Synthesis and characterization of a novel polyaniline@montmorillonite-copper (II) sulfate nanocomposite

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Abstract. Polyaniline (PANI) and its nanocomposites (PANI-MMT and PANI-MMT-Cu) were prepared with montmorillonite nanoparticles as well as the preparation of nanocomposite polyaniline with adjusted copper sulfate montmorillonite clay. As X-ray fluorescence and X-ray diffraction technology were used to calculate nanocomposite type and crystal sizes of PANI, PANI-MMT, PANI-MMT-Cu, respectively, samples were measured. The FTIR charts revealed new absorption peaks for nanocomposites that varied from polyaniline absorption values, and this is proof of nanocomposite preparation. UV calculations were often used to calculate the overall wavelength, red, and blue shifts. To inspect the morphology of polyaniline and its nanocomposites, FESEM was also carried out.

Keywords: polyaniline, nanocomposite, montmorillonite, and XRD.

1. Introduction
Polyaniline (PANI) is one of the most studied conductive polymers for electronic, optical, gas sensors and as corrosion protection due to its tunable conductive properties, thermal stability and unique doping properties – dedoping and redoxing [1–3]. The material has poor mechanical properties, requires expensive organic synthesis, and is not amenable to a straightforward chemical process [4]. There have been several suggested strategies for solving these problems, including the use of conventional thermoplastic polymers [5]. In situ polymerization of montmorillonite and exfoliation of the product was performed simultaneously [6]. The highly purified polyaniline's thermal stability was improved because of the layered clay coating of the particles. The superposition consists of two separate (two mediums) or (two phases), namely a continuous phase or (substance or medium), and is called a matrix [1,7–9]. Simultaneously, the other is the phase or dispersed medium and is also referred to as (substance material additives or filler material) [10]. Composite materials may be divided according to the matrix’s nature, including polymerization, ceramic, and mineral complexes [3,9,11,12]. The filler is typically incorporated into the host matrix to create a material with properties far from any process [13–16]. In comparison to other materials such as metals and ceramics, polymers have also benefited. Their unique advantages include ease of processing, lightweight, and ductility, and they are commonly used in various technological applications [17]. However, its mechanical properties and strength are lower than those of metals and ceramics [18,19]. The commercial importance of polymers and their increasing use leads to a constant demand to improve their properties to meet the necessary conditions [20,21]. Compound technology improves the properties of polymers while maintaining their light weight and ductility [22,23]. Polymeric nanocomposites consisting of organic polymers and inorganic nanoparticles in nano-scaled regimes constitute a new class of materials that have shown a significant interest in recent years compared to both micro-and macro-scale traditional polymer composites [24]. Montmorillonite (MMT) is a be-long hydrophilic mineral consisting of stacked aluminium octahedron and silicon tetrahedron layers [25]. It is a 2:1 general family (so-called smectite).PANI-MMT nanocomposites are prevalent and interesting among organic-inorganic nanocomposites because of the particular properties of polyaniline, the composition, abundance, low cost of MMT and attractive features like large surface area and ion exchange [26]. In this paper, PANI synthesized a novel material with modified-clay (polyaniline@montmorillonite-copper
(II) sulfate) at room temperature. The UV-vis, FTIR, XRD, XRF, and FE-SEM measurements characterize the PANI/modified clay.

2. Experimental part

2.1. Materials and instruments

Aniline and Montmorillonite clay were purchased from Sigma Aldrich. Methanol and cupric sulfate were obtained from B.D.H. The instrumentals were used in the present work, X-Ray Fluorescence (XRF), X-ray diffraction 6000 (XRD) FT.I.R spectroscopy (Breuker 8400), Field-Emission Scanning electron spectroscopy (FE-SEM, ZEISS 450), and Ultraviolet–visible spectroscopy(UV-Vis Spectroscopy).

2.2. Preparation of polyaniline and polyaniline@montmorillonite-copper (II) sulfate

0.9 mL of aniline has been dissolved in a known volume of 10 mL ethanol with continuous stirring. 0.5 mL of a solution of copper sulfate (0.1 mol dm\(^{-3}\)) was added in drops very slowly at room temperature under constant magnetic stirring. During the distillation of copper sulfate, the color of the solution changed to a light green color, and after the completion of the addition of copper sulfate, the color of the solution became dark green, indicating the completion of the polymerization process [27], and the sediment was then washed several times and dried at 70\(^{0}\)C by a vacuum oven for 8 hours. As shown in Figure 1.

![Figure 1. Synthesis of polyaniline by the polymerization process](image)

After that, copper sulfate (CuSO\(_4\)) was added, where 2 grams of montmorillonite clay was taken with 7 mmol of copper sulfate and dissolved in a known volume of ionic water 500 mL with the use of a mechanical stirrer for 24 hours. After the end of the stirring period, the sediment was filtered and washed several times with deionized water, then dried at 70\(^{0}\)C for 24 hours.

3. Result and discussion

X-ray fluorescence (XRF) measurements of montmorillonite clay and montmorillonite clay - copper sulfate were taken to determine the proportions of the elements in it the extent of the modification of the new composition as shown in Table 1. It is clear to record an increase in the ratio of MgO, Al\(_2\)O\(_3\), SiO\(_2\) and Fe\(_2\)O\(_3\) and a decrease of Na\(_2\)O.

| Table 1. XRF for montmorillonite clay and montmorillonite clay - copper sulfate |
|-----------------------------|-----------------------------|-----------------------------|
| Element                    | MMT                        | Element                    | MMT-CuSO\(_4\)               |
|                            | Concentration %            |                            | Concentration %              |
| Na\(_2\)O                   | 0.606                      | Na\(_2\)O                   | 0.479                       |
| MgO                        | 1.654                      | MgO                        | 1.799                       |
| Al\(_2\)O\(_3\)             | 15.04                      | Al\(_2\)O\(_3\)             | 15.74                       |
| SiO\(_2\)                   | 54.71                      | SiO\(_2\)                   | 57.03                       |
| Cl                          | 0.694                      | SO\(_3\)                    | 1.821                       |
| Cao                         | 0.628                      | CuO                        | 1.349                       |


|      | Fe₂O₃  | 3.725  | Fe₂O₃  | 4.196  |

Figure 2 shows the XRD patterns of MMT and MMT-CuSO₄, shows the pattern (diffractogram) plotted between the intensity of the reflected radiation according to the different angles of diffraction. It’s clear the inversion values that appeared at the angles' values of 22.140°, 34.921° and 62.038° which correspond to the values of Miller (020), (006) and (080) with the basal spacing values between the levels at (0.146, 0.250, 0.392) nm, respectively, as shown in Table 1. Furthermore, the Miller value (080) for a value of 2 θ = 62.038° is an important indication of the naturalness of the composition of MMT to be in the di-octahedral structure [28].

![XRD patterns of MMT and MMT-CuSO₄ clays.](image)

The peaks that illustrated in the pattern of the X-ray spectrum give an indication of the nature of the composition of the montmorillonite clay with the formula MMT-CuSO₄ structure. In addition to the emergence of other peaks in Fig. 2, belonging to the crystalline form of Quartz (SiO₂). It is also shows the diffractogram of MMT - CuSO₄ after a process of dispersing the copper sulfate salt in a certain weight ratio (7 mmol) with the MMT montmorillonite clay in the aqueous medium. The results showed that the same dominant peaks of the silica layers with a slight change in the values of the reflection angles (2θ) with relatively lower values appearing clearly at the angles 21.877°, 34.722° and 61.859° with the interpolation and interference of CuSO₄ between the layers of silica, which gave a relative increase in the values of the interlayer distances, as shown in Figure 1. Also, the average crystal size (Table 2) of the MMT and MMT-CuSO₄ was estimated using the Debye-Scherrer equation (D = kλ/βcosθ) [29] which is 6.547 and 7.867 nm, respectively. It is necessary to indicate that there is a value of 2θ that appears in the range of ~ 6°. We could not determine it because the measurements were made within the range (10°-80°) for MMT and MMT-CuSO₄ as indicated by Shivani [30] and co-workers.
The synthesized polyaniline has a chemical structure in the emeraldine salt form, as shown in Figure 1 was investigated through XRD technique to study the d-spacing levels and crystallinity of polyaniline as-prepared by the chemical oxidant. The XRD patterns shown in Figure 3 illustrates that the polyaniline nanoparticles displays are amorphous reflection (020) at 2ϴ = 20.02°, also another peak at 2ϴ = 6.8°, which revall the local crystallinity of polyaniline nanoparticles. This is peak may be established by the scattering along the orientation paralleled to the polymer chains. The peak at 2ϴ=20.02° also represents the close-contact distance between the ring planes across chains in adjacent benzene rings. These results demonstrated that the PANI chain in nanoparticle size becomes more compacted and ordered (21.17-25.36) nm. The PANI nanoparticles produced by our method have higher crystallinity by the copper sulfate as an oxidant agent. The (100) reflection at 2θ = 17.92° is attributed to the parallel and perpendicular polymer chain periodicity of PANI. A peak centered at 2θ = 25.28°, assigned to the (200) plane of the orthorhombic unit cell of PANI, is due to scattering from PANI chains at interplanar spacing. It can be seen from the graph that the neat PANI has relatively high crystallinity. The peaks corresponding to 44.15°, 47.50°, and 50.78° are due to the nanocrystalline structure of PANI in the solid-state [27]. It denotes the successful synthesis of polyaniline in nanoscale.

| Table 2. XRD values for MMT and MMT-CuSO₄ |
|------------------------------------------|
| 2Θ (deg)  | FWHM | Crystal Size (nm) | Intensity | d-spacing (nm) | Miller index |
|-----------------|-------|------------------|----------|----------------|--------------|
| MMT             |       |                  |          |                |              |
| 19.979          | 0.862 | 4.77             | 90       | 0.433          | (001)        |
| 22.140          | 0.86  | 4.80             | 62       | 0.392          | (020)        |
| 34.921          | 0.60  | 7.08             | 47       | 0.250          | (006)        |
| 62.038          | 0.496 | 9.54             | 41       | 0.146          | (080)        |
| MMT-CuSO₄       |       |                  |          |                |              |
| 19.699          | 0.772 | 5.33             | 122      | 0.440          | (001)        |
| 21.877          | 0.386 | 0.70             | 55       | 0.396          | (020)        |
| 34.722          | 0.596 | 7.13             | 41       | 0.252          | (006)        |
| 61.859          | 0.471 | 8.31             | 40       | 0.146          | (080)        |
Figure 3. XRD patterns of PANI, PANI/MMT and PANI/ MMT-CuSO₄.

The XRD patterns of the PANI/MMT and PANI/ MMT-CuSO₄ are shown in Figure 3, and the resulting structure and parameters are evaluated by Miller values at (001) and (020). The peaks marked as PANI/MMT and PANI/ MMT-CuSO₄ indicate swelling clay and confirm montmorillonite's clay mineralogy. The peak, which appears around approximately 2θ ≈ 6°, incorporates the same PANI value at the same value of reflection angle. The X-ray diffraction peaks match with the JCPDS data (00-007-0330, K₆Al₄(Si₁₈O₅₂)(OH)₂₄H₂O) for the PANI/MMT and (00-029-1498, Na₀.₃(Al, Mg)₂Si₄O₁₀(OH)₂·₄H₂O) with (00-045-0410, Cu(NH₃)₂SO₄) for PANI/ MMT-CuSO₄.

The infrared spectrum displays absorption peaks for MMT-Cu, MMT, where a peak of 3365 cm⁻¹ of the N-H bond appears. It also has a peak of 1604 cm⁻¹ that is part of the double bond. Due to the presence of the C-H clamp, there is also a peak absorption at 2921cm⁻¹. The C-N bond is attributable to its peak at 1109 cm⁻¹, as shown in Figure 4.
Figure 4. Infrared spectrums of MMT-Cu, MMT

The infrared spectrum of (PANI / PANI- MMT/ MMT-CuSO₄) (Figure 5) indicates that the N-H stretching vibration of the (N-H) bond is 3261.04 cm⁻¹. While the apparent peak at 1494.56 cm⁻¹ is due to the vibration of the quinoid ring double bond, the peak at 1604.48 cm⁻¹ is due to the benzoid ring double bond. Whereas the sharp peaks occurring at (1110.8, 1166.72) cm⁻¹ are due to the (C-N) bond vibration, and at 586.25 cm⁻¹ is essentially due to the presence of the aromatic ring's C-N-C bond. The band at 3446.17 cm⁻¹ corresponds to the (OH) stretching vibration, while the apparent peak of the bended vibration of the (Si-O) bond has a peak of 1024.02 cm⁻¹. The peak at 470.01 cm⁻¹ is due to the vibration of the O-Si-O bond. It indicates the presence of montmorillonite clay, peak of MMT- Cu bond is disappeared due to the overlap of peaks, and a small amount of copper sulfate may not make it appear clearly in the spectrum.

Figure 5. Infrared spectrum (a) PANI (b) PANI - MMT, and (c) PANI / MMT-CuSO₄
The UV–visible spectrum (MMT) shows the appearance of a broad peak of absorption at 275 nm, explaining the existence of iron ions (Fe$^{2+}$) as an alternative to the interlayer (Ca$^{2+}$) and the presence of strong absorption in the ultraviolet region is indicated by these mineral ions, or the cause may be due to the transfer of the metallic cation charge as described in previous studies [31]. The UV–visible spectrum of the clay revealed that it has an emerging absorption peak at a wavelength of 277 nm. A decrease in wavelength indicates the presence of a shift towards a shorter wavelength, indicating that the MMT has been modified as shown in Figure 6, in accordance with the results from previous XRF analysis in our present work.

![Graph showing UV–visible spectra of MMT and MMT-Cu.](image)

**Figure 6.** UV–visible spectrums of MMT, and MMT-Cu.

The UV–visible spectrum of PANI (Figure 7) shows a peak absorption at 244 nm due to the presence of ($\pi$-$\pi^*$) transitions in benzenoid rings. Thus, the apparent absorption peak at 291 nm is due to the presence of (n-$\pi^*$) transitions in the quinonoid rings. In the PANI-MMT and PANI/MMT-Cu nanocomposites, as shown in Figure 8, the absorption peaks appear with a slight change towards a shorter wavelength (blue shift).
Figure 7. UV-visible spectrums of the PANI, PANI-MMT, and PANI-MMT-Cu.

It is because of the prolonged conjugation resulting from the creation of the orbitals that overlap. The apparent large peak in the wavelength range (404-508) nm is due to the electrons affected by the incident photons being absorbed bipolarly (polaron/bipolaron). The decrease in absorption rate to this peak implies a decrease in the polymer prepared conductivity.

For the PANI images (Figure 8 a), the field emission scanning electron microscope (FESEM) reveals that the polymer has emerged in unique shapes in the form of chips or sheets, as the particle size exceeds 31.74 nm. Figure 8 b displays the EDX of the polymer elements, as the percentage values for the carbon about 49.7 %, oxygen to be 40.9 %, and nitrogen is 9.4%.

Figure 8. (a) FESEM images and (b) EDX for polyaniline.

Figure 9 a shows the change in the particles' shape from thin sheets to larger sheets. All the particles' dimensions had changed. The particle size ranged from 93.79 to 113.9 nm.
This change is due to the addition of MMT to the polymer, where the clay interfered with the spread of the polymeric network, which increased the thickness of the layers, as mentioned in earlier studies [32–35]. Figure 9b, which represents EDX for the PANI / MMT, notes the appearance of elements 0.1% Ca, 0.2% Mg, 0.3% Al, 1.0% Fe, 1.7% Na, 7.4% N, 39.5% O, and 49.8% C.

Figure 10a shows the FESEM image of PANI / MMT-Cu. There is a more significant increase in nanoparticles’ size due to the addition of copper, which is considered to be crystallization centers, which has led to an increase in the crystallization process while maintaining the general shape of the layers. Figure 10b shows the EDX spectrum showing the elements that make up the PANI / MMT-Cu nanocomposite. This figure shows the copper element (Cu) with a weight ratio of 27.9 % and elements C, O, N, Al, N, Si, and S with different weight ratios.

4. Conclusion
The PANI, PANI-MMT, PANI-MMT-Cu were prepared successfully by the oxidation process. The X-ray diffraction results showed PANI, PANI-MMT, and PANI-MMT-Cu, which gave polyaniline peaks that differed from their nanocomposites. Fourier transforms infrared spectroscopy findings have been employed to describe and analyze the effects of the components (MMT and MMT-Cu) in polyaniline polymerization. Ultraviolet-visible spectroscopy indicates that the nanocomposites varied in maximum wavelength, redshift, and blue shift. Polyaniline’s morphology was different from its nanocomposites in the field emission scanning electron microscopy measurement. All these tests confirm that polyaniline and its nanocomposites are being prepared.
5. Reference

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