Optical, Structural and Electrical Properties of Electrochemical Synthesis of Thin Film of Polyaniline

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Abstract:
Polyaniline membranes of aniline were produced using an electrochemical method in a cell consisting of two poles. The effect of the vaccination was observed on the color of membranes of polyaniline, where analysis as of blue to olive green paints. The sanction of PANI was done by FT-IR and Raman techniques. The crystallinity of the models was studied by X-ray diffraction technique. The different electronic transitions of the PANI were determined by UV-VIS spectroscopy. The electrical conductivity of the manufactured samples was measured by using the four-probe technique at room temperature. Morphological studies have been determined by Atomic force microscopy (AFM). The structural studies have been measured by (SEM).

Keywords: Polyaniline, electrochemical methods, FT-IR, XRD studies, conducting Polymers.

Introduction:
The electrochemical manufacturing process is unique, and it is an exceptional way to manufacture polymers capable of conduction with the possibility of measuring the thickness and area of the product. Polyaniline (PANI) lodges the main place between the conducting classes of polymers. Polyaniline is odd phenyl built polymer own chemically elastic -NH-series in the polymer bounded via phenyl loop on both side (1). It symbolizes a group of macromolecules which conductivity of electrical enables changes from an insulator for a conductor through the redox procedure. The electron conductivity of polyaniline in the formula of emeraldine depends on its protonation and growths by ten orders of magnitude as the degree of protonation is increased from 0 to 20% (2). Indeed, the summary of heteroatoms makes it possible to compensate for the lack of stability of the Polyacetylene in the presence of atmospheric conditions. Their physical and chemical properties make them particularly attractive, they have great electrical conductivity (3). The stability of the environment is good in oxygen (4). In attendance is too scope of work in the field of solar power (5, 6). Organic photovoltaic cells (7), gas sensors (8, 9,10), a solid electrolytic capacitor (11), electrochromic windows and displays, and packaging, polymeric batteries (12, 13). in addition to electric chromic projectors (14). History from conjugated polymers began in the early 1970s when the sulfur polynitride [(-S=N-)x] at room temperature has conductivity close to that of metals. This remarkable property attracts the interest of many the searchers, and it has rapidly studied other compounds that may be similar properties.

Structure of Polyaniline
The protonation and deprotonating and various other physics -chemical properties of PANI can be said to be due to the presence of the -NH- group. PANI has included the most usually calculated as a characteristic fellow for the polymer conductive private for the following causes, easy production. It is the individual conductive polymer that has an electronic and electrical structure. Properties can reversibly be controlled together via oxidation and protons. It takes exciting electrochemical performance, it appearances environment stability, easiness of non-redox doping by protonic acids (15).

Symmetry and Group Theory
In order to determine the vibrational modes that can be observed experimentally, the Group theory lets calculating the number of modes of vibration of molecules, as well as their symmetry. By applying, operations of symmetry can obtain the representation reducible of the molecule. Its
decomposition into irreducible representations provides the number and the symmetry of the normal modes of vibration, once the modes corresponding to rotations and translations. The public formula with regard to aniline and polyaniline is shown in Fig.1 (a, b).

\[ \Gamma_{\text{vibration}} = 13A_1 + 12A_2 + 4B_1 + 7B_2 \ldots (1) \]

Figure 1. a- public formulas concerning together aniline and the conductive b- polyaniline (emeraldine shape and there calculated structure).

The formula for aniline contains 14 atoms as shown in Fig. (a), application about group theory with monomer show C\(_2\)v similarity so will have 42 degrees of freedom, 6 of them are external vibrations, then will have 36 styles of hesitations, which are active together I.R and Raman as shown in Eq.1

\[ \Gamma_{\text{vibration}} = 13A_g + 4B_{1g} + 12B_{2g} + 7B_{3g} + 5A_u + 12B_{1u} + 12B_{2u} + 7B_{3u} \ldots (2) \]

**Ring Vibrations:**

By taking the 4,4'-bianiline as shown in Fig. b without their radical we can suggest C\(_{2h}\) point group, and exist have 36 irreducible representations and exist have 30 ring modes of vibrations as shown in Eq. 3

\[ \Gamma_{\text{vibration}} = 11A_g + 5A_u + 4B_{1u} + 10B_{1u} \ldots (3) \]

**Experimental:**

A quantity of 0.05, 0.1 and 0.2 M aniline was added to 0.1M from electrolytic middle contained acetonitrile and Lithium tetrafluoroborate; they were located in a cell. Interaction was carried out inside the cell at a temperature of 0 ° C. Thin films for polymer designed on the gold pole; however, other rod materials like ITO are as well apposite for that intent while the effort amount > 4V the flow is gradually rising, The gold outward was thereafter shielded by means of a blue, strongly glued deposit; it’s great accessibility that permitted a quick evolution on the membrane of numerous \(\mu\)m thickness. The color of the product was detected through the interaction; the blue was initially unclear and converted to blackish green while it was glutinous at the end of the interaction.

**Result and Discussion:**

**Infrared Spectroscopy (FT-IR)**

The structure of the produced PANI was described by Fourier Transform Infrared Spectroscopy (FTIR) and was able to obtain the spectra in the range of (4000 – 400) cm\(^{-1}\). FT-IR bands of PANI model prepared by 0.1M of aniline are shown in Fig. 2. In a spectrum, the bands detected at 3456, 3421 and 3263 cm\(^{-1}\) is consequent at N-H stretching. The observed absorption bands of the polymer at 3066.82, 3028, 2974, 2931, 2862, 2835 and 2021 cm\(^{-1}\) are due to irregular C-H stretching of the aromatic ring (17). The absorption points seen at 1635, 1620, 1554 and 1504 cm\(^{-1}\) were credited to C=C stretching in aromatic nuclei (1). The polymer appearances captivation bands at 1381, 1288 and 1230 cm\(^{-1}\) which form the C-N stretching of primary aromatic amines (18). The absorption bands centered at 1176, 1126 and 1091 cm\(^{-1}\) reveal the C-H bending vibrations (19, 1).
processes was executed with the Hyperchem 8 bundle program. The computations values for wavenumber and intensity of the bands in Raman and I.R spectra were applied to explain the values of the experimentally gained of Raman and I.R ensembles. Some of these frequencies are given in Tables 1 and 2.

Table.1 Comparison of some experimental and theoretical harmonic frequencies in (cm$^{-1}$) from shapes in the infrared spectra of the nanopowder and thin membranes of polyaniline and the theoretical compatibility frequencies for bianiline and aniline in (cm$^{-1}$).

| Bianiline Ab-initio small (3-21G) only IR active (MNDO) | PANI expt$^a$ Thin film Monomer concentration 0.1M Prepared with saline solution | Thin film Monomer concentration 0.2M Prepared with saline solution | Nano powder Prepared with acidic solution | Assignment |
|---|---|---|---|---|
| 3731 | 3516 | 3428$^b$ | 3456 | 3478 | 3464 | $\nu_{\text{N-H}}$(Ring4) |
| 3611 | 3540 | 3352$^{c,d}$ | 3555 | 3560 | 3556 | $\nu_{\text{N-H}}$(Ring3) |
| 3326 | 3493 | 3421 | 3417 | 3414 | $\nu_{\text{C=N}}$(Ring1) |
| 3290 | 3484 | 34763336 | 3450 | 3440 | $\nu_{\text{C=C}}$(Ring2) |
| 3277 | 3487 | 3010$^{e,f}$ | 3275 | 3132 | $\nu_{\text{C=C}}$(Ring3) |
| 3266 | 3487 | 3036 | 3236 | 3240 | 3240 | $\nu_{\text{C=C}}$(Ring4) |
| 3359 | 3096 | 3028 | 3024 | 3032 | $\nu_{\text{C=C}}$(Ring1,2,3) |
| 3334 | 3071 | 3066 | 3055 | 3064 | $\nu_{\text{C=C}}$(Ring1,2,3,4) |
| 1881 | 1762 | 1500$^{g,h}$ | 1680 | 1680 | - | $\nu_{\text{C=C}}$(Ring1,2) |
| 1854 | 1825 | 1636 | 1636 | 1640 | $\nu_{\text{C=C}}$(Ring1,2) |
| 1820 | 1724 | 1746 | 1746 | 1766 | $\nu_{\text{C=C}}$(Ring3) |
| 1650 | 1537 | 1554$^w$ | 1560$^w$ | 1558$^m$ | $\nu_{\text{C=C}}$(Ring1,2) |
| 1551 | 1504 | 1496 | 1493 | - | $\nu_{\text{C=C}}$(Ring3) |
| 1576 | 1544 | 1294$^i$ | 1381 | 1381 | 1384 | $\nu_{\text{C=C}}$(Ring1,2) |
| 1512 | 1389 | 1415$^{j,k}$ | 1314 | 1315 | 1296 | $\nu_{\text{C=C}}$(Ring1) |
| 1426 | 1256 | 1293$^l$ | 1230 1288 | 1230 | 1234 | $\nu_{\text{C=C}}$(Ring4) |
| 1375 | 1262 | 1127,1153 | 1176 | 1176 | 1172 | $\delta_{\text{C-H}}$(Ring4) |
| 1283 | 1230 | 1175,1312$^{c,d,g,h}$ | 1230 | 1238 | 1234 | $\delta_{\text{C-H}}$(Ring1,2) |
| 779 | 995$^{d}$ | 806 | 802 | 798 | $\gamma_{\text{C-H}}$(Ring1) |
| 1172 | 1009 | 860$^m$ | 999 | $\approx$1003 | 995 | $\gamma_{\text{C-H}}$(Ring2) |
| 832 | 955 | 902$^e$ | 952 | 956 | 956 | $\gamma_{\text{C-H}}$(Ring3) |
| 986 | 744$^p$ | 898 | 848 | 837 | $\gamma_{\text{C-H}}$(Ring4) |
| 849 | 1039 | 806 | 802 | 798 | $\delta_{\text{N-H}}$(Ring3) |
| 743 | 431 | 752 | 748 | 740 | 740 | $\gamma_{\text{N-H}}$(Ring3) |
| 603 | 307 | 506 | 502 | 504 | 504 | $\gamma_{\text{N-H}}$(Ring4) |
| 1074 | 1050 | 1091 | 1087 | absent | Breathing Ring1 |
| 1015 | 1126 | 1130 | 1138 | 1138 | Breathing Ring2,3,4 |
| 1023 | 1014 | 1062 | 1065 | 1065 | Breathing Ring4,3,2 |
| 1632 | 1577$^i$ | 1620 | 1620 | 1616 | $\nu_{\text{C-C}}$(Ring1) |

$^a$ Experimental wave number (18) $^b$ Experimental wave number (21) $^c$ Experimental wave number (22)

Raman of Spectroscopy

Fig.3 shows the Raman of a spectrum of polyaniline. The incidence of the conducting PANI (emeraldine salt) was also sure of the most important band set at 1354 cm$^{-1}$ corresponding to the C-N bond. This so-called “protonation band”. Characterizing the protonation state of the PANI is more intense in the PANI spectrum (35). The band at 1565 cm$^{-1}$ has been gave to an in-plane bend the C-C tie of the quinoid loop of the doped polyaniline. The stretching band at 1292 cm$^{-1}$ is attributed to C-N$^+$ vibration of delocalized polarons in the extended polymenicchain. (36). A small number of the band was taken at 1063 cm$^{-1}$ an in-plane distortion of the C-C chains of the quinoid ring from polyaniline. The band at 2185 cm$^{-1}$ gave in C=C-C triple bond stretching. (37).
Table 2. Comparison for some empirical oscillate numbers (cm\(^{-1}\)) from shapes in Raman spectra of the nanopowder and thin membranes of polyaniline and the theoretical compatibility frequencies for bianiline and aniline in cm\(^{-1}\).

| Bianiline | PANI | PANI expt\(^b\) | Assignment | Raman | observed \(\omega\) |
|-----------|------|-----------------|------------|-------|------------------|
| 1844Ag    | 1633\(^g\) | 1618\(^a\) | \(\nu_{C=C}(B\,\text{ring})\) | 1635 or 1774 |
| 1682Ag    | 1592\(^g\) | 1578\(^a\) | \(\nu_{C=C}(B\&Q\,\text{ring})\) | 1593 |
| 1662Ag    | 1604\(^g\) | 1615\(^a\) | \(\nu_{C=C}(B\,\text{ring})\) | 1593 |
| 1646Ag    | 1472\(^g\) | 1480\(^i\) | \(\nu_{C=C}(\text{amine})\) | 1480 |
| 1533Ag    | 1562\(^g\) | 1564\(^i\) | \(\delta_{N-H}(\text{semi-Q})\) | 1558 |
| 1616Ag    | 1220\(^g\) | 1220\(^i\) | \(\nu_{C=C}(\text{amine})\) | 1348 |
| 1152Ag    | 1166\(^g\) | 1163,1165\(^u\) | \(\delta_{C-H}(\text{semi-Q})\) | 1255 |
| 1256Ag    | 1074\(^g\) | 1076 | \(\delta_{N-H}(B)\) | 1083 |
| 864Ag     | 811\(^g\) | 815 | \(\delta_{C-C}(\text{BRing})\) | 816 |
| 750Ag     | 785\(^g\) | 788\(^i\) | Breathing | 1019 |
| 683Ag     | 743\(^g\) | 750\(^i\) | \(\delta_{\text{Ring}}(\text{amine})\) | 745 |
| 512Ag     | 611\(^g\) | 632\(^i\) | \(\delta_{\text{Ring}}(\text{amine})\) | 676 |

\(^{a}\)Experimental wave number (38) \(^{b}\)Experimental wave number (39) \(^{c}\)Experimental wave number (40) \(^{d}\)Experimental wave number (41) \(^{e}\)Experimental wave number (42) \(^{f}\)Experimental wave number (43,44).

\(\omega\) (cm\(^{-1}\)) Symbols: B, benzenoid loop; Q, quinoid loop or benzene ring with quinoid Figure; \(\nu\), dilation; \(\delta\), in-plane curvature; \(\gamma\), out of a level.

Figure 3. Raman spectrum of polyaniline thin films prepared by 0.1M of aniline. X-ray diffraction analysis

Fig.4 shows X-ray diffraction pattern of PANI thin film. XRD analysis of the prepared sample of thin film and nanopowder of polyaniline were diffracted meter system which records the intensity as a function of Bragg’s angle. Our Data was taken from the 2\(\theta\) range of 10 to 80 degrees and listed in Table 3.

Table 3 XRD analysis of PANI thin film.

| \(2\theta\) | 15.5\(^a\) | 22\(^a\) | 26\(^a\) | 27\(^a\) | 30.7\(^a\) | 38\(^a\) | 44\(^a\) |
|-----------|----------|----------|----------|----------|----------|----------|----------|
| (hkl) plane | 011 | 020 | 200 | 121 | 022 | 2 1 3 | 222 |

The intense bands observed at different angles of diffraction for a given pair of (hkl) values 2\(\theta\) \(\approx\)11.5\(^a\), 15.5\(^a\)(011), 22\(^a\) (020), 26\(^a\) (200), 27\(^a\)(121), 30.7\(^a\)(022), 38\(^a\) (213), 44\(^a\)(222), 65\(^a\), and \(\sim\)78\(^a\) confirm the synthesis of emeraldine salt of PANI.

Presence of distinguished peaks suggests the formation of highly crystalline and condensed structure of PANI thin films (45). The pattern appearances sharp and fine defined peaks, which show semi-crystalline nature of Polyaniline. The plains of Benzinoid and Quinoid rings of PANI chain are accountable for crystalline assembly(46).

The average crystallite size (D) in Å was calculated by using Scherer’s formula given by Eq. 4.

\[ D = \left[ \frac{K\lambda}{\beta \cos \theta} \right] \]….(4)

Where \(K\) is the shape factor usually has a value 0.94, \(\lambda\) is the X-ray wavelength and \(\theta\) the Bragg angle and \(\beta\) offers, the full width of the half maxima (FWHM). The determined average crystallite size was 32.45 nm.

Figure 4. X-ray diffraction pattern of PANI thin films
UV-Vis Spectroscopy

Fig. 5 shows the absorbance as a function of the wavelength observed from the graph appearing two peaks. For absorption films (PANI), a note from the graph the appearance of two peaks of the absorption of the first peak appears at the wavelength (361 nm) indicating the transitions $\pi \to \pi^*$ (47). The second peak appears at a wavelength (311 nm) due to the transitions $\pi \to \pi^*$ as a result of the transition from the nitrogen oxide-containing non-atomic electrons to the conduction package. Reaching a peak of 361 nm into the level of doping and polaron formation (48). (protonation in PANI backbone, polaron/bipolaron transition that occurred in doped PANI) (49).

Electrical Conductivity

Electrical conductivity is to a great extent dependent on the way of polymer synthesis, purging of the polymer, physical action of the polymer etc. In adding to the environment of the dopants and the method of doping. The conductivity of polyaniline is measured in different capacities by a four-point investigation mechanism and thereafter studied via such equation:

$$\sigma = \frac{L}{(R*S*d)} \ldots (5)$$

in which the conductivity is calculated from the resistance, $R$, between two sections separated by distance, $L$, having an average area, $S$. wherever $d$ is the thickness of membranes, $\sigma$ is the conductivity (50). The conductivity of pure PANI appearances metal behavior in which conductivity losses slightly with growing temperature (22), Fig. 6 and 7 show that.

Atomic Force Microscopy (AFM)

The face morphology of the thin membrane from the polyaniline as seen from the atomic force microscopy (AFM) appear that the grains are uniformly divided into the survey area (2548.46 nm x 2586.84 nm). A first photographic examination from the placed membrane substrate has displayed is compacted and have a good commitment to the substrate. Fig. 9 appear 3D and 2D in order to nanostructure of polyaniline. The thin membrane has been placed on ITO substrates through employ electrochemical polymerization (19). The surface roughness average of the polyaniline film is 6.46 nm and the average particles size is 83.43 nm. These results can be shown in Fig. 8 (a, b). The particle distribution of PANI thin film is shown in Fig. (c) showing a 10% molecular size= 45.00 nm and 50% Diameter = 75.00 nm and 90% Diameter =120.00 nm.
The SEM image of the thin film of PANI is shown in Fig. 9. As can be seen from this Figure, the PANI nanoparticles indicate that there is uniform distribution of the polyaniline nanoparticles, which are spherical in shape in the PANI matrix with few gold clusters of particles (51).

**Conclusion:**
Polyaniline production by electrochemical polymerization produced a nano-thin film using electrolytic middle contained Lithium tetrafluoroborate and acetonitrile, the size of its particles is about 83.43 nm. Noted nanopowder was also produced using acidic solution and its molecular size was 128.43nm. The samples were examined using FT-IR and RAMAN and compared with the theoretical results found to be approximated. Note that the electrical conductivity is inversely proportional to the opposite temperature. The variety, quantity and sizes of the nanostructures manufactured depend powerfully on the electropolymerization settings such as monomer concentration, scan rate, and the would be.

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الخصائص البصرية والكهربائية للتصنيع الكهروكيميائي لغشاء رقيق من البولي أثيلين
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الخلاصة:
تم انتاج أغشية رقيقة من البولي أثيلين باستخدام طريقة التحلل الكهروكيميائي للانيلين في خلية مكونة من قطبين. وتم ملاحظة تأثير الشوائب على لون الغشاء الرفيع، التي تغيرت من الأزرق إلى اللون الأخضر الداكن. تم تشخيص PANI بواسطة تقنيات FT-IR و Raman (Raman) حيث شمل دراسة البلورية للمدخنات ATPS، باستخدام تقنية حيود الأشعة السينية. تم تحديد الانتقالات الإلكترونية المختلفة للبولي أثيلين الحالي من خلال قياس التوصيلية الكهربائية للعينات المصنعة باستخدام تقنية الأقطاب الرباعية (AFM، المجهر الماسح الإلكتروني (SEM)) في درجة حرارة الغرفة. ان طريقة رفع سطح البوليمر تم قياسها باستخدام كلاً من المجهر الفوتوغرافي والمجهر الماسح الإلكتروني (AFM) والمجهر الماسح الإلكتروني (SEM).

الكلمات المفتاحية: بولي أثيلين، الطرق الكهروكيميائية، FT-IR، دراسة الأقطاب، XRD، خصائص الموصلات.