Ionic liquid based on PEDOT:PSS/PVA/metal slats composites for flexible Paper based applications

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Abstract

The paper-based electronics were prepared using PEDOT:PSS:PVA:Metal salts ionic liquid. The latter has been investigated by several characteristics such as pH, conductivity, molar conductivity, activation energy, and degree of dissociation. These results have verified that pH greatly influences the device structure as it directly affects the cellulose structure. The electrical conductivity is increased and the molar conductivity is decreased. The cellulose paper was immersed in the ionic liquids with concentrations 1, 2, 3, and 4mg/ml for each type of metal salt and the results revealed that CuCl2 has the best performance among other metal salts with higher conductivity and less resistivity among other ionic liquids.

Keywords: Ionic liquid; PEDOT:PSS; PVA; Metal salts; conductivity, paper application

1. Introduction.

Polymer composite is widely used in the field of flexible electronics; PEDOT:PSS and PVA are promising candidates as for such application due to their unique properties and ability to be easily processed into high conductive, transparent, and flexible thin films [1,2]. The addition of metal salts can further enhance the conductivity of the conductive polymer composite to become ionic liquid, resulting in suitable electrolyte for flexible paper applications. The choice of metal salts and their concentration can greatly impact the properties of the ionic liquid, allowing for fine-tuning of the material to meet specific requirements of different applications [3,4]. The electrical conductivity of ionic liquids is influenced by various factors, including the concentration of metal salts, the extent of hydration, the composition of the liquid, the liquid pH, the degree of electrolytes dissociation within the liquid, and the processing conditions used during the preparation of the liquid [5,6]. PVA is usually used as a binder to keep the metal salts and the conductive PEDOT:PSS particles evenly dispersed in the liquid. The development of flexible paper-based electronic devices has the potential to revolutionize the field of flexible electronics, providing new opportunities for the development of flexible and low-cost electronic devices for a wide range of applications. Ionic liquids are a type of liquid that contain ions suspended in a solvent. The specific ions present in an ionic liquid depend on the substances that are dissolved in the solvent, the strength of the ion-dipole forces of attraction between water and ionic compound determines whether the ionic compound will dissociate in liquid [7]. The solute-solute interactions are usually ignored as they are relatively weak compared to the ion-dipole forces of attraction [8]. In addition, the liquid pH can also affect the degree of dissociation of the ionic compounds [9,10]. The liquid pH is considered as acidic, if more hydrogen ions (H+) are present in the liquid, which can compete with the ionic compound for solvation by the solvent. This can affect the dissociation behavior of the ionic compound and its resulting electrical conductivity. On the other hand, if the liquid pH is basic, hydroxide ions (OH-) are present, which can also affect the dissociation behavior of the ionic compound. In general, by controlling the liquid pH, it is possible to optimize the electrical conductivity of the ionic liquid for specific applications [11,12]. Examining the magnitude of ion-dipole forces of attraction between water molecules and an ionic compound is essential for explaining the properties and dynamics of ionic liquids. Generally, the strength of these ion-dipole interactions between water and metal ions determines the degree of dissociation demonstrated by the metal ions within the liquid; stronger ion-dipole forces result in greater dissociation and increased conductivity [8]. The liquid conductivity is important in various applications, such as batteries, where a precise control over the ionic conductivity is needed to optimize the performance and functionality of the system. The combination of conductive polymer PEDOT:PSS and PVA can improve the mechanical stability and flexibility of the final device. The optimal combination of PEDOT:PSS and PVA may vary depending on the specific application and desired performance of the device, and further experimentation and optimization may be necessary. The combination of the cellulose paper substrate, PEDOT:PSS, PVA, and metal salts provides a cost-effective, lightweight, and flexible substrate for various electronic devices, making it an attractive option for future development and commercialization. In the current study, focusing on such combination with different meal salts ($CuCl_2$, $CdCl_2$, and $LiCl_2$) was carried out to give further information about such composite for future paper-based application.

2. Experimental

2.1.Preparation methods:

The preparation of the PEDOT:PSS:PVA:Metal salts ionic liquid and coating the cellulose paper with this liquid involves multiple steps as shown in Fig.1. This figure suggests the arrangement of the ionic liquid on the cellulose fiber structure, this suggestion is similar to the observation by other groups [13,14].

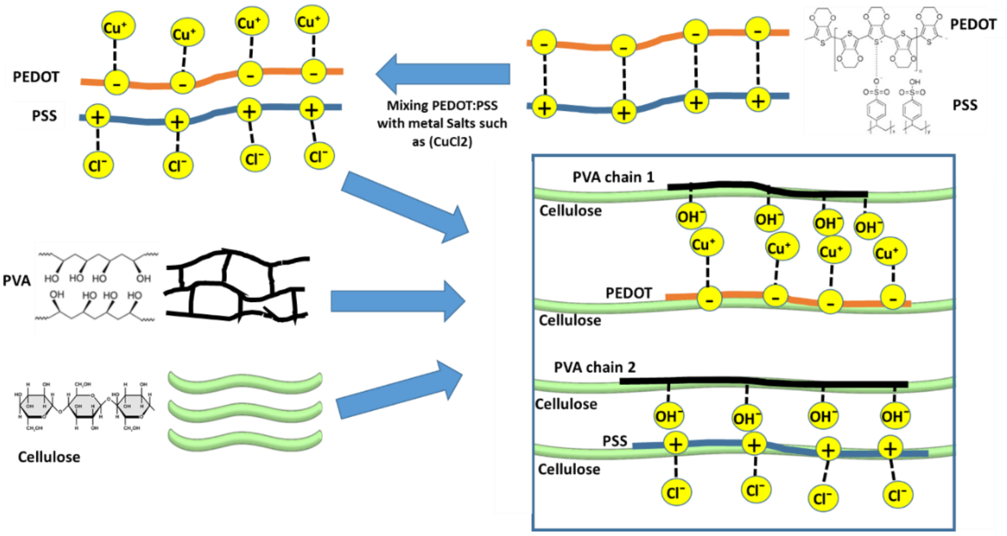

Fig.1: Illustration of the PEDOT:PSS, PVA, Metal salts and cellulose fiber composite

2.2.PVA preparation:

To prepare Poly vinyl alcohol (PVA), 10gm of the polymer (PVA) is dissolved in 50ml of double distilled water. Firstly, water is heated on a hot plate at 70^{o}C for few minutes, PVA is then added slowly to the water under stirring and the liquid kept under stirring at 70^{o}C until a clear viscous liquid is obtained.

2.3.PDOT:PSS preparation:

PEDOT:PSS dry re-dispersible pellets are purchased from Sigma Aldrich and used after soaked in the 10ml HCL acid. PEDOT:PSS liquid stirred under 40oC on a hot plate stirrer for 1 hour. The final liquid is kept in dark overnight.

2.4.Metal salts liquids:

Different metal salts were used including $CuCl_2$, $CdCl_2$, and $LiCl_2$. The metal salts dissolved in double distilled water, separately under stirring without heat, at different concentrations (from 1gm/ml to 10gm/ml and from 10gm/ml to 100gm/ml).

Ionic liquid preparation

The ionic liquid is prepared by mixing PEDOT:PSS, PVA and metal salts liquid under stirring. The final concentrations of the liquids are considered to be from 1gm/ml to 10gm/ml and from 10gm/ml to 100gm/ml.

2.5.Paper based samples:

A sheet of cellulose paper is used to prepare paper-based flexible devices. Dip coating method is applied to prepare the final flexible samples, once PEDOT:PSS/PVA/Metal salts liquid is prepared, a sheet of cellulose paper substrate is immersed in the ionic liquid for 30min to ensure good absorption. The choice of coating method affects the desired thickness and uniformity of the coating, as well as the size and shape of the substrate. The coated paper substrate is then dried to ensure complete evaporation of the solvent and to improve the mechanical stability of the coating. The addition of metal salts to the PEDOT:PSS/PVA liquid must be done with caution, as the presence of impurities in the metal salts can affect the properties of the final liquid. Proper purification techniques, such as recrystallization or distillation, may be necessary to ensure the quality of the metal salts. The paper-based sample is prepared by using two electrodes on both sides of the cellulose paper as shown in Fig.2.

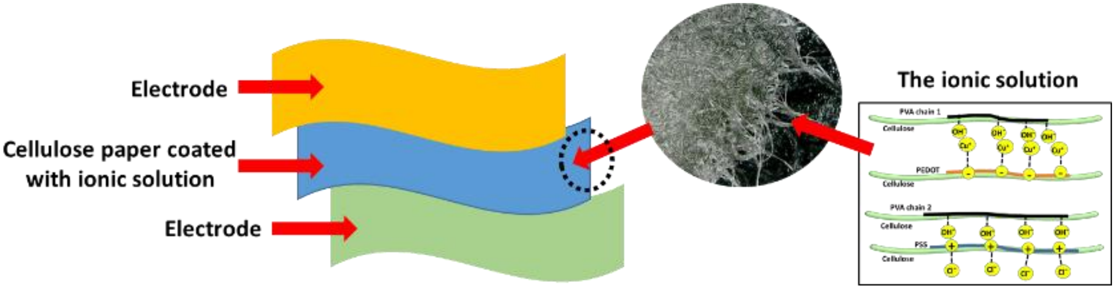

Fig.2: illustration of paper-based device

3. Results and Dissections

3.1.Surface structure of the samples:

The surface structure of the paper-based samples is investigated using optical microscope with 500x magnification as shown in Fig.3. It is noted that after adding only PVA liquid to the cellulose fiber (paper sheet), there is no change in the cellulose structure. Due to its inadequate mechanical properties, PVA has been investigated for the incorporation of reinforcing materials to enhance its performance [15].

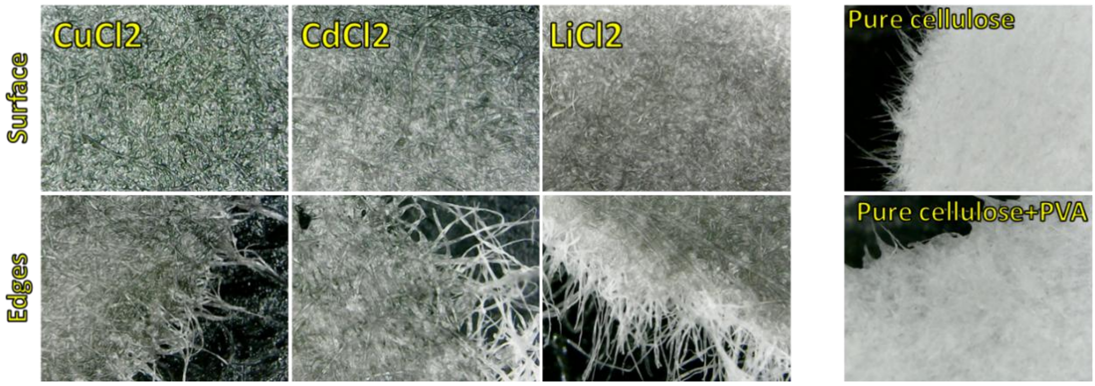

Fig.3: Optical images of the paper-based composites (PEDOT:PSS:PVA:metal salts liquid covered the cellulose sheet)

Several studies have been conducted to consider PVA as a promising matrix for sustainable composites. This is ascribed to its characteristics, including water solubility, biodegradability, bio and eco compatibility, high tensile strength, excellent adhesive properties, as well as chemical resistance and gas barrier properties [16-18]. Besides, literatures specify that cellulose fiber is a significant reinforcement with PVA, resulting in enhanced tensile modulus, mechanical strength, thermal stability, and thermo-mechanical properties in the produced composites [15, 19,20]. As PEDOT:PSS is prepared by HCl; adding the ionic liquid (PEDOT:PSS:PVA:metal salts) to the cellulose fiber could results in partial degradation of cellulose by acid hydrolysis [21]. The acid can effectively damage the cellulose fiber by etching of the crystalline contacts of the nanocrystallites surface [22]. The images in Fig.3 show that there is no clear damage in the cellulose fiber and the ionic liquid is regularly distributed within the cellulose structure, even at the edges of the cellulose fiber. This is attributed to the use of PVA which assist the cellulose fiber structure.

3.2.Liquid properties:

The potential of hydrogen (pH) of the PEDOT:PSS/PVA/Metal salts ionic liquid is measured based on different metal salt concentration and type. As mentioned in the preparation section, PEDOT:PSS is acidic, therefore, it is important to monitor and adjust pH to confirm best performance of the ionic liquid [23] especially when this acidic liquid used with cellulose fiber [22]. Usually, PVA act as an acid-base equilibrium [24]; sometimes PVA is considered as a weak base due to the presence of acidic hydroxyl groups in its chemical structure [25]. The controlled pH is very important in the current study as the acidic liquid can damage the cellulose fiber as mentioned earlier. Herein, PEDOT:PSS liquid is acidic as it is prepared using HCl acid, therefore, adding PVA could slightly decreasing the acidity. Moreover, adding 1mg/ml of metal salts liquid to the PEDOT:PSS:PVA liquid affects the acidity of the samples (see Fig.4A).

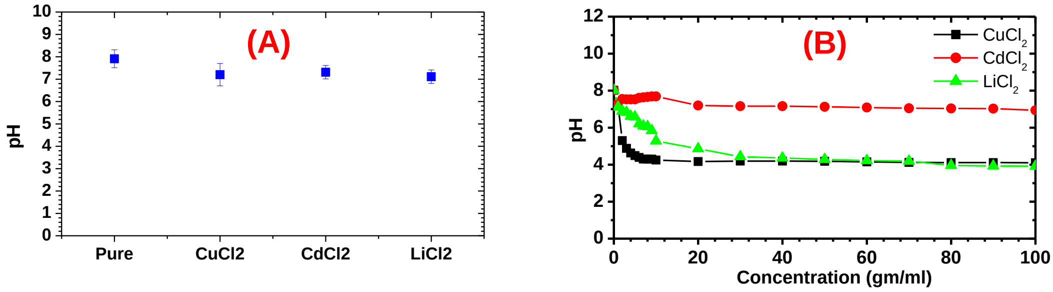

Fig.4; pH values of pure PEDOT:PSS:PVA (A) with and without different metal salts at concentration (1mg/ml), and (B) with different concentrations of metal salts

The pH values decreased from 8.2±0.1 in pure sample (PEDOT:PSS:PVA) to reach 7.8±0.2 by adding 1mg/ml of $CuCl_2$ liquid; 7.6±0.3 by adding 1mg/ml of $CdCl_2$ liquid; 7.4±0.3 by adding 1mg/ml of $LiCl_2$ liquid. Furthermore, adding different metal salts concentration to the PEDOT:PSS:PVA liquid is considered and the results are shown in Fig.4B; pH values are found to be different based on the metal salts and type. Increasing the $CuCl_2$ concentration to higher values has resulted in reducing the pH and the liquid becomes acidic again until its pH value reached 4±0.1 at concentration (4mg/ml). Increasing the $CuCl_2$ concentration demonstrate no change in the pH value due to the saturation of the liquid. Similar trend is observed after adding LiCl2 salts to the PEDOT:PSS:PVA liquid. However, the decrease in the pH value using this metal salt is gradual until it reaches a value of 5±0.3 after adding (10mg/ml). This value is further decreased to 4.9±0.2 after adding 20mg/ml of LiCl2. Further increase in the concentration results in no change in the pH value. Moreover, increasing the CdCl2 concentrations decrease the pH value slightly to a value of 7.1±0.2. Further increase in the CdCl2 concentrations has demonstrated pH values in the range 6.9-7. This steady state value of the pH at high concentrations may be attributed to the saturation of the liquid. The electrical conductivity (σ) of the ionic liquid is measured and the results are illustrated in Fig.4A. The electrical conductivity exhibited gradual increase with increasing the metal salts concentration, higher concentration results higher electrical conductivity for the metal salts under study. The electrostatic attraction between an ion and a molecule with a dipole is called an ion-dipole attraction. These attractions play an important role in the disliquid of ionic compounds in water. When the latter dissolves in a proper solvent, the ions in the solid form separate and disperse uniformly throughout the liquid because solvent molecules surround and solvate the ions, reducing the strong electrostatic forces between them resulting in increasing ionic conduction [26]. This process represents a physical change known as dissociation. Under most conditions, ionic compounds will dissociate nearly completely when dissolved, and so they are classified as strong electrolytes. The variances in the electrical conductivity may be due to the differences in charge and size of the cations which can affect the way these cations interact and influencing the liquid properties such as solubility and ionic conductivity [27]. To further understand the electrical conductivity of the ionic liquids, molar conductivity (Ɱ) is considered. The latter is calculated using the following equation [28]:

$$\text{Ɱ} = \frac{\sigma}{c} \qquad (1)$$

Where c is the molar concentration of the liquid. The molar conductivity decreased with increasing metal salts concentrations (see Fig.5B) due to the increase in the dielectric constant of the ionic liquid resulting in stronger hydrogen bonding interactions. Higher concentration results in less intermolecular interactions, and the electrostatics repulsing leads to a reduction in the intermolecular bonding and increase the dimensions between the molecules of the ionic liquid [29]. In conclusion, small reduction in the molar conductivity is observed with increasing concentration more than 10mg/ml for all samples.

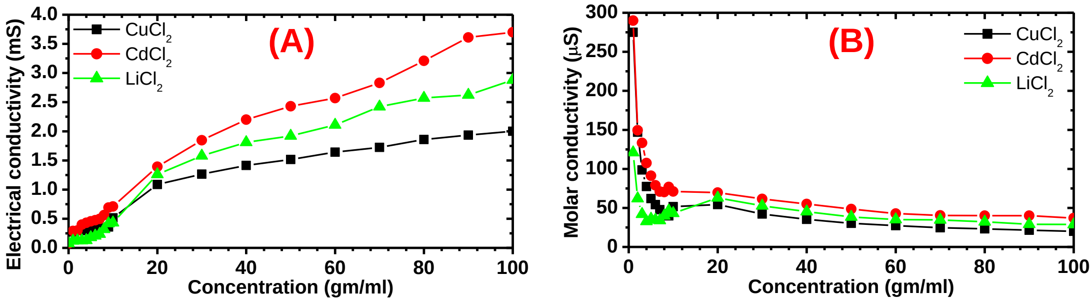

Fig.5; (A) electrical conductivity and (B) Molar conductivity, of all samples

To understand the dissociation process of these metal salts within the PEDOT:PSS:PVA liquid, activation energy is calculated. The ionic liquids treated with different temperatures in order to investigate the activation energy, results are illustrated in Fig.6.

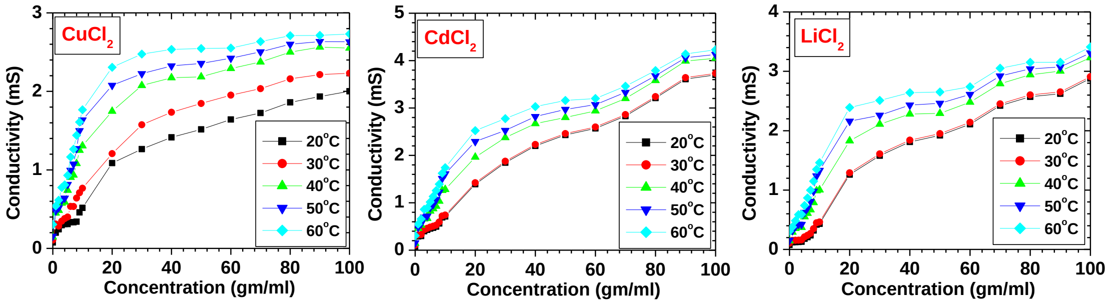

Fig.6; electrical conductivity of all ionic liquid under different heating temperature

The increase in the heating temperature results in increasing the electrical conductivity. The activation energy is calculated using Arrhenius equation [30]:

$$K = K_{\infty} Exp(-\frac{E}{RT}) \qquad (2)$$

where k∞ is pre-exponential factor, E is the activation energy, R is the universal gas constant and T is the absolute temperature. A linear dependence of ln(k) on 1/T with slope (−E/R) is obtained, which is represented graphically is called the Arrhenius plot. As the activation energy is the energy barrier that reactant molecules must overcome and dissociate in the liquid, results of the activation energy with different metal salts concentration is illustrated in Fig.7A. The degree of dissociation is considered as it gives deep information about the dissociation of the molecules inside the liquids, results based on adding metal salts with different concentration to PEDOT:PSS:PVA are demonstrated in Fig.7B and confirm the dissociation degree with different metal salts concentrations. Generally, Ostwald's dilution law is considered in this relation, which is frequently used to approximate the dissociation degree and completely ignores the solvation effect and the concept of free solvent molecules [31]. Ostwald of dilution indicates that complete dissociation when the dilution approach infinite therefor the dilute liquid may be represented as strong electrolyte, the value of degree of dissociation has the range $0 \leq D \leq 1$; for strong electrolyte D=1 and for weak electrolyte D=0. Results revealed that low concentrations have higher D compare to higher concentration. The relation between activation energy and the degree of dissociation of a liquid is not straightforward and depends on the specific chemical reaction and the nature of the solute. However, higher activation energy results in less dissociation as demonstrated in Fig.7 (A and B). Generally, reactions with higher activation energies tend to have lower degrees of dissociation. This is because a higher activation energy implies a greater energy barrier that reactant molecules must overcome to undergo the reaction. If the reaction requires more energy, fewer molecules will possess sufficient energy to reach the activated state and dissociate. Moreover, the degree of dissociation is often influenced by temperature. As temperature increases, more molecules gain the necessary energy to overcome the activation energy barrier, leading to an increase in the degree of dissociation.

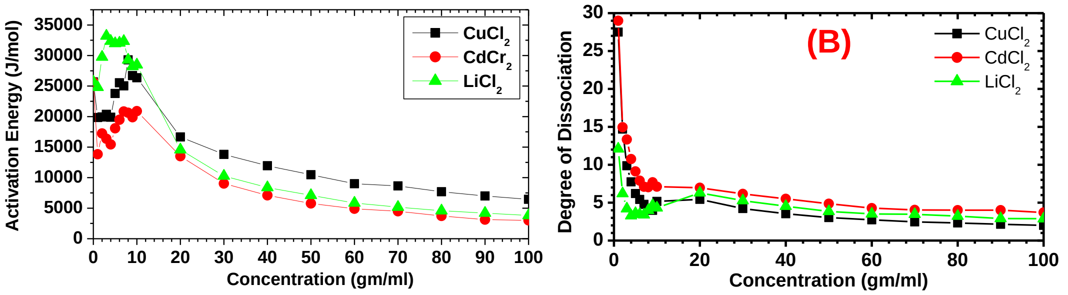

Fig.7: (A) Activation energy and (B) Degree of dissociation, of the ionic liquid as a function of different metal salts concentrations

3.3.Paper based device:

The electrical conductivity and sheet resistance were measured using 2400 Keithley source-meter and results are presented in Fig.8 (A and B, respectively). The bulk conductivity was carried out using a paper sheet of cm (length) × 5cm (width) dimensions with the structure shown in Fig.2; the thickness is considered to calculate the electrical conductivity and resistivity and was measured using a digital micrometer. The metal salts at concentrations (1mg/ml, 2mg/ml, 3mg/ml and 4mg/ml) are chosen to prepare the paper-based samples due to pH value which prevent the cellulose from degradation, as well as the high degree of dissociation and high molar conductivity of their ionic liquid. The electrical conductivity and resistivity (ρ) are measured using the following equation [32]:

$$\sigma = \frac{L}{RA} = \frac{1}{\rho} \qquad (3)$$

Where σ is the conductivity, L is the sample thickness, A is the active area of the sample and R is the resistance which was calculated from the slope of the IV curve presented in Fig.8.

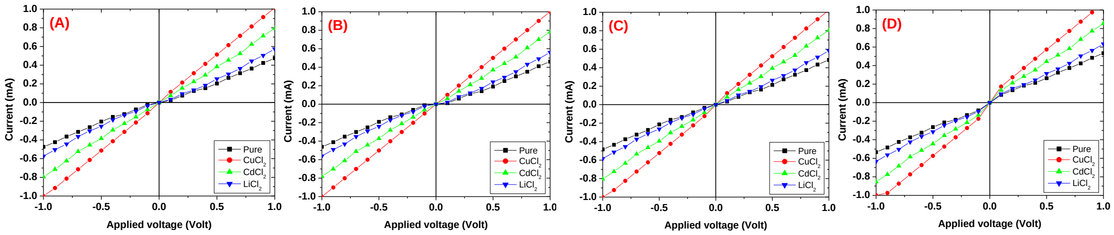

Fig.8; IV curves of the samples based on metal salts concentration (A) 1mg/ml, (B) 2mg/ml, (C) 3mg/ml, and (D) 4mg/ml

It is clear that the resistance is varied in samples with metal salts compare to the pure sample (without metal salt); sample with $CuCl_2$ has demonstrated high current and low resistance compare to other samples. This has resulted in low resistivity as shown in Fig.9; the resistivity of $CuCl_2$ sample is lower than CdCl2 and LiCl2 samples. In comparison to the pure sample, the resistivity of $CuCl_2$ sample is decreased more than 50% from the resistivity of the pure sample. Such enhancement is favorable for electronic application. The use of PVA which is generally an insulating polymer may contribute to increasing the overall resistivity. But, using metal salts as conductive additive has resulted in reducing the resistivity, depending on concentration. On the other hand, cellulose paper is likely an insulator, and the composite's overall resistivity will depend on the interaction of the ionic liquid components with the cellulose paper.

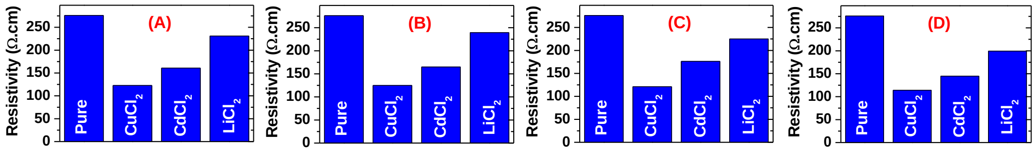

Fig.9; Resistivity of the samples based on metal salts concentration (A) 1mg/ml, (B) 2mg/ml, (C) 3mg/ml, and (D) 4mg/ml

The electrical conductivity of the sample is calculated based on the IV curve using eq.3. results revealed an increase in the electrical conductivity of cellulose-based device when using $CuCl_2$ which exhibited the higher value compare to other metal salts. Changing the metal salts concentration exhibited similar trend for all samples, however, $CuCl_2$ still obtain the higher conductivity among other metal salts.

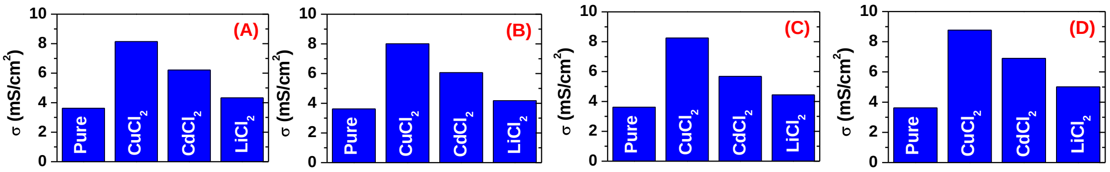

Fig.10; Conductivity of the samples based on metal salts concentration (A) 1mg/ml, (B) 2mg/ml, (C) 3mg/ml, and (D) 4mg/ml

4. Conclusion

Composites based on the ionic liquid PEDOT:PSS:PVA:Metal salts were prepared and investigated for paper-based application. Results revealed that the electrical conductivity is improved by increasing metal salt concentration. The pH has great influence on the device structure as it directly affects the cellulose structure. Cellulose paper was immersed in the ionic liquids with concentration 1, 2, 3, and 4mg/ml and the results revealed that $CuCl_2$ has the best performance among other metal salts.

Conflicts of interest

The authors declare that there are no conflicts of interest

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سائل أيوني مُركّب من PEDOT:PSS/PVA/أملاح معدنية لتطبيقات ورقية مرنة

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الخلاصة:

تم تحضير الإلكترونيات الورقية باستخدام سائل أيوني من PEDOT:PSS:PVA/أملاح معدنية. تمت دراسة هذه الخاصية من خلال عدة خصائص مثل الرقم الهيدروجيني، والتوصيلية، والتوصيلية المولية، وطاقة التنشيط، ودرجة التفكك. وقد أكدت هذه النتائج أن الرقم الهيدروجيني يؤثر بشكل كبير على بنية الجهاز، حيث يؤثر بشكل مباشر على بنية السليلوز. تزداد التوصيلية الكهربائية بينما تنخفض التوصيلية المولية. تم غمر ورق السليلوز في سوائل أيونية بتراكيز 1، 2، 3، و4 ملغم/مل لكل نوع من أملاح المعادن، وكشفت النتائج أن كلوريد النحاس الثنائي (CuCl2) يتمتع بأفضل أداء بين أملاح المعادن الأخرى، حيث يتميز بأعلى توصيلية وأقل مقاومة بين السوائل الأيونية الأخرى.

الكلمات المفتاحية: سائل أيوني؛ PEDOT:PSS؛ PVA ؛ أملاح المعادن؛ التوصيلية؛ تطبيقات الورق.