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Article

Bonding theories between polyaniline and ionic liquids: Aspects and impacts

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Abstract: The characteristic properties of Polyaniline (PANI) motivate the scientific community to develop PANI materials with enhanced properties and performances for many applications (e.g., supercapacitors, sensors, anticorrosion coatings…). In their attempts to improve the properties and performances of PANI, they have replaced the conventional polymerization medium or the conventional electrolytes with ionic liquids, which are considered "green solvents". It was reported in the literature that ionic liquids generally positively impact the morphology and properties of PANI as compared to the conventional PANI. It has been agreed that nanostructured homogeneous morphology of PANI is obtained in ionic liquids, while agglomerated morphology is characteristic of PANI prepared in the conventional medium. Furthermore, the electrochemical performances of PANI synthesized in ionic liquids generally better than that of the conventional PANI. The ameliorations that occurred on the properties of PANI-ionic liquids have been attributed to bonding theories, detailed and discussed in this manuscript, between the imidazolium-based ionic liquids and PANI. Furthermore, the beneficial effects of non-covalent interactions between PANI chains and the ions of the ionic liquids, as well as between the ions of ionic liquids themselves, have been introduced. Finally, by understanding the bonding theories, researchers can tailor the properties of PANI by selecting convenient ionic liquids.

Keywords: polyaniline; ionic liquids; imidazolium-based ionic liquids; bonding theories; non-covalent interactions

1. Introduction

Although Polyaniline (PANI) is one of the oldest known synthetic polymers [1,2], it is still of interest owing to its properties (e.g., electrical conductivity, mechanical properties…) [3–7]. The characteristic properties of PANI motivate the scientific community to continue studying and developing PANI materials [8]. On the other hand, ionic liquids have attracted great attention thanks to their properties (e.g., low vapor pressure, thermal and chemical stability, inflammability…) and are labeled as "green solvent" [9–15]. Recently, the scientific community has been directed towards the replacement of the conventional polymerization medium or the conventional electrolytes of PANI with the ionic liquids, with a view to improving physicochemical properties, electrochemical performances and other properties of PANI, as discussed and detailed in our previous literature reviews based on scientific research published articles [16–27]. It has been reported that the synthesis or electrochemical characterization of PANI in ionic liquids (i) leads to the formation of nanostructured PANI morphology, (ii) promotes the enhancement of the solubility properties of PANI and (iii) enhances the electrochemical behaviors in terms of cycle life stability of PANI investigated as electrode materials for supercapacitor applications [18,25–27].

Although PANI-ILs are considered a promising combination, no study covers the reasons behind the formation of PANI-ILs or ionic liquid-free PANI, to the best of our knowledge. Indeed, some studies have reported the incorporation of ionic liquids into PANI backbone (i.e., PANI-ILs) [28–30], while others have shown that the ionic liquids are removed from the backbone of PANI during its purification (i.e., ionic liquid-free PANI) [31]. Infrared, UV-visible, NMR or Raman spectroscopies have been used to examine the impact of ionic liquids on PANI structure, and thus estimation of the incorporation of ionic liquids into PANI backbone [17]. For example, the infra-red spectrum of the conventional PANI has its characteristics bands located at 1559 cm^{-1} (quinone ring N = Q = N stretching), 1470 cm^{-1} (benzene rings N-B-N stretching), 1287 cm^{-1} (C-N stretching), 1079 cm^{-1} (C–H in-plane deformation) and 785 cm^{-1} (out-of-plane bending vibration of C-H of benzene rings) [32]. In the case of formation of PANI-ILs, the resulting infra-red spectrum presents infra-red bands of ionic liquids in addition to those of the conventional PANI [28]. However, for ionic liquid-free PANI neither additional peaks nor shift of the characteristic peaks of PANI has been detected on the infra-red spectrum of PANI-ionic liquids [18,20,31,33].

Incorporating ionic liquids into PANI backbone means that ionic liquids act as dopants, which are defined as charge carriers in the form of extra electrons and make PANI material conductive [34]. As a result, the properties of PANI-ionic liquids complexes (often called PANI-ILs) differ from those of ionic liquid-free PANI (i.e., ionic liquids assist PANI synthesis without doping it). Some theories have attributed the formation of PANI-ILs to the non-covalent interactions between PANI and ionic liquids [20, 21, 35]. However, each of these theories considered different types of non-covalent interactions between PANI and the ionic liquids. Some researchers have focused on π-π interactions [29], while others have concentrated on hydrogen bonding between PANI and the cations of the ionic liquids. Still other researchers have introduced bonding interactions between PANI and the anions of the ionic liquids instead of its cations, while others have considered ionic liquids as a soft template agent during the synthesis of PANI [36].

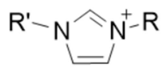
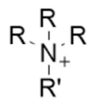
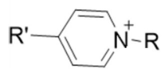
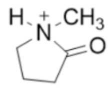
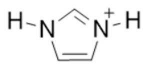
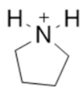
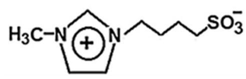
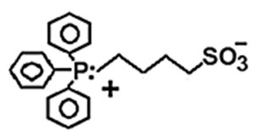
In this study, different bonding theories between PANI and the ionic liquids are gathered in order to understand the relationship between the structure of the ionic liquids and their effect on the properties of PANI.

2. Chemical structures of ionic liquids investigated for PANI

Ionic liquids are mainly classified into four groups based on their ion structures: protic, aprotic, zwitterionic, and functionalized (task-specific) [32, 37]. Protic ionic liquids are characterized by an available proton, while aprotic ionic liquids lack a mobile proton. Protic and aprotic ionic liquids, also known as conventional ionic liquids, are composed of separate cations and anions. However, zwitterionic liquids have both a positive and a negative charge within the same molecule, making them a dipolar ion.

Protic ionic liquids, aprotic ionic liquids and zwitterionic liquids have been used as an alternative to conventional acidic media (e.g., sulfuric acid H_2SO_4, hydrochloric acid HCl…) in order to improve PANI properties. **Table 1** summarizes the structure of the different investigated types of ionic liquids: imidazolium [18,20, 21,25,27,28,31,35,36,38–49], 1-alkyl-2-pyrrolidonium [50], pyrrolidinium [32,51–53], 1,4-dialkypyridinium [20], tetraalkylammonium [51], 3-(1-methyl-imidazolium) butylsulfonate [48] and so on. It is apparent from **Table 1** that imidazolium-based ionic liquids are the most investigated, as evidenced by the number of literature reports that have reported their use for PANI material. This given attention of imidazolium cation is attributed to its characteristic features and its ability to bond with PANI [18,20,21,27].

Table 1. Molecular structures of the different ionic liquids investigated for PANI in literature.

Class of ionic liquids	Name of representative cations	Molecular structure of cations
Aprotic ionic liquids	Disubstituted imidazolium [18,20,21,25,28,35,36,38–47]	
	Tetraalkylammonium [51]	
	1.4-dialkylpyridinium [20]	
Protic ionic liquids	1-alkyl-2-pyrrolidonium [50]	
	non-substituted imidazolium [27,31,49]	
	Non-substituted pyrrolidinium [32,52–54]	
Zwitterionic ionic liquids	3-(1-methyl-imidazolium) butylsulfonate [48]	
	triphenyl-phospho nium-3-butyl sulfonate [48]	

It is admitted that the class of the ionic liquids as well as their structure play a crucial role in determining how ionic liquids influence the morphology, properties and performances of PANI [20,51]. Indeed, it was reported that the ionic liquids, depending on their structure, affect the nucleation mode (the initial step in polymerization) and growth of PANI [17, 55]. However, the mechanism of its interference during the formation of PANI is still not clear, to the best of our knowledge. Diverse suggested mechanisms of formation of PANI in ionic liquids have assumed that ionic liquids interact with PANI chains through non-covalent interactions [20,21,35,56]. However, the controversy lies in the types and the manner of bonding. Different bonding theories between PANI and imidazolium ionic liquids (i.e., the most investigated ionic liquid for PANI) have been reported in literature and developed in the following sections.

3. Bonding theories between PANI and ionic liquids

The reported processes of formation of PANI in ionic liquids are similar to those in conventional acidic media, except for the interference of ionic liquids in mechanism of formation of PANI through non-covalent bonding interactions [20, 21, 29, 35, 36]. Different types of non-covalent bonding interactions, including π-π stacking, hydrogen bonding through anions or cations of ionic liquids, between PANI and ionic liquids have been suggested in the literature [20,35,56,57].

In conventional acidic media, PANI is typically synthesized via chemical oxidative polymerization of aniline monomers as illustrated in **Figure 1a**. Briefly, aniline monomers are oxidized (the ammonium persulfate is a common oxidizing agent), forming aniline cation radicals or nitrenium ionic. These active centers propagate to form PANI chains. The resulting PANI chains are then doped with an acid. Concerning the PANI prepared in ionic liquid, three main bonding theories between PANI and imidazolium ionic liquids have been suggested. Here is a detailed breakdown:

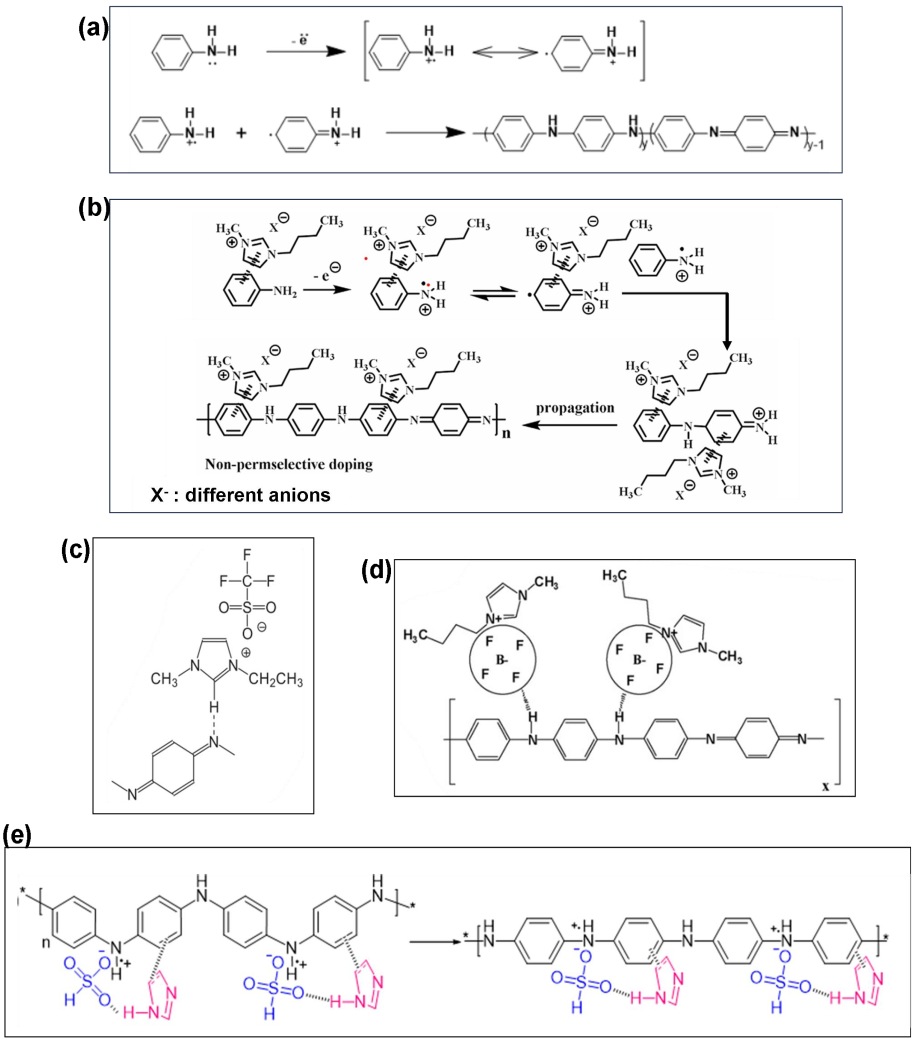


Figure 1. **(a)** mechanism of polymerization of aniline in conventional acidic media [34], **(b)** mechanism of polymerization of aniline in disubstituted imidazolium-based ionic liquid where benzene rings of aniline and imidazolium interact via π-π stacking interactions [29], **(c)** hydrogen bonding interactions between quinonoid moiety of PANI and the cation of imidazolium ionic liquids [21], **(d)** hydrogen bonding between the anionic part of ionic liquids and PANI chains [35], and **(e)** schematic illustration of the interactions between the cation of the ionic liquid and simultaneously with PANI chains and its dopant agent (my illustration).

3.1. *π-π* interactions

π-π stacking interactions can occur between the aromatic rings of the PANI chains and the imidazolium cation [20]. These interactions either (i) make from ionic liquids a soft template agent or (ii) lead to the formation of PANI-ILs complexes [20].

Regarding the role of imidazolium ionic liquids as a soft template agent, David Pahovnik et al. [20] have suggested that acidic aqueous solutions of ionic liquids promote the formation of nanostructured homogeneous PANI materials thanks to the π-π interactions between PANI and imidazolium. In other words, soft templates ionic liquids mean that they create ordered structures in acidic aqueous solutions and promote the formation of nanostructured PANI through their ability to interact with PANI through π-π interactions. These interactions do not alter the structure of PANI chains but only their assembly during polymerization. Furthermore, imidazolium ionic liquids are totally removed from PANI backbone as confirmed by UV-Vis, IR and Raman spectra [20]. A detailed description of how it is possible to confirm by Infrared spectroscopy that ionic liquids are removed from PANI backbone is presented in the introduction.

Concerning the formation of PANI-ILs complexes owing to the π-π stacking interactions between PANI and ILs, Ke Qu et al. [29] have reported that imidazolium ionic liquids dope PANI during the aniline electropolymerization processes. Their suggested mechanism of the incorporation of imidazolium ionic liquid into PANI backbone is shown in **Figure 1b**. They suggested that disubstituted imidazolium ionic liquids bond with the aniline monomer via some favorable π-π interactions between imidazolium ring and benzene ring. The imidazolium ionic liquids have been proposed to be attached to the benzene ring during all the elementary steps of aniline electropolymerization. The resulting PANI is doped with imidazolium ionic liquids as proved with NMR and UV-vis spectra [29]. It is important to note that the electropolymerization of aniline has been carried out in neat imidazolium ionic liquid (no extra organic or inorganic acids have been added) since imidazolium is considered acidic in the 2-position [29].

3.2. Hydrogen bonding

A hydrogen bond (H bond) is the attractive non-covalent interaction between a hydrogen atom covalently bonded to an electronegative atom and another electronegative atom (i.e., nitrogen, oxygen, fluorine) [58]. Two possibilities of hydrogen bonding between PANI and imidazolium ionic liquids, as follows:

i. **Interactions between PANI and cationic half of imidazolium ionic liquids**

Interactions of the quinonoid moiety of the PANI (i.e., imine nitrogen) with the imidazolium ring of the ionic liquids (i.e., cationic half of the ionic liquids) [21,56], as shown in **Figure 1c**. In this approach, disubstituted imidazolium cation acts as a Lewis acid thanks to the hydrogen at position 2 of imidazolium cation which is known to be relatively acidic [21,59]. In other words, the lone pair of electrons in imine nitrogen links to the acidic proton in imidazolium cation.

ii. **Interactions between PANI and anionic part of imidazolium ionic liquids**

In this theory, it was suggested that the hydrogen atom of amine group in PANI can bond with an electronegative atom in the ionic liquid anions: oxygen atoms in trifluromethanesulfonate [21], fluorine atom in teterafluoroborate [35] (see **Figure 1d**).

iii. **Simulation techniques**

Some research groups have only considered the interactions between imidazolium cation and PANI chains, ignoring the anionic half of the ionic liquids [27]. Others have only taken into consideration the interactions between PANI and anionic half of the ionic liquids [35]. Nowadays, the use of simulation techniques may be a useful technique to examine which part (i.e., anion or cations) of ionic liquids can interact with PANI chains. Gandhi et al. [44,45] have used all-atom molecular dynamics simulations and reported that both anions and cations of 1,3-dialkylimidazolium ionic liquids interact with PANI chains, whereas anions of ionic liquids have a dominant interaction with the polymer chains when compared to cations.

3.3. Combined π-π interactions and hydrogen bonding

In the previous section, it was reported that a disubstituted imidazolium cation (i.e., aprotic) can interact with imine nitrogen of PANI via hydrogen bonding through the hydrogen atom in the 2-position. As for nonsubstituted imidazolium ionic liquids (i.e., protic ionic liquids), they act like the typical Brønsted acid according to the literature [31,60]. In other words, disubstituted imidazolium cation shares its hydrogen atom in the 2-position with PANI backbone, while nonsubstituted imidazolium cation gives its proton to PANI backbone. It was thus suggested that the resulting nonsubstituted imidazole during the synthesis of PANI interacts with benzene ring of PANI via π-π interactions and with the heteroatom of hydrogen sulfate (i.e., dopant agent, anion of the ionic liquids) via hydrogen bond, affecting the conformation and organization of PANI [31], as illustrated in **Figure 1e**. It is important to note that the synthesis of PANI was conducted in water/ionic liquids mixture [31]. This suggested bonding hypothesis is supported by the fact that non-substituted imidazolium is sterically uncrowded, as compared to disubstituted imidazolium. These interactions, according to the scientist's suggestion, lead to a more planar chain conformation of PANI, elongation of effective conjugation length (i.e., free path), and thus, improved carrier transport by reducing the interlayer spacing between PANI chains [27,31].

3.4. Electrostatic interactions

All the previously discussed bonding theories between PANI and ionic liquids have been completely disregarded by Li et al. [36]. Indeed, Li et al. [36] have assumed that the interference of ionic liquids in controlling the formation process of PANI is due to the opposite charge of positively charged PANI and the anions of the ionic liquids. The formation process of PANI involves the following steps [36]: first, aniline is dissolved in an ionic liquid with the free movement of anions and cations. Subsequently, PANI particles are formed through chemical polymerization. During the polymerization process, anions of the ionic liquids (i.e., negatively charged) are adsorbed on the surface of PANI (positively charged). This adsorption causes the cations of ionic liquids to move to the outer layer. The resulting PANI exhibit spherical morphology.

4. Beneficial effect of non-covalent interactions between PANI and ionic liquids

It is generally agreed that the ionic liquids have a beneficial effect on the properties of PANI (i.e., nanostructuring of PANI morphology, enhancement of PANI solubility, enhancement of electrochemical performances and so on) [16]. To explain the modification that occurred in the properties of PANI-ionic liquids as compared to the conventional PANI, bonding theories between the imidazolium-based ionic liquids and PANI have been developed and adopted, as previously discussed in section 3 of this manuscript. It is believed that the bonding between PANI and ionic liquids leads to mixing characteristics of ions and electrons in PANI and ionic liquids, and thus improving PANI properties [27]. It is also shown that the resulting properties of PANI-ILs depend on the structure of the ionic liquids [16].

Most studies on mixing properties between PANI and ionic liquids have been realized using aprotic liquids, especially disubstituted imidazolium-based aprotic ionic liquids as previously shown in section 2 of this manuscript. Despite the characteristic features of disubstituted imidazolium aprotic ionic liquids, they are sterically more crowded and require more complex synthesis than protic ionic liquids. Furthermore, the suggested bonding theories between PANI and disubstituted imidazolium are completely different as compared to that between PANI and non-substituted imidazolium cation (see Section 3.3). Hence, the properties of the PANI prepared in non-substituted imidazolium hydrogen sulfate [Imi][HSO_4] (discussed in a previous work by Al-Zohbi et al. [31]) and PANI prepared in pyrrolidinium hydrogen sulfate [Pyrr][HSO_4] (presented in another previous article [53]) are gathered and described in the following paragraphs.

Looking deeply at the chemical structures of the protic ionic liquids [Imi][HSO_4] and [Pyrr][HSO_4] (**Figure 2a**,**b**), one can notice that there is a strong similarity between the chemical structures of these two protic ionic liquids (PILs). However, imidazolium cation of [Imi][HSO_4] possesses an aromatic ring system, while pyrrolidinium cation in [Pyrr][HSO_4] is a saturated (non-aromatic ring). As a result, [Imi][HSO_4] can interact via π-π interactions with PANI, while these interactions are not possible between [Pyrr][HSO_4] and PANI. The following is a comparison between the properties of PANI/[Pyrr][HSO_4] and PANI/[Imi][HSO_4] prepared in [Pyrr][HSO_4] and [Imi][HSO_4], respectively.

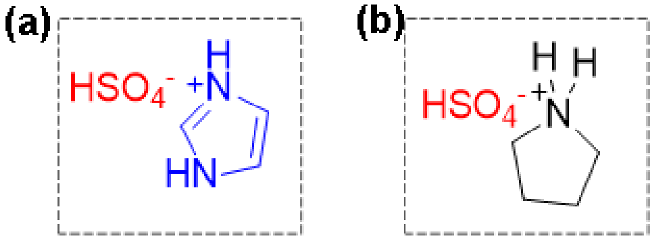


Figure 2. Chemical structures of **(a)** imidazolium hydrogen sulfate [Imi][HSO_4] and **(b)** pyrrolidinium hydrogen sulfate [Pyrr][HSO_4].

PANI/[Pyrr][HSO_4] and PANI/[Imi][HSO_4] were prepared by one-step oxidative polymerization process of anilinium hydrogen sulfate in aqueous solutions of [Pyrr][HSO_4] and [Imi][HSO_4], respectively. After the addition of ammonium persulfate (i.e., oxidant agent), the color of the polymerization mixture has taken almost half an hour to transform from colorless into green, indicating that the rate of polymerization is similar in the two polymerization media [31, 53]. Furthermore, both investigated PILs promote the fibrillar morphology of PANI as shown in **Figure 3a**,**b**. As for the effect of [Pyrr][HSO_4] and [Imi][HSO_4] on the structure of the PANI, the FTR-ATR spectra PANI/[Imi][HSO_4] and PANI/[Pyrr][HSO_4] and the conventional PANI has been studied [31]. It has been shown that non-substituted imidazolium ionic liquids affect the position of the infra-red characteristic peaks of PANI, while non-substituted pyrrolidinium ionic liquids do not have any effect on PANI structure [31].

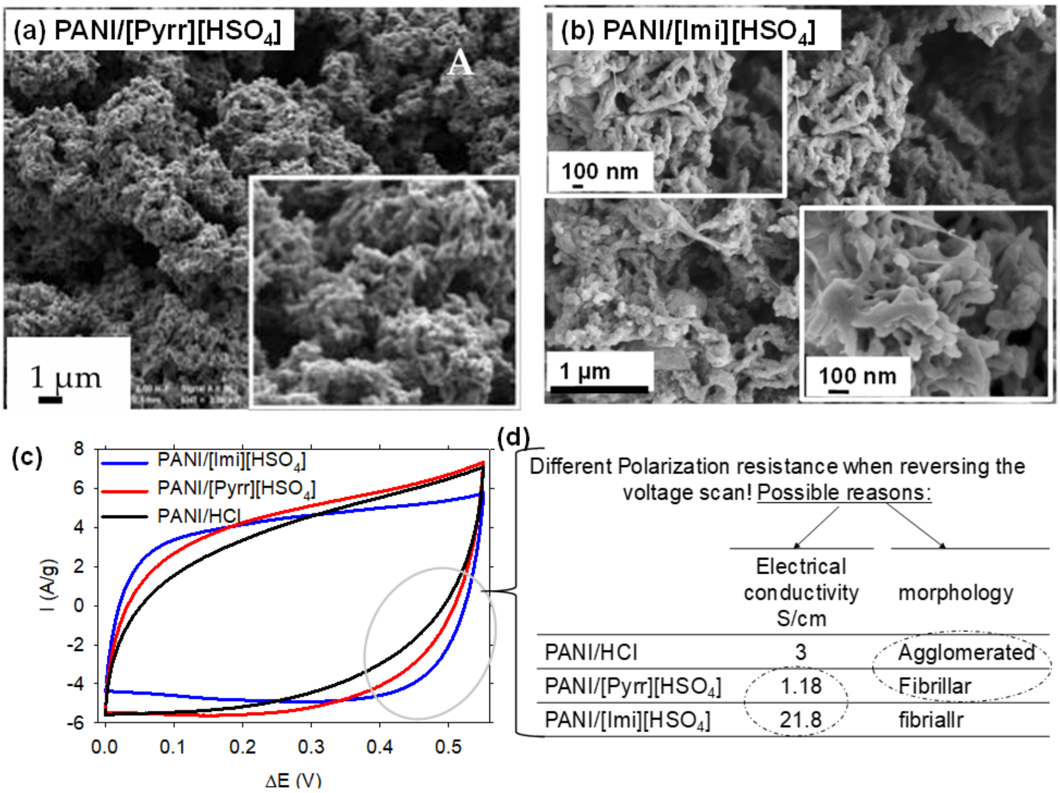


Figure 3. SEM images of **(a)** PANI/[Pyrr][HSO_4] and **(b)** PANI/[Imi][HSO_4], **(c)** CV curves at scan rate of 100 mV/s of PANI/[Imi][HSO_4], PANI/[Pyrr][HSO_4] and the conventional PANI (i.e, PANI/HCl) **(d)** illustration of the possible reasons behind the difference of the polarization resistance when reversing the voltage for PANI/HCl, PANI/[Imi][HSO_4], PANI/[Pyrr][HSO_4].
Source: **(a)** is adapted from the research by Al-Zohbi et al. [53], copyright 2021, MDPI, Basel, Switzerland; **(b)** and **(c)** are adapted from the study by Al-Zohbi et al. [31], copyright 2022, ISSN 2737-5323.

The electrochemical behaviors of PANI/[Pyrr][HSO_4] and PANI/[Imi][HSO_4], as electrode materials for supercapacitor applications, have been studied, using cyclic voltammetry CV technique in a potential range between 0 and 0.55 V, in a symmetrical two-electrode cell configuration and H_2SO_4 (1 mol/L) as electrolyte [31]. For comparison, PANI/HCl (i.e., the conventional PANI) has also been investigated as an electrode material [31]. The resulting CV curves recorded at a scan rate of 100 mV/s, reproduced from reference [31], are shown in **Figure 3c**. As one can see, the polarization resistance when reversing the voltage is not the same for the three investigated materials: it is more resistive for PANI/HCl due to its agglomerated morphology [31]. Comparing the CV shape of PANI/[Pyrr][HSO_4] and PANI/[Imi][HSO_4] (both exhibit fibrillar morphology), the polarization resistance when reversing the voltage of PANI/[Imi][HSO_4] is lesser relative to PANI/[Pyrr][HSO_4]

thanks to the higher electrical conductivity of the former (s: 21.8 S/cm) compared with that of the later (s: 1.18 S/cm) as summarized in **Figure 3d**. It is intriguing that the electrical conductivity value of PANI/[Imi][HSO_4] is higher by more than 18 times higher than that of PANI/[Pyrr][HSO_4]. To the best of our knowledge, this is the highest value of the electrical conductivity, measured with a four-point probe for a pellet of PANI, for PANI prepared with the assistance of the ionic liquids. This improvement in electrical conductivity has been ascribed to the bonding possibility between [Imi][HSO_4] and PANI during its synthesis (see Section 3.3) [31]. One can thus conclude that nonsubstituted imidazolium could be an excellent alternative to disubstituted imidazolium since the resulting PANI/[Imi][HSO_4] exhibits enhancement properties. Furthermore, PANI/[Imi][HSO_4] and PANI/[Pyrr][HSO_4] exhibit different properties (e.g., electrical conductivity, electrochemical performances) and different conformations that justify the importance given to the bonding theories between PANI and ionic liquids.

5. Conclusion

Many scientists have attempted to improve the properties of PANI by investigating ionic liquids as a replacement for traditional conventional polymerization medium or electrolytes. Substituted imidazolium cation-based ionic liquids with different substituents and different anions have been largely investigated. The main reason for the choice of this type of ionic liquid is the possibility of non-covalent interactions between PANI and disubstituted imidazolium cation itself. Different bonding theories have been suggested in the literature, as summarized in this manuscript. However, the role of the interactions between substituted imidazolium and the dopant agent of PANI has been neglected since substituted imidazolium is not willing to interact with PANI and its dopant agent at once.

In this manuscript, it has been suggested that the non-covalent interactions between the cation of the ionic liquids, on the one hand, and the PANI via π-π interactions, and on the other hand, with the dopant agent (via hydrogen bonding) lead to electrically conductive and electrochemically performant PANI materials. Indeed, it has been suggested that these combined interactions affect the conformation of PANI chains, leading to a more planar conformation. This study points out the importance of combined non-covalent interactions between the cation of the ionic liquids, PANI chains and their dopant agent.

Finally, this short overview will guide the future directions for research and development of PANI/ionic liquid. Among numerous ionic liquids, this review shifts the attention to the ones that can interact with PANI and its dopant agent and leads to control of the conformation of PANI.

Conflict of interest: The author declares no conflict of interest.

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