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# Electrical, textural and structural characterization of EMIMAc silica ionogels and their corresponding aerogels

N.Bengourna\*, L.Bonnet<sup>1,2</sup>, R.Courson<sup>†</sup>, F.Despetis<sup>1,2</sup>, N.Olivi-Tran<sup>1,2</sup>, P.Solignac<sup>1,2</sup>, H.Satha\*

<sup>1</sup>*Laboratoire Charles Coulomb, UMR CNRS 5221, cc 074*

*Place Eugène Bataillon, 34095 Montpellier cedex 5, France*

<sup>2</sup>*Laboratoire Charles Coulomb, Université Montpellier 2*

*Place Eugène Bataillon, 34095 Montpellier cedex 5, France*

*\*Laboratoire d'Analyses Industrielles et Génie des Matériaux, Département de Génie des Procédés, Université 8 mai 1945, BP 401, Guelma, Algeria*

*†Laboratoire d'Analyse et d'Architecture des Systèmes, 7 Avenue du Colonel Roche, 31 077 Toulouse cedex 4, France*

**ABSTRACT:** Silica gels were synthesized from TMOS and MTMS in different proportions changing the ionic liquid concentration. EMIMAc have significant influence in the gelation processe. The textural characterizations showed an effect of these concentrations on the corresponding aerogels: pore size distributions and effectives surfaces. The structure of the aerogels was measured with a SAXS apparatus and was typical of acid catalyzed aerogels. Conductivity measurements, operated on the ionogels, were carried out using an original electrical 4 electrodes set up which allowed us to study the ionic conductivity at the pore scale. The results obtained showed that one can model the electrical temporal response of the EMIMAc silica ionogel with a RLC series circuit.

## 1 Introduction

Ionic liquids (IL) are molten salt which are liquid at a temperature lower than 100°C. Since the first IL synthesis, chemists and material scientists have been highly interested in this new type of conducting liquids because of their numerous properties and potential applications like green solvent, electrolytes, sensors, catalysts or batteries ...[1, 2, 3].

Moreover, it is possible to synthesize nano-composites materials mixing IL with silica precursors, via a sol gel route. The first ionogel (for gel synthesized with an IL) was obtained in 2000 by S.Dai [4]. Several investigations led to a variety of materials [5, 6, 7, 8] implying numerous characterizations: porous volume, fractal dimension, conductivity of the ionogel bulk ... [ 9, 10, 11, 12, 13, 14].

Asymmetric cation, N,N'dialkylimidazolium or 1-butyl-3-methylimidazolium associated with anions such as tetrafluoroborate, were the most commonly used IL for a few years [9, 15].

Here we chose to work with another imidazolium cation, 1-ethyl-2-methylimidazolium bonded to the anion acetate (EMIMAc). This IL is known to be a good solvent for cellulose and it exhibits a very high electric conductivity (2500 $\mu$ S/cm) at ambient temperature. Compared to halogen anions, this IL is less corrosive and toxic.

The present study is interested in the synthesis and the characterization of EMIMAc silica ionogels and their corresponding aerogels. The first characterization is an electrical one in order to study the effect of the ionogel pore walls on the ionic conductivity: by an original 4-electrodes set up, we measured the temporal response of our ionogels to a voltage pulse and we modeled these responses by a RLC circuit. Up to now, only two studies on ionogels conductivity were performed. One of these references dealt with two plate electrodes [10, 16] and the other with a 3-electrodes set up (17). Textural and structural characterizations as nitrogen adsorption/desorption and SAXS were then carried out on the aerogels.

## 2 Experimental setup

### 2.1 Synthesis of the ionogels and aerogels

#### 2.1.1 Chemicals

The ionic liquid: 1-Ethyl-3-methylimidazolium acetate (EMIM Ac) of purity  $\geq 90\%$ , and the silica precursors, tetramethoxysilane TMOS (98%) and methyltrimethoxysilane MTMS (98%), were from Aldrich.

Technical grade ethanol was used to extract water and IL from the gel before the CO<sub>2</sub> supercritical drying. Because of the hydrophilicity of IL, this last was heated under vacuum (70°C, 10 mbar) for 48h before each synthesis.

Gels were prepared via an hydrolytic one step process by mixing two solutions: solution (1) with TMOS and MTMS in different molar ratios and solution (2) containing IL and acidic water (HCl concentrations equal to 0.65N). This way was preferred in order to avoid the important shrinkage which is obtained using a non-hydrolytic process via formic acid. The molar ratio  $n_{IL}/n_{Si}$  was between 0.3 and 0.6 and for each sample, the hydrolysis molar ratio  $n_{water}/n_{Si}$  was equal to 16. After stirring, the mixture was poured into the conductivity cell and in molds for a further characterization of the ionogels. Gelling occurred after a time which depended on the HCl concentrations, on the IL ratio and the MTMS ratio. EMIMAc is a basic IL and despite of the dilution effect when the quantity of IL increased, the pH also increased and the gelation time decreased. Gels were transparent disks with a diameter of 16mm and a height of 4 mm without any cracks. All these steps were done at  $22 \pm 2^\circ\text{C}$ .

Table 1 lists the different samples synthesized by varying proportions of IL, MTMS and HCl concentration.

#### 2.1.2 CO<sub>2</sub> supercritical drying

The knowledge of the texture and the structure of (the) ionogels imply their drying under conditions which allows to obtain dry materials exhibiting properties very close to these(s) of (the) ionogels. For

these reasons, drying must be carried out under supercritical conditions to avoid capillary stresses which would lead to dense and cracked materials. For the same reasons and to avoid the dissolution/redeposition phenomenon which occurs at high temperature and which can induce textural modifications at a very low scale [18], we chose to dry the gels under supercritical CO<sub>2</sub> so-called COLD process.

The wet gels were aged during 12h at 22±2°C. For a further CO<sub>2</sub> supercritical drying, the liquid that impregnates the wet gels must exhibit a total miscibility with CO<sub>2</sub>. So, gels, which contained water and the IL, were first soaked into ethanol for 15 days to obtain gels with pores mainly filled by ethanol. Then ethanol were exchanged with liquid CO<sub>2</sub> at 8°C and 6 MPa during 5 hours. It is worth noting that IL is probably not totally removed from the gel during the soaking with ethanol and that one part of the IL is extracted by CO<sub>2</sub>. Finally, temperature and pressure were raised to 38°C and 10 MPa and after 10min, the pressure was released slowly during 12h.

Samples obtained were translucent (figure 1) without any cracks and exhibited a shrinkage which depended on the composition of the gel.

## **2.2 Characterization methods**

### **2.2.1 Electrical conductivity measurements**

These measurements were performed on ionogels using a new set up described on figure 2.

The cylindrical cell allowing these measurements consisted of a Teflon mold containing the ionogel and of 4 Pt-electrodes partially immersed in the ionogel and passing through the sides of the mold.

The 4 electrodes were Pt wires with a section of 0.5 mm. These wires were immersed in the bulk of gels along 7mm and fixed to the mold to make sure that all the measurements were done in the same conditions. The mold was cylindrical with a depth of 5 mm and a diameter of 15 mm. The 4 wires were

connected to a pulse generator (Aligent 33220A, 20MHz, Arbitrary waveform generator) and to an oscilloscope (TDS2022C, 200MHz, Tektronik).

A rectangular voltage pulse was applied between 2 (channel 1) of the 4 electrodes. Then the potential difference was measured between these 2 electrodes (channel 1) and between the 2 other ones (channel 2).:-

First, we measured the electrochemical domain of stability of the EMIMAc. We found that for an applied potential equal to 4V and an offset equal to -1.375V, there was oxydo-reduction of the IL at room temperature. We checked that for an applied potential equal to 2.5V and an offset equal to -1.25V, the IL was stable. Straightforwardly, we chose to study the temporal response of our ionogel samples at this voltage.

The voltage pulses had an absolute value of 3.75V (applied potential 2.5V+ offset 1.25V), a frequency of 50HZ and pulse durations of either 20ns or 100ns

This allowed us to measure the electrical response of the solvent contained in the porous network of the ionogels. Indeed, as the diameter of the electrodes is smaller than the depth of the set up (i.e. smaller than the height of the ionogel cylinder), these electrodes were always in contact with the IL contained in the pores. This lead to an electrical characterization of our ionogel samples at a microscopic scale. Moreover, as we applied rectangular voltage pulses to the ionogel, we were able to analyze the temporal response of the sample.

In order to clearly identify the role of the silica network, a blank was first done on EMIMAc + HCl solution. Then electrical conductivity measurements were performed on the ionogel samples within 1 hour after gelling occurred.

### 2.2.2 Textural and structural characterizations of the aerogels

The bulk densities of the samples  $\rho_a$  were evaluated from their weight and linear dimensions.

For the textural characterization, nitrogen adsorption/desorption isotherms at 77K were carried out on a Micromeritic ASAP2010. Samples were outgassed under vacuum during 16h at 50°C.

The specific surface area was obtained using BET theory. Its accuracy was about 4%. For rigid porous materials, when the nitrogen relative pressure  $P/P_0$  gets close to 0.99, the volume of adsorbed nitrogen must correspond to the porous volume [19]. The dwell time, which is the interval of time allowing a pressure change of 0.01%, was 10 seconds. The pore size distributions was derived from BJH method from the desorption branches of the isotherm.

SAXS (small angle X ray scattering) and WAXS (wide angle X ray scattering) experiments were performed with an in-house setup of the *Laboratoire Charles Coulomb*, using a X- ray tube GeniX<sup>3D</sup> from Xenocs and a Schneider 2D imageplate detector prototype, in order to estimate the fractal dimension of the materials and the size of the constitutive particles and aggregates.

## 3 Results

### 3.1 Conductivity: electrical characterization

Some results of electrical characterization of the ionogels lead to figures 3 and 4. The maximum of the measured amplitudes on both channel 1 and 2 are presented in table 3 for all the samples.

- For an input pulse duration of 20ns, the maximum of the response on channel 1 for all the ionogel samples was close to the value of the input voltage, within experimental fluctuations
- For an input pulse duration of 100ns, the maximum of the response on channel 1 for all the ionogel samples was larger than the input voltage

- For both durations, on channel 2, all the responses were damped.
- For the system EMIMAc + HCl solution, the maximum of the response on channel 1 was lower than the input pulse but with oscillations whatever the pulse duration.
- Samples A, B, C, D, E have the same response on channel 1 within experimental variations
- Samples B,C, D, E have the same response on channel 2 within experimental variations
- Sample A has a response on channel 2 which is damped with respect to samples B, C, D, E and EMIMAc + HCl solution.

An electrical modeling of these responses is presented in section Discussion.

### 3.2 Textural and structural Characterizations

The relative shrinkage which occurred during the different steps, from the gel to the aerogel, and the characteristics of the aerogels are shown in table 2.

Most of the samples exhibited low densities and high specific surface areas. The specific surface area showed a tendency to increase up to a maximum value with the higher ratio of  $n_{\text{MTMS}}/n_{\text{TMOS}}$  while it was quite similar whatever the IL content.

A divergence between the measured porous volume  $V_{\text{pm}}$  and the calculated one  $V_{\text{pc}}$  was observed. This divergence, which clearly increased when the bulk density decreased, was linked to the aerogel strength and corroborated that the pore volume could not be easily obtained by nitrogen adsorption – desorption, as reported in the literature [19]. We must also note that some IL probably remained in the aerogels (< 5% weight), as already described in the literature [11].

Nitrogen adsorption isotherms, given in Figure 5 for three compositions are of type IV. The pore size distributions, reported in Figure 6a and b, are given as an indication as it is known that these data carry



some incertitude. However, they corroborate that materials are mesoporous ones. Aerogel made without MTMS displayed a broad pore size distribution. This distribution is increasingly narrow and shifted to lower diameters when the MTMS ratio is increasing (Figure 7a). However, distributions are slightly the same with the IL ratio increase (Figure 7b).

All the ionogels synthesized are fractals ones even if the fractal domain is relatively small (one decade)(figure 5).The fractal dimension is between 2.2 and 2.5, values which are characteristics of acid catalyzed aerogels. Samples C,D and E exhibit the same behavior with a larger fractal domain than sample A or B, and a fractal value around 2.5.

## 4 Discussion

First, we can note, that the presence of MTMS (hydrophobic groups) in the sol postpones the hydrolysis and condensation reactions and thus increases the gelation time.

It is worth noting that for all the samples investigated except one (sample A), the shrinkage after drying is rather low (<10 %). For sample A, which is obtained via a synthesis without MTMS, the soaking governs the shrinkage amplitude. Capillary tensions induced by IL exchange are probably too high comparatively to the strength of the silica network. We can make the hypothesis that the affinity of the IL for the silica surface increases these tensions and probably prevents IL from being completely removed. For others samples, the MTMS induces a repulsive wall/liquid effect (stable  $\equiv\text{Si-CH}_3$  groups on the surface of  $\text{SiO}_2$  particles) and then reduces the capillary tensions. Consequently shrinkage decreases slightly as the increase of the MTMS content.

Though the bulk density of the samples C, D and E varies from 0,16 to 0,2 g.cm<sup>-3</sup> depending on the IL ratio, their specific surface area does not depend on the density. Their structure and texture are very similar (Figure 5b, Figure 7b and table 2).

On sample A, B and C, the methyl modification obviously acts on the specific surface area which increases with MTMS ratio. For the sample without MTMS, the fractal dimension is slightly smaller and the fractal domain is narrower. MTMS reduces cross-linkage and make the clusters grow slowly [21]. Regarding the aerogel texture, it is clear that as the  $n_{\text{MTMS}}/n_{\text{TMOS}}$  value increases the pore size distribution decreases towards the smaller pore sizes and becomes narrower [22].

EMIMAc and MTMS induce noteworthy changings in the textures or structures of the aerogels, the effects on conductivities are shown below.

The electrical response of our ionogels samples is typical of a RLC series circuit. We show here in figures 8 a few examples of RLC series circuits signals to which a rectangular voltage pulse is applied. The shape of the response which is taken at the capacity component (C) in the simulation depends on the duration of the voltage pulse: if the duration of the input pulse is too short, the sample is in the electrical transient state, this is the case for pulses durations equal to 20ns in the experiments and 0.1s in the simulations. For longer pulse durations (here 100ns in the experiments and 0.4s in the simulations), the transient state is almost finished. In the case of responses measured on channel 2 (see section Results), the damping is due to the resistance of the IL either contained in the ionogels pores or in the HCl + IL solution.

This may be seen also in the simulations: fig.8c represents the measured voltage on channel2. Although our simulations can only be qualitatively compared to the measured voltage on our ionogels, one may compare the parameters of fig.8a and 8b which correspond to fig.3b and fig.4b on channel1, to fig.8c which corresponds to channel2 in fig.3 and 4. The resistance leading to fig.8c is 15 times higher than

for the other simulations. This means that on channel 2 the measured temporal resistivity is much larger than on channel 1. Indeed, the voltage is applied perpendicularly to channel2, so the displacement of the ions is limited between electrodes of channel2.

What is worth noting, is the difference between the responses of the ionogels samples and that of the HCl+IL solution: the highest amplitudes of the ionogels electrical responses on channel 1 are always larger than the responses of the HCl+IL solution. Let us make a simple model to explain this difference: the IL molecules in the solution have a larger mean free path because they are not embedded in a silica matrix but the direction of their displacement is random (Brownian motion). The same IL molecules when embedded in a ionogel pore, have a mean free path which is smaller (due to the pore walls) but their motion is oriented (in the pore direction). So when a voltage difference is applied, the charge carriers (IL ions,  $H^+$  and  $Cl^-$  ions in the ionogel pores) have a collective motion which leads to an appearing smaller resistance than the HCl+IL solution (when the applied potential and the measured electrical response are parallel).

To understand all the characteristics of ionic conduction in nanometric pores it would be necessary to perform numerical simulations by Brownian dynamics or Monte Carlo simulations.

To conclude this part of the discussion, we may say that we found an original method to characterize ionogels electrically. This method can be applied to other types of ionogels (with a different IL, different IL ratios etc.).

## 5 Conclusion

We applied a rectangular voltage pulse to EMIMAc silica ionogels via a 4 electrodes set up : the temporal voltage response of our samples may be modeled by RLC series circuits. By comparison with an EMIMAc+HCl solution, the resistivity of the ionogels depends on the porous texture. Indeed, the ions within the ionogels pores have a collective motion induced by the pore walls contrarily to that in the EMIMAc+HCl solution where only thermal agitation acts. Indeed, textural characterization of the corresponding aerogels shows a significant effect of the TMOS/MTMS ratio on the pore network. Finally, the structure is typical of acid-catalyzed aerogels.

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# 1 Table list

Sample	$n_{IL}/n_{Si}$	molar ratio $n_{MTMS}/n_{TMOS}$	Concentration HCl (N)	Sol pH	Gelation time
A	0.3	0	0.65	4.5	25 min
B	0.3	0.2	0.65	4.5	35 min
C	0.3	0.4	0.65	4.5	2h40
D	0.45	0.4	0.65	4.5	1h40
E	0.6	0.4	0.65	5.5	1h

Table 1: Compositions and gelation times for the studied samples

Sample	Shrinkage after soaking $\pm 0.1$ (%)	Shrinkage after drying $\pm$ 0.1 (%)	Bulk density $\rho_a \pm 0.002$ ( $g \cdot cm^{-3}$ )	Calculated* Pore volume $V_{pc} \pm 0.1$ ( $cm^3 \cdot g^{-1}$ )	Desorption BJH Pore volume $V_{pm}$ ( $cm^3 \cdot g^{-1}$ )	$V_{pm}/V_{pc}$ (%)	Specific Surface Area ( $m^2 \cdot g^{-1}$ )
A	13.6	14.3	0,288	3.0	2,82	95	$640 \pm 25$
B	3	8.1	0,192	4,7	3,14	67	$885 \pm 35$
C	6	9.3	0,198	4,5	2,94	65	$1090 \pm 40$
D	6.6	7.8	0,173	5,3	3,29	62	$1040 \pm 40$
E	6.4	7	0,167	5,5	3,22	59	$1050 \pm 40$

Table 2: Shrinkage observed after each step of the drying and characteristics of the aerogels.

\*  $V_{pc}$  is calculated from the bulk density and the skeletal density of silica which has been estimated in the literature to about  $2 g \cdot cm^{-3}$  in silica aerogels [16]

Sample	20 ns		100 ns	
	Channel 1	Channel 2	Channel 1	Channel 2
A	3.56	0.13	4.08	0.29
B	3.58	0.68	4.08	1.54
C	3.56	0.67	4	1.52
D	3.52	0.58	3.88	1.44
E	3.52	0.536	3.84	1.38
EMIMAc+HCl	3.46	0.36	3.56	0.92

Table 3: Conductivity results for two voltage pulse durations 20ns and 100 ns measured on channel 1 and channel 2.

## 2 Figure list



Figure 1: Photographs of aerogel synthesized, samples (A) and (C)

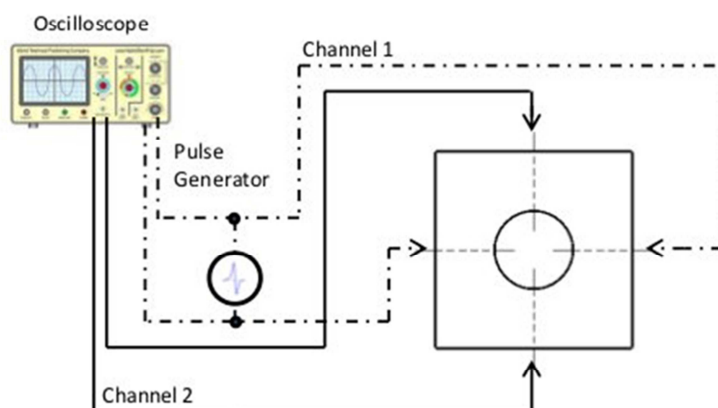
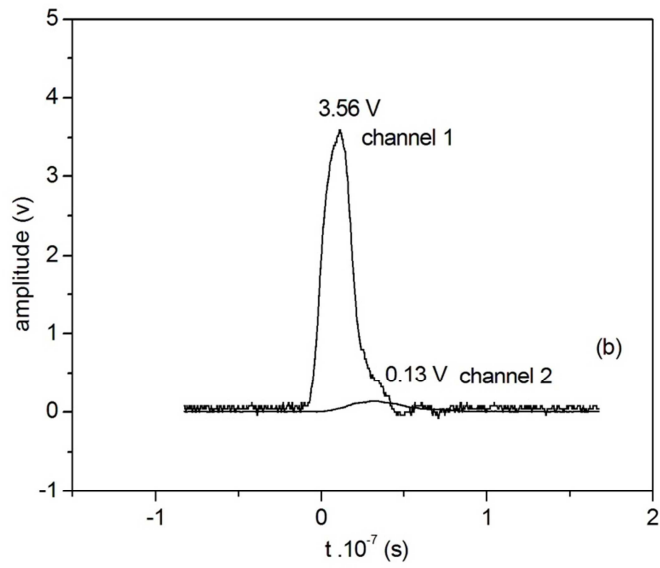
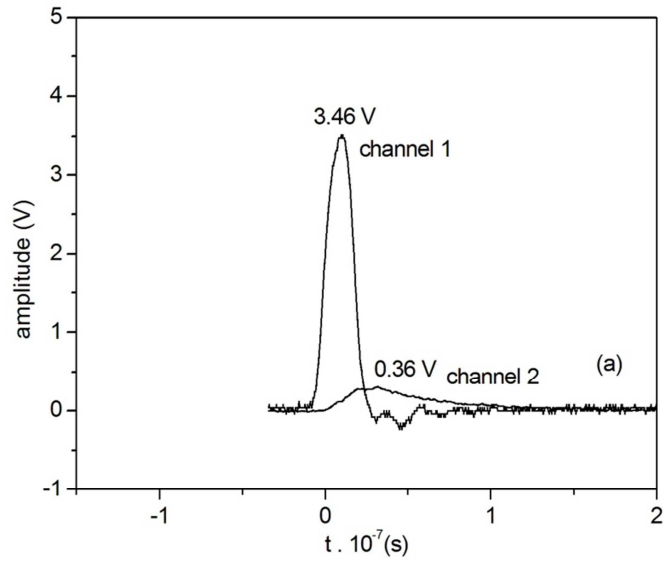
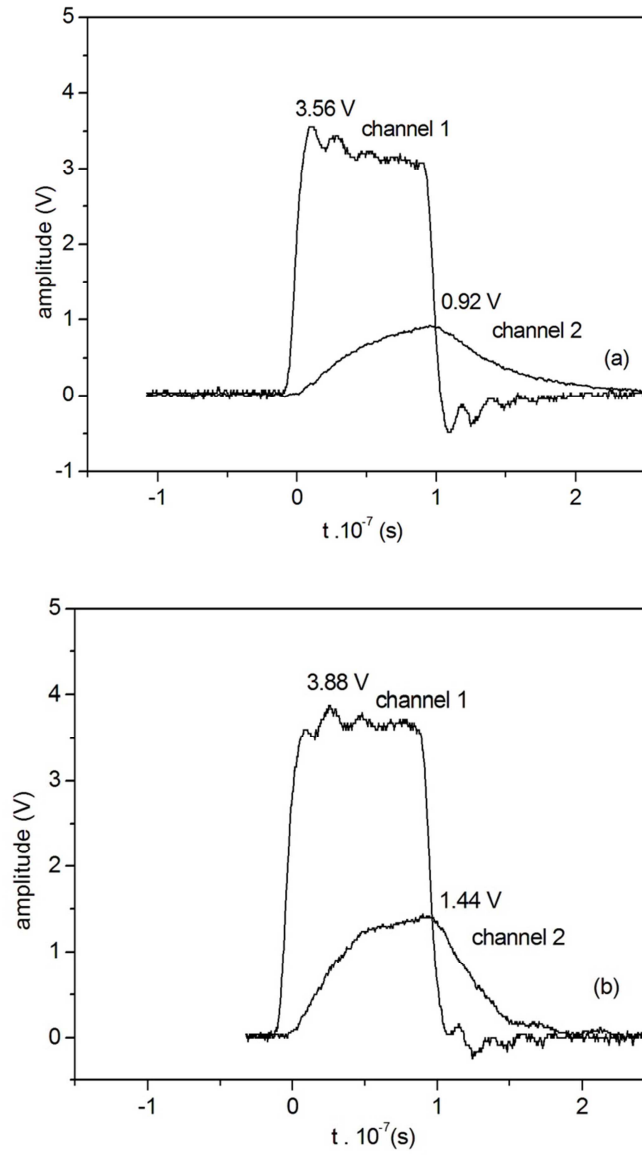


Figure 2: Experimental set-up for the electrical characterization of the ionogel. The ionogel sample is represented by the disk with 4 electrodes getting through the square mold.



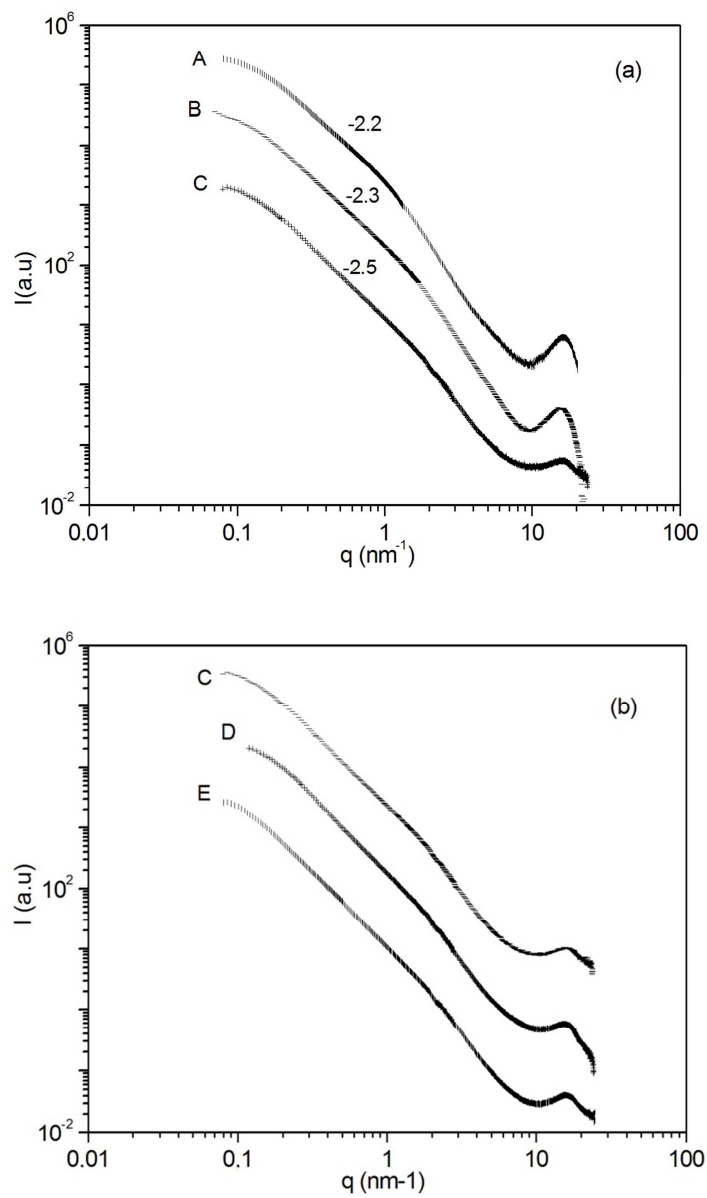


**Figure 3:** electrical temporal responses to a rectangular voltage pulse (for a total amplitude of  $V=2.5V$  and a duration of  $t=20$  ns) : (a) for an EMIMAc + HCl solution (b) for sample A

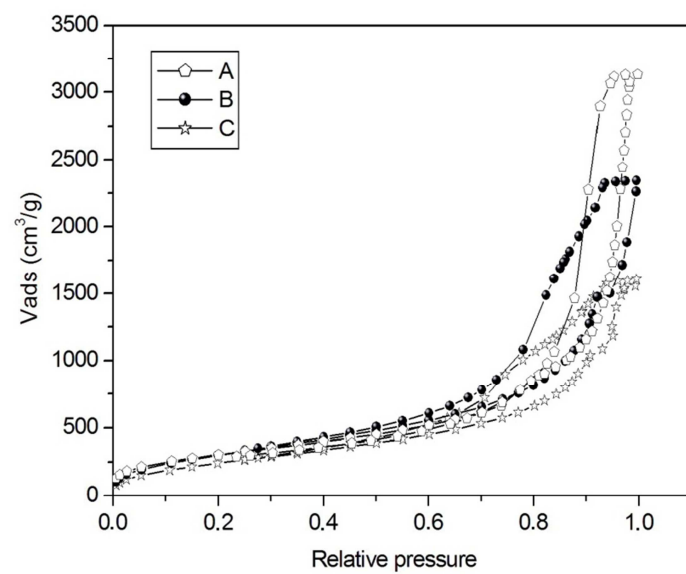


**Figure 4:** Electrical temporal response to a rectangular voltage pulse (of total amplitude 3.75 V and duration 100 ns) (a) for an EMIMAc + HCl solution

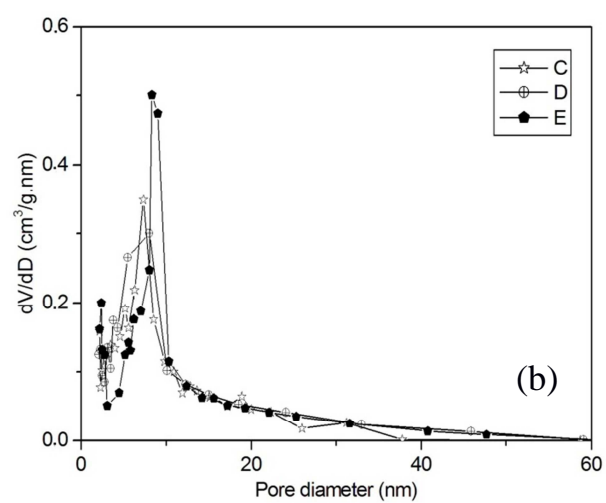
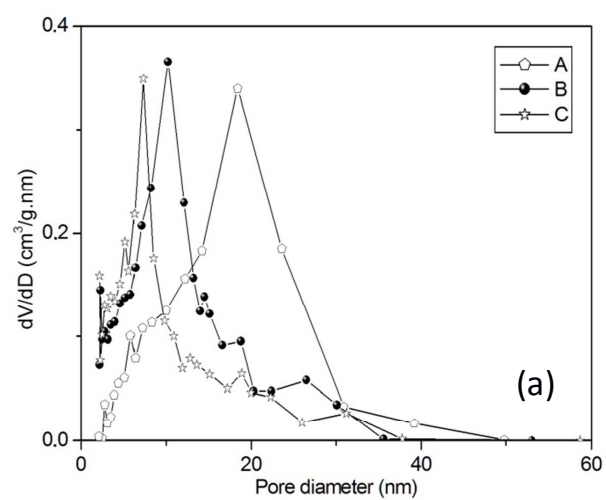
(b) for sample E



**Figure 5:** SAXS/WAXS curves. (a) Effect of the MTMS ratio (b) Effect of IL ratio.



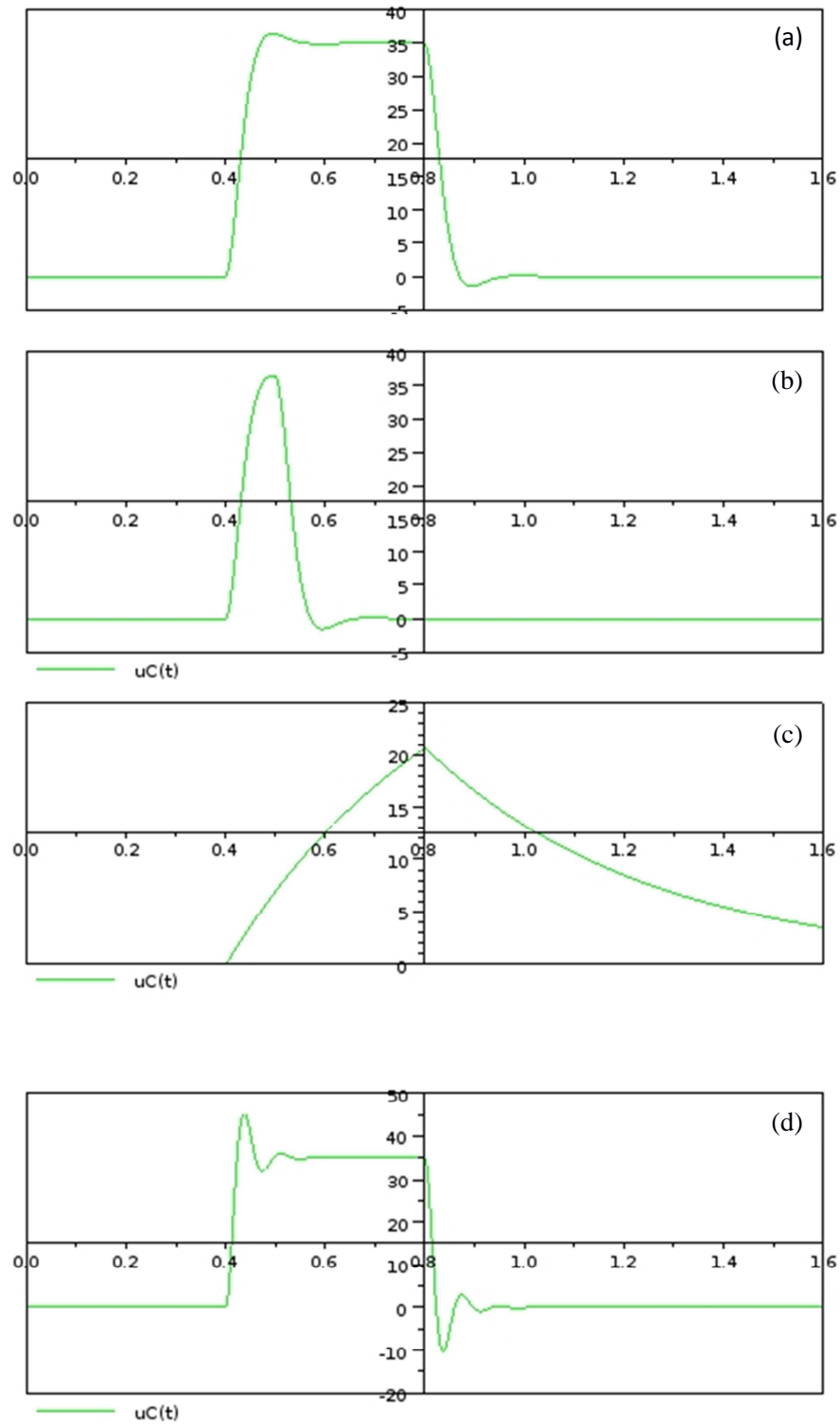
**Figure 6** : Adsorption/Desorption isotherms.



**Figure 7:** Pore size distributions obtained from BJH method.

(a) Effect of the MTMS/TMOS ratio

(b) Effect of the IL ratio.



**Figure 8:** (a)  $R=100\ \Omega$ ,  $L=1.5\ \text{H}$ ,  $C=0.0003\ \text{F}$ ,  $\Delta t=0.4\ \text{s}$  (b)  $R=100\ \Omega$ ,  $L=1.5\ \text{H}$ ,  $C=0.0003\ \text{F}$ ,  $\Delta t=0.1\ \text{s}$

(c)  $R=1500\ \Omega$ ,  $L=1.5\ \text{H}$ ,  $C=0.0003\ \text{F}$ ,  $\Delta t=0.4\ \text{s}$  (d)  $R=100\ \Omega$ ,  $L=1.5\ \text{H}$ ,  $C=8 \cdot 10^{-5}\ \text{F}$ ,  $\Delta t=0.4\ \text{s}$