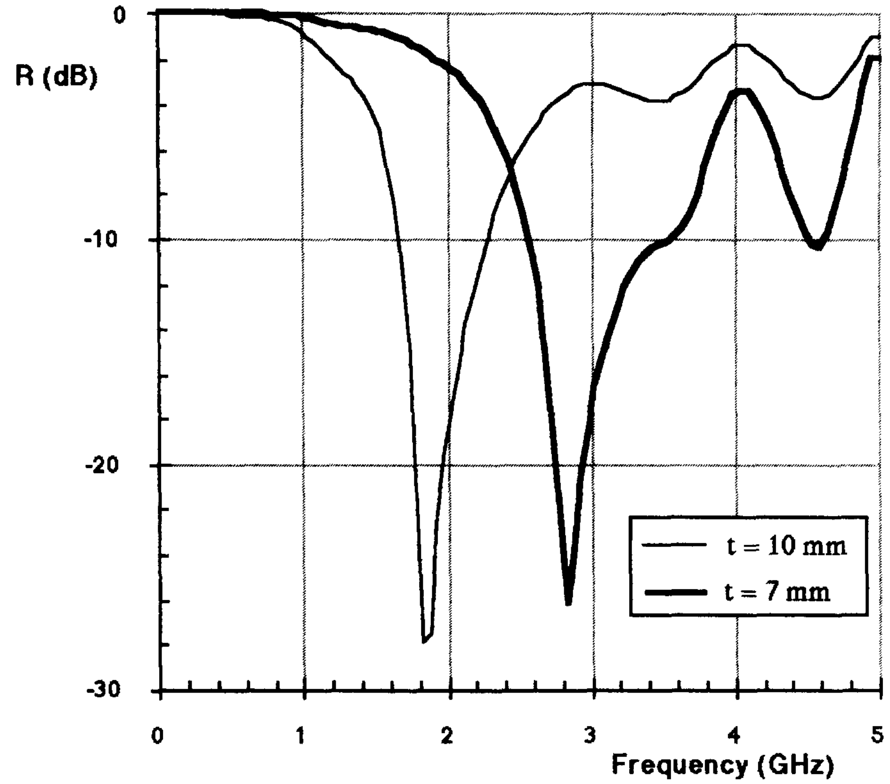


Figure 10 Calculated reflectivity as a function of frequency and of thickness of DMeO-PPV treated with $FeCl_3$ for 190 min

with $FeCl_3$ give moderated values of permittivity, or a.c. conductivity which becomes appreciated at high frequency, and gives rise to higher absorbance which corresponds to more than 99% absorption of radiation. Nevertheless, this happens only with very thick samples, so the practical use of these materials as an EMI shielding absorber may be expected at a frequency range higher than the one used in the simulation, because the film thickness becomes smaller.

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