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A Study of Characterization of Pani and its Derivatives and Copper Doped Polyaniline Nanocomposites

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Abstract - Polyaniline Is An Example Of A Basic Conducting Polymer. Doping May Convert The Conductivity Of A Material From Insulator To Metal While Also Improving Its Mechanical Characteristics And Processability. Polyaniline (PANI) Is The Earliest Known Organic Synthesised Metal In History. Basically In Which Disused About The Polyaniline, Polyaniline Derivatives, Semiconducting Polymer, Applications Of Polyaniline, Uv-Visible Absorption Spectra, Photoluminescence Studies

Keyword - polyaniline,, Polymer

INTRODUCTION

Most polymers are good insulators, chemically inert and structurally easily – mouldable, which make these one of the most used materials in the modern world. Recently, some polymers have been discovered with metallic and semiconductor like conductivity, examples being polyacetylene, polyaniline; polypyrrole, polythiophene etc. These are simple chain polymers possessing conjugated double bonds. The extended π -orbitals of conjugated polymers result in quasi one – dimensional electronic structure with associated non – linear excitations, responsible for modified electrical / optical properties. One of the simple conducting polymers is polyaniline. Doping can change its conductivity over the full range from insulator to metal with attractive mechanical properties and processability. Historically, polyaniline is the oldest known organic synthetic metal. The unique combination of electronic, chemical and mechanical properties of above type of synthetic metals and semiconductors make them useful for electronic device fabrication, non-linear and integrated optics, light emission and magnetic applications. Usually, these polymers are useful in the form of solid bulk or thin films. The thin films are normally made by chemical, electrochemical and solution cast techniques. These techniques frequently involve wet processing and no strict control over the processing parameter is possible. The vacuum deposition method is also used for the preparation of polyaniline thin films. The polymer is evaporated onto suitable substrates in high vacuum. Thin polymer film of desired thickness can be prepared by this method. The films prepared by both wet processing and vacuum deposited types have been used in the present work.

Intrinsically conducting polymers are organic polymers that conduct electricity. Everyday polymers, which are more commonly known as plastics, are composed of simple repeating molecule units called monomers. Polymer name derived from the Greek words „poly" means „many" and „mer" means „part". Polymers are well known for their insulating properties, indeed it is this characteristic that has led to their wide usage in the packaging and electronics. Conducting polymers are differ greatly from these insulating polymers because of they are intrinsically conducting.

Most polymers are good insulators, chemically inert and structurally easily – mouldable, which make these one of the most used materials in the modern world. Recently, some polymers have been discovered with metallic and semiconductor like conductivity, examples being polyacetylene, polyaniline; polypyrrole, polythiophene etc. These are simple chain polymers possessing conjugated double bonds. The extended π - orbitals of conjugated polymers result in quasi one – dimensional electronic structure with associated non – linear excitations, responsible for modified electrical / optical properties.

POLYANILINE

The name "aniline black" has been in use for polyaniline (PANI) from the early nineteenth century. Cotton textiles might be made black with this substance by using it as a dye. Starting at the turn of the XXth century, several efforts were completed with the goal of figuring out the PANI structure. There was a time when scientists believed that aniline black was only a variety of aniline octamers with varying degrees of oxidation. In recent years, it has been shown that aniline oxidation under certain circumstances yields a polymer. Woodhead and Green are credited for naming the three fundamental types of polyaniline (explained below), and these designations are still in use today.

In the early 1980s, polyaniline was identified as a member of a new class of macromolecular compounds conductive polymers marking a significant advancement in the field of polyaniline study. Since then, no other polymer in this class has been examined more thoroughly.

However, the term "polyaniline" really refers to a large family of polymers that may be defined by the formula shown in fig.

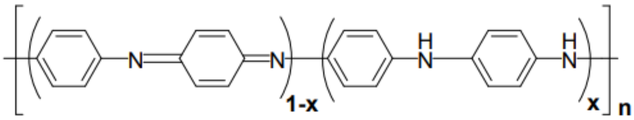
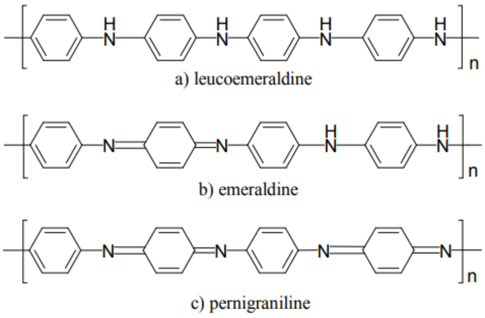


Figure 1. General formula for different forms of polyaniline base

Wudl and coworkers' spectroscopic experiments were among the several that demonstrated the above formula to be correct.

Previously, we established that there are essentially three distinct types of polyaniline. The totally reduced (a) and fully oxidised (c) states are both environmentally unstable, while having fascinating spectroscopic features. White powdered leucoemeraldine (a) is a potent reducing agent that readily forms emeraldine (b) when exposed to atmospheric oxygen. Hydrolytic type degradation by chain scission is a simple process for the oxidised unit-containing compound pernigraniline (c) (red-purple, partly crystalline powder). The semi-oxidized form of PANI, known as emeraldine base (a dark blue powder with a metallic sheen), is stable in air and may be kept for extended periods of time without undergoing any chemical changes. The emeraldine kind of polyaniline has been the subject of the greatest research. Therefore, "polyaniline" and "emeraldine" are often used interchangeably in the literature.



a) leucoemeraldine

b) emeraldine

c) pernigraniline

Figure 2. Popular types of polyaniline bases

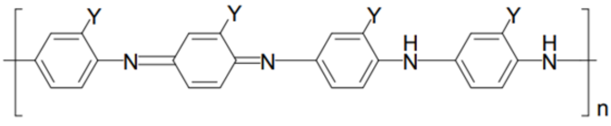
PANI is unique among conjugated systems in that the -electrons of aromatic rings are not the only ones contributing to the conjugation; interactions between the lone electron pairs of nitrogen atoms and electrons (p- interactions) also play a role. In addition, the amine and imine groups in neighbouring chains interact with one another rather strongly in emeraldine base due to the presence of hydrogen bonds. These phenomena are what make the basic form of polyanilines so difficult to comprehend. In order for the PANI base to dissolve in part, interchain interactions must be replaced by solvent-polymer hydrogen bond interactions. Emerald base may be dissolved in a select group of solvents, including N-methyl pyrrolidinone (NMP), tetramethylurea (TMU), and dimethyl acridine (DMA) (N,N-dimethylacetamide). It is possible to witness a gelation process in highly concentrated PANI solutions. A combination of solvents may be used to halt this process (combination of electron donors and acceptors that interact with amine and imine groups, respectively). Conjugation and the presence of hydrogen bonds induce insolubility in most common solvents and are also responsible for PANI's infusibility. Polymer eventually decomposes without melting at very high temperatures (above 400 °C).

The electrical conductivity of pure polyaniline is less than 10^6 S/cm. The band gap between the HOMO and LUMO levels, i.e. 3.8 eV, is responsible for the low conductivity. An electrically conductive version of PANI may be created. The doping procedure section will address this issue.

POLYANILINE DERIVATIVES

Polyaniline ring-substituted derivatives are known from the beginning of XX century. In 1910 Green and Woodhead studied the synthesis of 2-methyl substituted polyaniline, i.e. poly (o-toluidine). These polymers can be obtained using “ortho” as well as “meta” substituted aniline. The chain structures of the polymers obtained by the polymerisation of aniline derivatives, which differ only in the position of the side group (such as ortho-toluidine and meta-toluidine), seem to be almost identical independently whether the chemical synthesis or the electrochemical one is applied. Small differences are caused mainly by the fact that in the case of o-substituted anilines one of the “ortho” positions is blocked - it diminishes the percentage of undesirable “ortho” coupling. The product has a more regular structure which, consequently, leads to higher polymerisation yields. “Para” substituted anilines do not polymerise, because long chain formation is blocked by the “para” substituent; the formation of oligomers is observed in this case.[6]

For above reasons o-substituted anilines were chosen in this research as the monomers for the polymerisation and for the comparison with polyaniline.



Y= $-CH_3$; $-CH_2CH_3$; $-OCH_3$;etc.

Figure 3. Ring substituted Polyaniline in semi-oxidized form

The properties of the obtained polymers depend principally on the type and the size of substituent. An improvement in the solubilisation was one of the main goals of this research, so relatively flexible alkyl and alkoxy groups were chosen. Polymerization of monomers with bulky side group improves solubility, but drastically decreases the molecular weight.

It should be emphasized, that the methods applied for the syntheses of alkyl and alkoxy ring-substituted poly (amineimines) are generally the same as for Polyaniline both in chemical as well as in ectrochemical preparations. Similarly to the Polyaniline case; chemical route (using ammonium persulfate) is frequently used.

Moreover, it was shown that the polymerization mechanism of aniline and its ring-substituted derivatives is analogous. Unfortunately, optimization of the polymerization process seems to be less developed comparing to the case of Polyaniline, i.e. low temperature synthesis (below –5 °C) of ring substituted Polyaniline derivatives was not systematically investigated to date. It may be noted, that other types of substituted poly (amineimines) were also obtained, namely N-substituted Polyaniline derivatives (alkyl or phenyl as substituents), poly (2, 3-dimethylaniline) and Polyaniline derivatives with halogen atom attached to the aromatic ring.

Semiconducting Polymer

The conducting polymers, in general, can have conductivity in the range 10^{-12} to 10^{1} ohm^{-1} cm^{-1} encompassing the entire spectrum of materials like insulators (σ< 10^{-7} ohm^{-1} cm^{-1}), semiconductors (σ in the range 10^{2} – 10^{-7} ohm^{-1} cm^{-1}) and metals (σ> 10^{2} ohm^{-1} cm^{-1}). Such variability in electrical conductivity has been mostly achieved in “conjugated” polymers by anion / cationdopings. Broadly, conducting polymers can be classified as:

1. Ion Conducting Polymers

The charge carriers are ions like Li+ , H+ , Na+ , Ag+ , Pd ++ , Cd ++, etc. Examples of conducting polymers are Polymethylacrylate mixed with $LiClO_4$, POLYANILINE doped with palladium and cadmium and Polyanilinecompleted with H_3PO_4 etc. These are useful in polymer batteries, fuel cell and electrochemical sensors.

2. Electron - Conducting Polymers

Here the charge carriers are electrons and/or holes. These polymers could have conductivity of the order of metals (thus are often termed as synthetic metals) or semiconductors. The major classifications of semiconducting polymers are as follows.

- **Polymer- Metal Composites**

In these composites metallic, carbon black etc. particles are disposed in a suitable polymeric matrix. Polyaniline-metal oxalate composites also constitute the compounds of this category.

- **Charge - Transfer Complex type**

This is possibly the just most established semiconducting organic system because of its commercial use in Xerographic photo - receptors. The first of such devices was a single layer charge transfer polymer named as tri- nitro fluorenone doped poly vinyl carbozole (PVK). Most charge transfer polymers including PVK, triarylamine doped polycarbonate and polysilanes are p-type materials. Although n-type materials have been reported, their charge mobility of 10-2 cm2 /V.s is about three orders of magnitude lower than p-type materials. The lack of high mobility electron transport materials is a critical weakness associated with semiconducting organics. The world of semiconducting polymers is likely to remain p-type, till high mobility n-type materials are discovered.[7]

- **Conjugated Polymers**

It is the discovery of such materials that polymeric electronic devices are becoming increasingly possible. Polymers having alternate single and double carbon-carbon (sometimes carbon- nitrogen) bonds are known as conjugated polymers. Single bonds are referred to as sigma (σ) - bonds and double bonds contain a σ - bond and a π - bond. All conjugated polymers have a sigma – bond backbone of overlapping sp2 hybrid orbitals. The remaining out of plane pz orbital on the carbon (or nitrogen) atom overlaps with the neighboring pz orbital to give a π - bond. It turns out that the best delocalization of π - electron. is achieved by having alternate single and double bonds between carbon atoms (although semiconducting bonds are not limited to C – C bonds). In this case, bringing together of more and more atoms to one molecule, the levels will split up, but still these would stay close in energy. This is called “conjugation” and the “conjugation length” determines how many bonds in a row we have, with alternating single and double bonds.

Applications of Polyaniline

The study of conducting polymers has become a major part of modern material science and in many institutes and commercial establishments. This involves multidisciplinary research in chemical synthesis, polymer preparation, electronics, physics and applied physics. The combination of metal-like or semiconducting conductivity and process ability of classical polymers has created opportunities for scientists and technologies to investigate possible technological applications.

LITERATURE REVIEW

Sachin S. Bangade, Vivek M. Raut et al.,(2020) The conductivity of polymers such as polyaniline, polythiophene, polypyrrolepolyacetylene, and others has been investigated intensively by polymer scientists during the past thirty years. A significant group of hybrid materials falls under this heading. All around the globe, because to its large electro-active surface area, high specific capacitance and electrical conducting capacity, it is utilized in laboratories instead of metal conductors for high stability, A conjugated conducting polymer is a kind of this material. Polyaniline (PANI) has this property because of the -electron. Polyaniline contains an imine-conjugated -conjugated backbone structure. The metal ion may be stabilized in an aqueous medium as well as a polymer via -interactions, steric effects, and hydrophilic interactions due to its structure and group. Due to its inherent electrical conductivity, simple and quick synthesis process, high environmental stability, cheap and easy monomer supply and behavior redox characteristics, Polyaniline has gained considerable attention in the past 10 years. [8]

Sacchidanand S. Shinde, JayantA.Kher(2014) Polyaniline (PANI), a conducting polymer with excellent environmental stability, high processability, and intriguing redox characteristics related with its chain heteroatom, has been the subject of intense research over the last 10 years. Here, for the first time, is a summary of recent research into Polyaniline derivatives and their numerous inherent oxidation states as well as their synthesis, characterizations, development and applications. Several benefits of Polyaniline and Polyaniline Nobel metal composites are discussed in the study, as well as the superiority of Polyaniline. To better understand how PANI is made, this article explains the many ways used to synthesize it as well as the mechanisms, theoretical research and number of specific procedures utilized to create it. [9]

Juan Francisco Armijo, **Eduardo Ortega et al.,(2013)**The substituents inserted into Polyaniline' monomers provide their macroscopic characteristics a wide range of variation. In polymers that conduct, conductivity may rise or decrease depending on the substituent and where it is located in the chain. For the purpose of this study, the properties of aniline monomers are altered, allowing the influence of a substitute on the polymer to be evaluated. Suzuki and Ullman reactions were used to create thiophenes using commercially available methoxy- and bromine-substituted monomers for this purpose. All polymers were found to have conductivities below 10-6 S.cm-1. As compared to aniline, the substitutions made had a detrimental impact on conductivity (albeit they would make them more suitable for solar cells) (PANI). Because they were more soluble than PANI, these alterations have also improved the solubility of the respective polymers. [10]

M. Inés Toral, Nelson Lara, Javier Gómez & Pablo Richter (2002) Solid phase derivative spectrophotometry has been developed to simultaneously measure iron and copper at sub-microg/L levels. To create a blue iron complex and an orange copper complex (both at pH = 5.0), chromogenic reagents TPTZ (2,4,6-tripyridyl-1,3,5-triazine) and Neocuproine (2,9-dimethyl-1,10-phenanthroline hydrochloride) were used. These complexes were then fixed and concentrated on a cation-exchange resin called Sephadexsp C-25. The effect of experimental factors on the resin's ability to retain complexes is examined. The signals in derivative units were obtained by performing spectrophotometric measurements on a solid packed in a 1 mm cell. In third-derivative spectra, the typical amplitudes of the signals at 622.0 and 477.0 nm (RSD = 1.8 percent) and copper (0.451–300 ng mL1; RSD = 1.3 percent) are appropriate for quantitative determination. The approach was used to determine the concentrations of iron and copper in a variety of water samples.[11]

Johnny Easmon (2005) Many enzymes and proteins involved in energy metabolism, respiration and DNA synthesis are dependent on copper and iron, which are present at very low levels in the human body. They may catalyze redox processes in their ionic forms, which are prone to one-electron transfer reactions. However, this ability allows copper and iron to create free radicals, which may activate signaling pathways that promote cell growth. – Cancer cells have been proven to need more copper and iron than normal cells in order to develop and function properly. As a result, drugs that disrupt the cell's homeostasis of copper and iron are highly sought after as potential cancer therapies. For this goal to be achieved by different methods, such as the discovery of new iron-binding siderophores, like exochelins and myochels, with better physical-chemical properties than desferrioxamine, and the development of macrocyclicpolydentate amines with high binding capacity for copper or iron ions in comparison to other trace elements have been pursued.[12]

Ivanov, V.M., Antonova, E.V. &Uskova, E.N. (2009) Carriers (silochrome S120, anion exchanger AB-17, enterosgel and polysorb) noncovalently modified by 8-hydroxynoline, 5,7-dibrom-8-hydroxyquinoline and 8-hydroxynoline-5-sulfo-acid have been explored for the sorption of copper, lead and iron ions. The ideal conditions for sorption have been discovered. A 103-fold excess of potassium, natrium, calcium, and magnesium does not hinder the determination. Using water and soil samples collected in Saransk, the approaches were evaluated. Model solutions were used to examine the systematic error of determination in the option that was introduced/discovered., Using a sample size of five, the relative standard deviation is less than or equal to 0.08.[13]

Bert De Boer and Antonio Facchetti(2008) As long as the backbone of the polymer is aromatic, it may be used in a range of optical and electrical devices, such as photovoltaic cells, photodetector cells, and light emitting diodes. Several kinds of p-conjugated polymers are described and evaluated in this work and the other publications in this special issue. There is also information about organic semiconductor devices such as field-effect transistors (FETs), polymer light-emitting diodes (PLEDs), and organic photovoltaic cells (OPVs).[14]

Suraya Abdul Rashid, SuhaidiShafie et al.,(2021) Preparations, Properties, and Applications of Polyaniline and Polyaniline For the enhancement of sensors, optoelectronic devices, and photonic devices, polyaniline (PANI) is a well-known conductive polymer that has attracted considerable attention from researchers in the area of nanotechnology. In addition to its ease of synthesis and environmental stability, PANI is a good candidate for doping with a variety of acids and dopants. The emphasis of this study is on chemical and physical approaches used to create PANI thin films. This study focuses on the properties of PANI thin films, including their magnetic, redox, and antioxidant, anti-corrosion, and electrical and sensing characteristics. In terms of conductivity, PANI is a polymer. A wide range of applications have been drawn to this material because of its unique features, ease of synthesis and inexpensive cost as well as its great environmental stability. It is at the conclusion of this study that we quickly discuss the most significant PANI applications. [15]

M.A. Sangamesha, K. Pushpalatha, G.L. Shekar (2014) Polyaniline (PANI) and Cu2Se nanocomposite were effectively generated by in situ polymerization using a simple and safe method. Structural analysis was carried out by means of an XRD technique called Brucker D2. At a normal incidence light wavelength of 330-1100nm, the optical measurements were conducted using a UV-Visible spectrometer The existence of functional groups and the interaction between polymer chains and Cu2Se nanoparticles was verified by FTIR spectroscopy. The FT-IR and UV-Visible spectra were found to have altered their typical peaks to higher wave numbers, which is attributable to the above-mentioned interplay. It was clear from the XRD pattern that the polymer composite was well-ordered. Research shows that nanocomposite may be employed as a multipurpose material for diverse nanoelectronic devices. The results gained corroborate this. [16]

PROPOSED METHODOLOGY

A vacuum deposition process and several characterizations was presented for the fabrication of doped and undoped semiconducting nanocrystallinepolyaniline. Polyaniline undoped and its copper and iron derivatives was synthesised in this study, as were other related compounds. Metal-doped Polyaniline thin films were also be produced. UV-visible absorption, Fourier transforms infrared spectroscopy, and x-ray diffractometry would be used to characterize the materials created in the preceding steps. Polyaniline and its derivatives were evaluated based on the data above, and conclusions were reached about their nature. Polyaniline samples containing iron were further tested using the electron paramagnetic resonance method.

DATA ANALYSIS

UV-visible Absorption Spectra

At ambient temperature, a spectrophotometer was used to record the UV-visible absorption spectra of the undoped PAni and the PAni/CuO nanocomposite within the wavelength range of 200-800 nm, as shown in Fig. Understanding the conducting states of conducting polymers that correspond to the absorption bands of the inter and intra gap states requires the use of optical spectroscopy. The primary absorption peaks at around 405 nm are seen in Fig. The degree of conjugation between the neighbouring phenylene rings in the polymeric chain and the forced planarization of the -system brought on by aggregation are both factors that contribute to the bathochromic shift at the strong band 405 that has been observed. Increased conjugation results in a smaller band gap, which is in good agreement with the band gap finding found in polyaniline. The increase in electrical conductivity of the nanocomposites is caused by the transition of the benzenoid ring to the -* state and the development of the polaron band. According to Fig. the addition of CuO filler particles in the polyaniline matrix has a significant impact on the absorption spectra in the PAni/CuO nanocomposite. This is demonstrated by the high intense blue shift of PAni's absorption peaks from their actual position in the PAni/CuO nanocomposite. As the dopant % rises, the absorbance peaks diminish owing to the hypochomic effect, The existence of an electron with a drawing sulfonic group in the complex causes the two spectra to vary, and as a result, the transition band is seen at a lower wavelength.

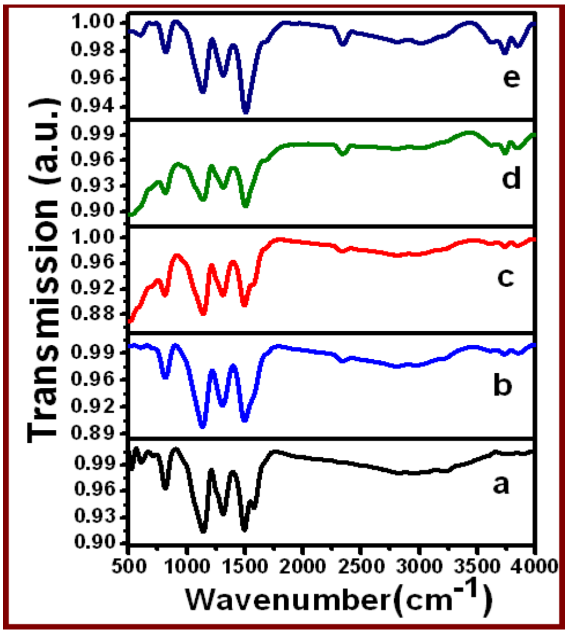


Figure 4. Fourier transform infrared (FTIR) spectra for the sample a, b, c, d and e curves a, b, c, d and e correspond to 0, 2, 4, 6, and 8wt% CuO doped PAni composite respectively.

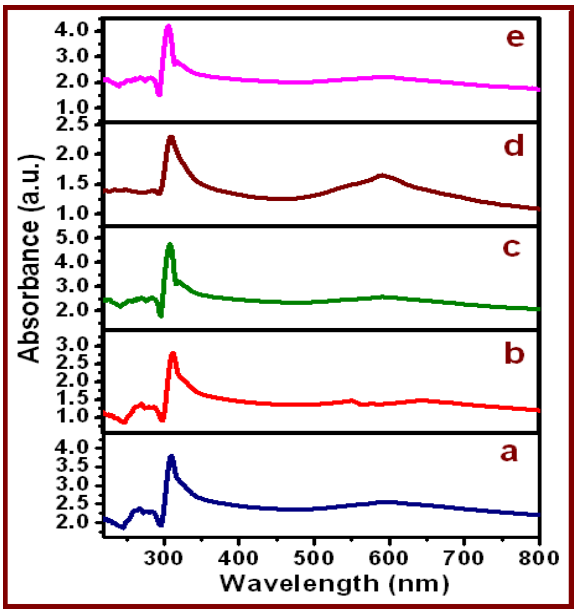


Figure 5. UV–visible absorption spectra for the samples a, b, c, d and e at room temperature. Curves a, b, c, d and e correspond to 0, 2, 4, 6 and 8 wt

% CuO and PAni/CuO composites respectively

Photoluminescence Studies

A new family of substances called photo luminescent organic molecules has several intriguing characteristics. They emit light in a variety of colours, from violet to red. White light may also be created by combining them in a variety of ways. Conjugated organic polymers are one kind of organic substance exhibiting photoluminescence characteristics. All four samples' PL spectra were measured between 400 and 650 nm, and 425 nm was used as the excitation wavelength for all samples. CuO doped PAni has undergone photoluminescence spectroscopy (PL), and the spectra are shown in Fig. The 0, 2, 4, 6, and 8 weight percent PL spectra Peaks in the visible range are seen in CuO doped PAni samples at 462 nm, 405 nm, 459 nm, 486 nm, and 528 nm. Due to polarity, the relative heights of the emission peaks change with varied dopant concentrations and solvent types. Additionally, this peak becomes acute and strong, which may be the result of inter-chain species, which are crucial to conjugated polymers' emission process. The degree of chain overlapping, polymer-solvent interactions, polymer-dopant interactions, polymer coil size, and others all affect peak intensity. The fact that the PL spectra of every sample are the same suggests that using a particular dopant with a varied weight percentage at various concentration levels is an effective technique to tailor the peak intensities. Overall, it is evident that a variety of parameters, such as the size of the polymer coil, the kind of interactions between the polymer and its solvent and dopant, and the degree of chain overlapping, affect the form of conjugated polymer aggregation.

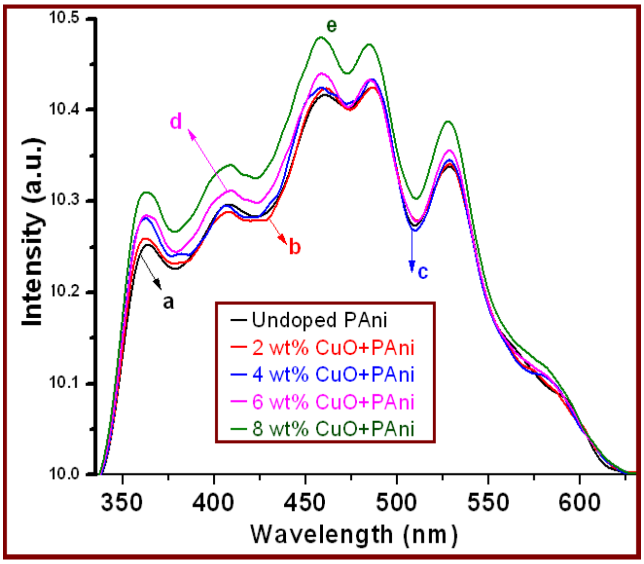


Figure 6. Photoluminescence (PL) spectra for the samples a, b, c, d and e at room temperature. Curves a, b, c, d and e correspond to 0, 2, 4, 6 and 8 wt % CuO and PAni/CuO composites respectively.

CONCLUSION

Polymers that are semi-conducting and conducting are introduced. The conjugated polymers lead to the production of conducting polymers, which has been explained. Conjugated polymer conduction mechanisms are also described. The chemical production of the PANI compound has been explained at the beginning of the article. PANI filmmaking methods are also covered. PANI's many oxidation states and forms are also discussed.

PANI's broad benefits and uses are well-explained in the literature. Polyaniline significance is underscored by its unique conduction mechanism, ease of synthesis, environmental stability, and ease of processing. Polyaniline and its metal ion derivatives are examined using UV-visible spectroscopy, FTIR, and X-ray diffractometry. It has been shown through the use of UV-visible spectra that the emeraldine form of PANI is formed by hydrogen bonding with the solvent molecules, and this provides an introduction to the phenomenon of Electron Paramagnetic Resonance from both a classical and a quantum perspective. The importance of EPR in both fundamental research and applied research has also been highlighted by these results.

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