
PREPARATION AND CHARACTERIZATION OF POLYANILINE (PANI) AND POLYANILINE ENCAPSULATED CELLULOSE FROM HEMP MATERIALS

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ABSTRACT

This paper gives the detail of the Preparation and characterisation of Polyaniline(PANI) and Polyaniline encapsulated cellulose from Hemp materials. Further it also describes the cellulose encapsulated polyaniline nanocomposite . The chemicals which were used for the preparation and characterization are given below. Aniline monomer (SigmaAldrichchemicals) were distilled under reduced pressure and the chemicals ammonium per sulphate (APS, Aldrichchemicals) sodium hydroxide (NaOH, sigma), ethanol, Acetic acid, concentrated HNO₃, HCl from Himedia, India were used as such. Cellulose was extracted from raw materials sugarcane bagasse (agro-waste) and hemp. The whole experiment was carried out using double distilled water. The investigation's findings were reviewed and interpreted appropriately in this study. The FT-IR spectra reveal that the peaks in PANI were preserved in the cellulose nanocomposite. The crystalline nature of PANI and their composites was investigated using the X-ray diffraction (XRD) technique. The diffractogram of composites made from sugarcane bagasse and hemp material reveals a considerable shift in the diffractograms PANI, confirming the interaction of cellulose with PANI conducting polymers. The surface morphology of PANI and cellulose, as well as their composites, was examined using SEM. The surface morphology of virgin polymers such as PANI demonstrates the creation of irregular agglomerated structures.

KEYWORDS: Polyaniline (PANI) and PANI encapsulated cellulose, HEMP materials, Polymer characterization, UV, FTIR, XRD, SEM, TGA/DTA techniques etc.

INTRODUCTION

This research is concerned with the extraction of cellulose from agricultural waste materials such as hemp waste materials. Polyaniline (PANI) conducting polymers was synthesized using a chemical oxidative polymerisation technique. Ascorbic acid levels in fruit juices will also be measured electrochemically utilizing PANI/Cellulose composites. Anselme Payen, a French scientist, isolated cellulose from plant materials in 1838 and identified its chemical formula. Cellulose is a polysaccharide composed of a linear chain of hundreds to thousands of β (1, 4-linked D-glucose) units. [1] C₆H₁₀O₅ is the molecular formula for cellulose. The primary sources of cellulose include wood, rice straw, cotton, and so on.,[2] It is a significant component present in both primary and secondary cell walls. The exterior layer of the cell wall is composed of 9-25% cellulose micro fibrils, an interpenetrating matrix of 25-50% hemi cellulose, and 10-35% pectin. Carbohydrates make about 90% of the cell wall, and carbohydrates hold the cellulose fibers together. [3]

Polyaniline (PANI) is a polymer that may conduct electricity. It was produced from aniline monomer; the most common synthetic methods are oxidative polymerization and electrochemical polymerization (consistent voltage, constant current, and clearing voltage

techniques). In addition to these two techniques, interfacial polymerization, electro turning, seeding polymerization, and format polymerization are commonly used to create nanostructured polyaniline.

Compound oxidative polymerization (radical cationic) of aniline is often started in an acidic solution with a suitable oxidizing specialist, such as ammonium persulfate, $(\text{NH}_4)_2\text{S}_2\text{O}_8$, and then proceeds to form polyaniline. The growth of the corrosive doping operator during polymerization might cause the supplied polyaniline to be electrically leading. Polyaniline has a unique polymeric structure that consists mostly of successively switching benzene rings and oxygen particles. The existence of nitrogen molecules in the form of imine (in the sp^2 hybridized state) or amine (in the sp^3 hybridized state) frames, as well as their relative abundance in the total polymer spine chain, dictates the future structure and many properties of polyaniline. According to their reduced/oxidized rehashing unit extents, there are four well-known polyaniline variants available in the literature. Various oxidation states of polyaniline may be achieved employing oxidants such as iodine (I_2), but the subsequent conductivity of these compounds is lower than that obtained by acid doping [4].

Polyaniline has completely decomposed to yield leucoemeraldine [5]. This kind of polyaniline has a slight dark color shading and no directional properties. The emeraldine state of polyaniline lacks leading property and may be identified by its blue hue. The protonated emeraldine structure, which has been somewhat oxidized/diminished, remains the primary electronic driving state of polyaniline. The conductivity of this state is due to protonated charge transporters produced after corrosive treatment of the emeraldine structure (reversible reaction by base treatment) [6].

This polyaniline condition has a dull green hue, a flexible strong structure that is insoluble, infusible, and recalcitrant in natural solvents, and a conductivity range of 2-10 S/cm. In contrast to this structure, emeraldine polyaniline is a processable product with a conductivity of 1-10 S/cm and may be degraded in N-methyl pyrrolidone (NMP) or dimethyl sulphide (DMSO) solvents. Pernigraniline is a completely oxidized form of polyaniline with pink/purple coloration. Unlike the vast majority of analogous polyaromatics, the pernigraniline condition of polyaniline does not display leading properties in its fully oxidized state.

The dopant in the polymer structure has the biggest influence on its conductivity. Polymers, for example, have a lower conductivity than dopants in their structure. As a result, polymers with more dopant material show higher conductivity. Polyaniline has distinct properties, such as a wide and regulated range of conductivity, a naturally fibrillar morphology, and softening or organization processable capabilities that surpass other guiding polymers. This polymer can be employed as a conductive component in various composites containing traditional polymers such as polystyrene. Directing polymers are rapidly being employed in a variety of electronic and electrical applications, including as sensors, light discharge diodes, antistatic devices, and electromagnetic shielding [7].

Polyaniline is the most widely utilized of the several major polymers due to its superior strength and processability.

Polyaniline is a macromolecule made up of monomer pyrrole ring units linked together by a chain. Polyaniline has electrons and alternating double and single bonds. Polyaniline contains a long conjugated compound backbone, as seen in Figure 1. As shown in Fig.1, the polymer has a resonance structure that corresponds to aromatic or quinoid forms.

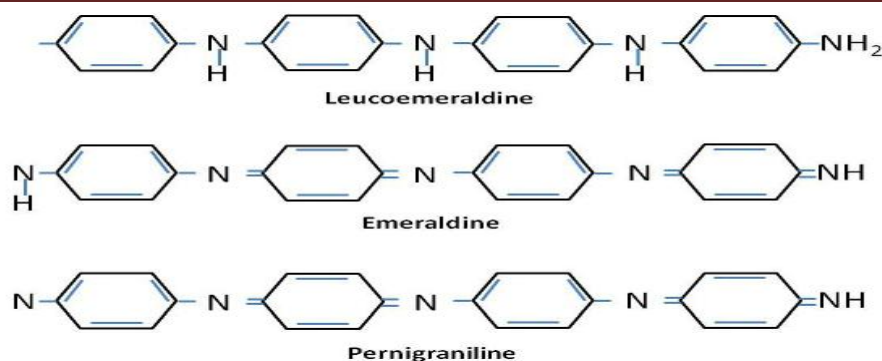


Figure 1: Resonating structure of PANI

The nonacidic approach is used to create conducting polyaniline. This requires a head-to-tail bonding (1, 4, or para) and an even oxidation state with 50% benzenoid and 50% quinoid units. Figure 2 displays the PANI preparation technique.

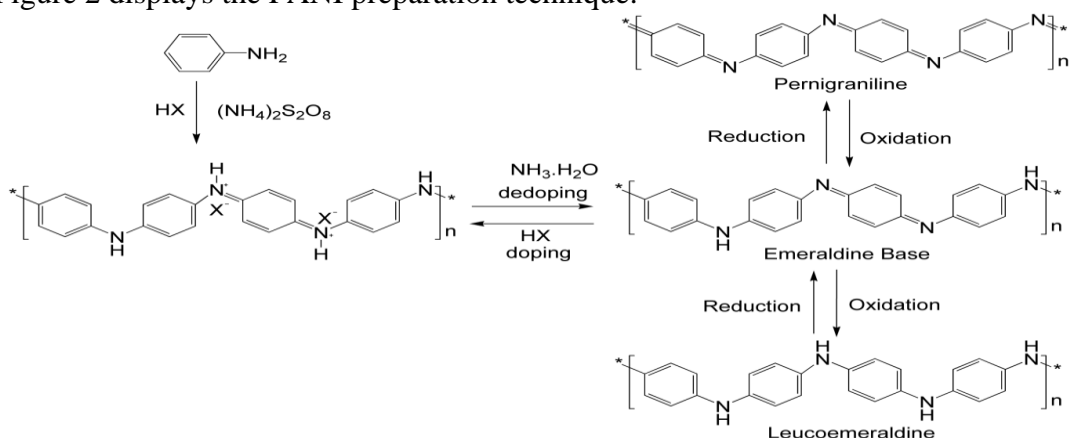


Figure 2: different oxidation states of Polyaniline

As the pH approaches 4, the polyaniline turns blue, and as the pH increases to 7, it changes to blue/violet, indicating that dedoping occurs. Polyaniline has the unusual feature of being able to be processed as an alternating block co-polymer comprising benzenoid and quinoid units. This is basically true for polyaniline in its emeraldine salt form. Nitrogen atom resonance exists between each benzenoid and quinoid. They are referred to as imino and amino groups. These heteroatom moieties are commonly used as acid doping sites, and their ability to be protonated or deprotonated allows the polyaniline to dissolve in strong acids or bases.

Polyaniline is air stable and processable; it may be doped chemically or electrochemically. It may be produced in bulk via chemical oxidation, with an average molecular weight ranging from 10 to 200 kD a.m.u (usually measured using GPC). It can also be produced electrochemically (3 - 10 kD), resulting in more regular material but increasing processing complexity.

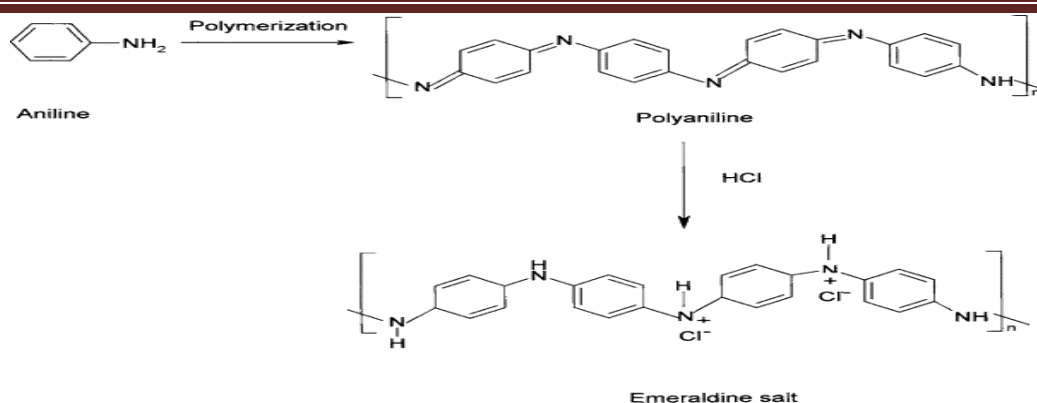


Figure 3: preparation of PANI

Many applications require that the polymer be dedoped or neutralized with ammonia. Because of the high acid content in the polymer, this procedure takes a long time. Furthermore, the discarded wash includes a large amount of acid that cannot be discharged promptly. To solve these shortcomings, polyaniline must be produced in a more ecologically friendly, less corrosive manner that is flexible to future product treatment. There is no prior art for the non-acidic method of generating conducting polyaniline.

Polyaniline is particularly tempting and fascinating among conducting polymers because of its strong conductivity, remarkable redox reversibility, and application stability. The main disadvantages of this polymer are its infusibility and insolubility in common organic solvents. Alternative ways have been explored to improve PANI solubility and processability.

LITERATURE REVIEW

Sharma et al (2010) explained in detail about the solvatochromic behavior of polyaniline and alkyl substituted polyaniline in the behavior of the polymers in different solvents. He also discussed the shifts and the effect of different solvents on conducting polymers through two main transitions namely $\pi - \pi^*$ and $n - \pi^*$. The polyaniline and alkyl substituted polyaniline prepared by chemical polymerization method showed the band gap values in the range 2.38 - 3.16 eV. The various studies evolved a trend in solvatochromic shifts of polyaniline and alkyl substituted polyaniline in different solvents.[8]

Kiran kumari et al (2011) prepared polyaniline doped with binary dopant $ZrOCl_2/AgI$ and investigated the DC conductivity and spectroscopic properties. Doped samples were characterized using various techniques such as I-V characteristics, UV-visible spectroscopy, X-ray diffractometry (XRD), FTIR and photoluminescence (PL). The chemical doping improved the conductivity of PANI. DC conductivity (0.66×10^{-8} to $0.77 \times 10^{-5} \text{ S cm}^{-1}$) was improved through the introduction of binary dopant. The UV-visible study showed that the optical parameters were changed considerably after doping. Both direct and indirect band gap was observed in the doped samples. XRD patterns showed the semi-crystalline nature of doped polyaniline. FTIR study showed the structural modifications in functional groups with doping in PANI. Photoluminescence spectra exhibited emission properties of the samples.[9]

D. Muller et al (2012) Bacterial cellulose/PANI micrographs revealed that the conducting nanofiber material was composed of a continuous conducting substance that was uniformly distributed over the Bacterial cellulose nanofiber surface. The method described in this article shows that $FeCl_3$ is a promising oxidant for preparing Bacterial cellulose/PANI composites with excellent mechanical properties and electrical conductivity values that are higher or comparable

to those found in Bacterial Cellulose/PANI composite materials prepared with ammonium persulphate and protonic acids [10].

Nandapure et al (2013) reported Magnetic and transport properties of conducting polyaniline/nickel oxide nanocomposites in which the conductivity of PANI/NiO nanocomposites decreased with the increase of weight percentage of NiO in PANI. Magnetization of the nanocomposite was increased with the increase of weight percentage NiO. Transport properties of nanocomposites using variable range hopping of charge model were also described.[11]

Z.Yu.H.Li.X.Zhang et al. (2016) studied the most promising applications of PANI composites. The development of electrochemical sensors based on PANI and PANI composites, in particular, has been a popular technique in a variety of electroanalytical and selective detection methods. The detection of glucose was reported using PANI and NiCo₂O₄ nanoparticle composites. In glucose analysis, our non-enzymatic glucose sensor displayed good electrocatalytic performance. The conductive catalytic active sites in NiCo₂O₄ exhibited good sensitivity and a low detection limit.[12]

According to A.Eftekhari et al. (2017), PANI has emerged as one of the most attractive conducting polymer materials in a wide range of applications. Polymer composites have been used to build a variety of devices, including batteries, biofuel cells, thermal devices, and devices that store energy.[13]

A. Shalini et al. (2019) says that the cellulose is the most common resource is present abundantly in most of the organism. Especially in hemp, which is one of the most renewable, sustainable and efficient resources for the protection of bio-based polymeric nanocomposites. Here, cellulose used as a template for surface modified synthesis of polypyrrole grafted cellulose (PPy@C) by in-situ oxidation polymerization with an oxidant ammonium peroxydisulfide (APS) at optimum condition. The structure, morphology, thermal stability, crystallinity and its catalytic properties were analyzed using various instrumental analyses. The objective of this work, PPy@C/GCE exhibit high sensitivity, good stability over the determination of ascorbic acid (AA) in commercial fruits real sample analysis. PPy@C/GCE modified electrode proposed good regression equation, the value of correlation coefficient and low detection limit at wide range of concentration 10–150 μM under optimal DPV condition. The real samples analysis of commercial fruit juices analyzed by PPy@C/GCE and it exhibits good recovery values.[14]

Mahesh Parit et al. (2020) says that the Polypyrrole (PPy) and cellulose nanofiber (CNF) based conducting composite films were synthesized using two new approaches, in-situ polymerization of pyrrole onto cellulose nanopaper (PPy/CNP) and polyvinyl alcohol coated cellulose nanopaper (PPy/PVA-CNP). Significant improvement in the conductivity, tensile strength, water resistance, and electromagnetic shielding effectiveness (SE) was observed for these composite films compared to commonly used in-situ nanofiber (ISF) approach, where PPy is coated on nanofibers prior to film preparation. Maximum improvement in conductivity, SE and tensile strength of PPy/PVA-CNP compared to ISF films was attributed to highly uniform and compact PPy coating and reduced porosity. SE of -23 dB (thickness upto 138.4 μm) and tensile strength of 103.8 MPa for PPy/PVA-CNP films are the highest values found in the literature for PPy and CNF based composite films at a comparable thickness. These new approaches could enable a scalable preparation of flexible conducting composite films with superior physical and electrical properties for EMI shielding applications.[15]

Beygisangchin, et al., (2021) Polyaniline (PANI) is a famous conductive polymer, and it has received tremendous consideration from researchers in the field of nanotechnology for the

improvement of sensors, optoelectronic devices, and photonic devices. PANI is doped easily by different acids and dopants because of its easy synthesis and remarkable environmental stability. This review focuses on different preparation processes of PANI thin film by chemical and physical methods. Several features of PANI thin films, such as their magnetic, redox, and antioxidant, anti-corrosion, and electrical and sensing properties, are discussed in this review. PANI is a highly conductive polymer. Given its unique properties, easy synthesis, low cost, and high environmental stability in various applications such as electronics, drugs, and anti-corrosion materials, it has attracted extensive attention. The most important PANI applications are briefly reviewed at the end of this review.[16]

Zhu Zhang et al. (2022) the cellulose nanofiber (CF) fabric was prepared by electrospinning technology. Then, conductive polyaniline (PANI) was deposited on the CF surface via a facile in-situ polymerization process. The interweaving cellulose/polyaniline nanofiber (CPF) composite constructs a conductive network, and the electrical conductivity can be adjusted by polymerization time. Benefiting from optimal impedance matching, strong conductive loss, as well as interfacial polarization, the CPF possesses excellent EM absorption performance. The minimum reflection loss (RL_{min}) value is -49.24 dB, and the effective absorption bandwidth (RL < -10 dB, fe) reaches 6.90 GHz. Furthermore, the CPF also exhibits outstanding electromagnetic interference (EMI) shielding capability with shielding efficiency (SE) of 34.93 dB in the whole X band.[17]

Abdulwahhab (2022), tried with great effort to shed light on this attractive polymer in terms of its different preparation methods, its distinctive properties, its nanocomposites, and the type of polymerization used for each nanocomposites, as well as its applications in its pure form or with its nanocomposites in the supercapacitor and gas sensor applications. The unique composition of PANI qualifies it for use in electrochemical applications in addition to many other applications whose use depends on its mechanical properties. Based on this, it is necessary to limit the reactions that produce PANI and the cheapest cost, and then limit the current uses in the formation of nanocomposites with metals, their oxides, and/or carbon nanocomposites in order to determine what is missing from them and work on it again to expand its chemistry.[18]

Riaz, U., Singh, N., Rashnas Srambikal, F. et al. (2023) conducting polymeric hydrogels have been extensively investigated due to their remarkable physicochemical as well as physico-mechanical properties. The review deals with the latest advancements in the field of polyaniline (PANI), polypyrrole (PPy)-based hydrogels. The synthesis strategies of PANI- and PPy-based hydrogels have been discussed along with the common characterization techniques employed to study the physical, chemical, mechanical as well as thermal characteristics of these materials. Current literature on biomedical and electrochemical applications has been dealt with in a separate section pertaining to design and fabrication of these materials applicable in the field of drug delivery, biomedical devices, tissue engineering, storage devices, sensors, photocatalysis and adsorption.[19]

METHODOLOGY

The cellulose extracted from hemp used here as a solid support template. 5 g of hemp taken and ground well and sieved (30 mesh). To decolorize the hemp, first it should soaked in 5% of NaOH solution for 2 days and washed thoroughly with ethanol and water several times till the filtrate become colorless for the complete removal of all NaOH and then dried well to remove sticking of fibers and given acid washing for the neutralization of the hemp using dilute

acetic acid. Finally the pH was adjusted to neutral. Then the compound was dried at 105°C for 3 h.



Figure 4: cellulose from HEMP

PREPARATION OF POLYANILINE–CELLULOSE NANOCOMPOSITES

In a typical procedure, 0.5 g cellulose mentioned previously was added to 1.0 mol/L HCl solution dissolving aniline monomer (10:1 by molar ratio). After the cellulose was activated for a period of time, 1.0 mol/L APS aqueous solution was added drop wise to oxidative polymerize aniline. The mixture was stirred for 20 h at ice cold temperature. The reaction product was filtered and washed with doubly distilled water until the filtrate become neutral, and then with anhydrous ethanol till the filtrate become colorless. The composites were dried at 40°C in an air oven for 5 h [20]. For comparison purpose, pure polyaniline without cellulose was also synthesized under the same conditions [21].

The methodology for the synthesis of PANI protected cellulose nanocomposites depicted by the following schematic representation shown in Fig. 5.

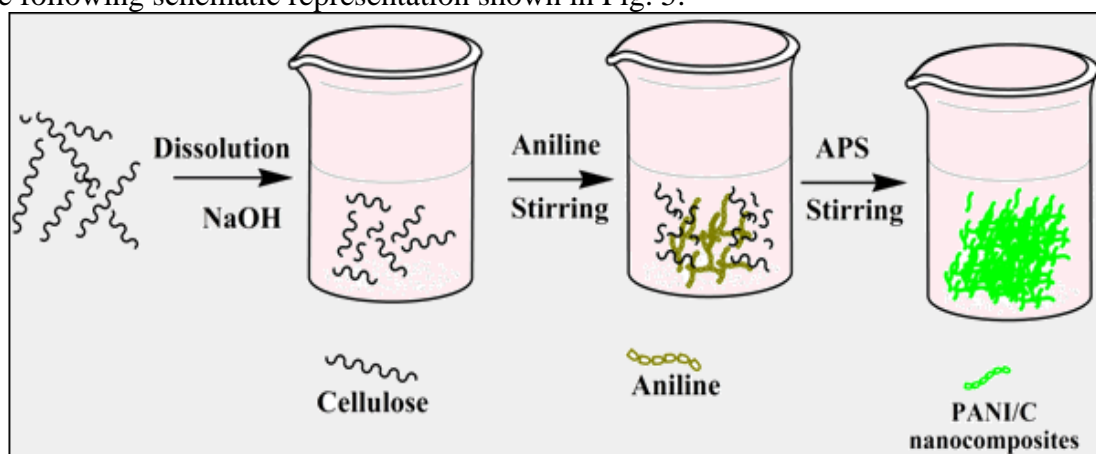


Figure 5: synthesis of PANI-cellulose (hemp) by chemical oxidation polymerization method

RESULT AND DISCUSSION

In a typical process, 0.5 g of the previously described cellulose was added to a 1.0 mol/L HCl solution to dissolve aniline monomer (10:1 molar ratio). After activating the cellulose for a length of time, a 1.0 mol/L APS aqueous solution was applied dropwise to oxidatively

polymerize the aniline. The mixture was swirled for 20 hours at ice-cold temperatures. The reaction product was filtered and rinsed with doubly distilled water until neutral, followed by anhydrous ethanol until colorless. The composites were dried at 40°C in an air oven for 5 hours.

UV-VISIBLE SPECTRA

UV-Visible spectra characterised the PANI and PANI- Cellulose is shown in Figure 6. The absorption peak of PANI suspension is far weaker than that of PANI-cellulose, as a result of PANI was poorly distributed in water and frequently precipitated out as giant aggregation peaks at 340 and 718 nm the primary peak at 340nm is due to $\pi-\pi^*$ transition of the aromatic hydrocarbon rings and therefore the second broad peak at 718nm is assigned to then- π^* transition of the quinoid rings of the PANI [22].

However, the $\pi-\pi^*$ transition and therefore the n- π^* transition bands of PANI supported on cellulose occurred a blue shift to 328 and 568nm, severally. This could be attributed to the interaction arising from the gas bonding between system and PANI. This demonstrates that cellulose is a good biotemplate for supporting conductive PANI.

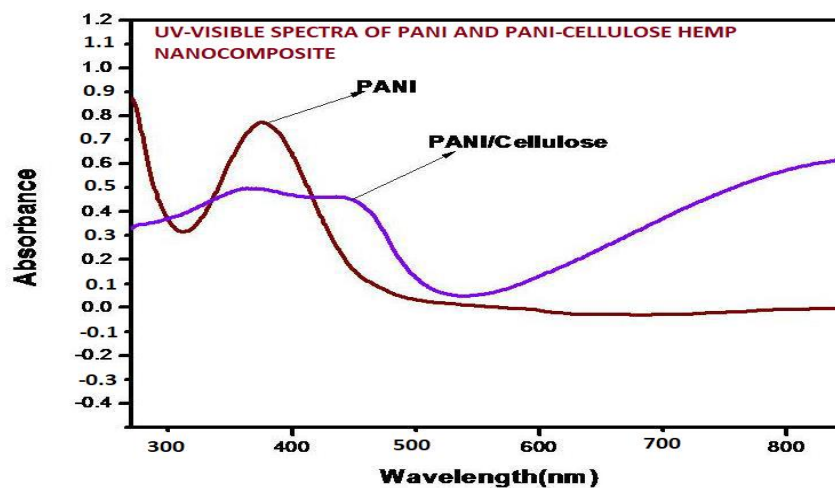


Figure 6: UV-visible spectra of PANI and PANI-Cellulose HEMP nanocomposite

FT-IR SPECTRA

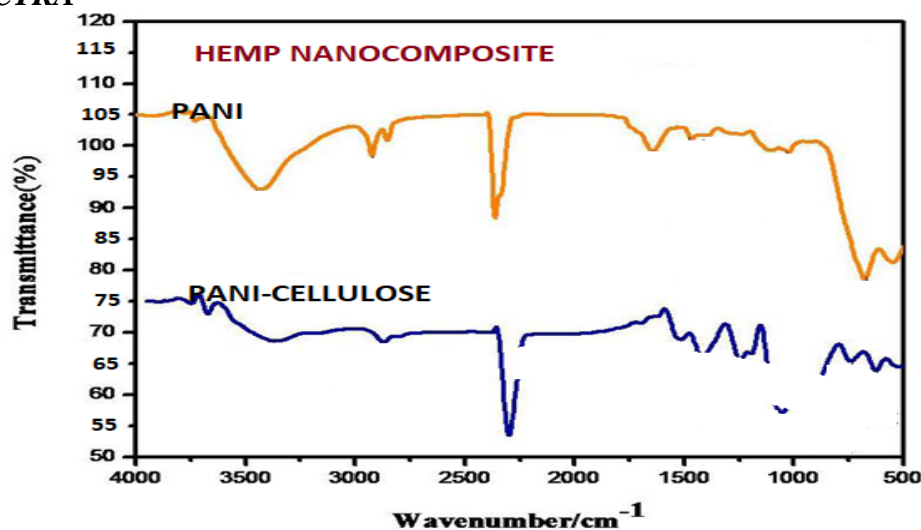


Figure 7: FT-IR spectra of PANI and PANI-cellulose (HEMP) Nanocomposite

Pure PANI has the characteristic peaks of 1523 cm^{-1} and 1405 cm^{-1} , which are ascribed to the C=C stretching deformation of the quinoid and benzene rings, respectively (Yu et al., 2012). The peaks at 1231 and 1131 cm^{-1} denote the C-N and C=N stretching band of an aromatic amine, and 1024 and 685 cm^{-1} are attributed to the in-plane and out-plane bending of C-H, respectively. PANI-Cellulose composite has the characteristic peak of both pure cellulose. When compared to the intensity differences at 3432 cm^{-1} , the composite band appears to be weaker than the stretching vibration of the -OH group in pure viscose. Furthermore, the absorbance peak at 3432 cm^{-1} in the visible spectrum of pure cellulose is blueshifted to 3412 cm^{-1} in the composite spectrum (see Figure 7). These results demonstrate that acids may successfully activate native cellulose.

Table 1: FT-IR spectra of PANI and PANI-cellulose HEMP

S. NO	WAVE No. (cm^{-1})	POLYANILINE	WAVE No. (cm^{-1})	POLYANILINE ENCAPSULATED CELLULOSE(HEMP)
1	3378	N-H stretching	3418	OH stretching band of cellulose
2	2881	C-H stretching	2912	C-O-C pyranose ringskeletal vibration
3	1523	Quinoid stretching vibration	1649	Quinoid stretching vibration
4	1405	Benzenoid stretching vibration	1444	Benzenoid stretching vibration
5	1246	C-N in plane bending	1223	C=N in plane bending
6	1065	C-H in plane bending	1120	C=H in plane bending
7	749	C-N stretching	1025	C-N stretching
8	622	=C-H bond	694	I β Phase of cellulose

X-RAY DIFFRACTION

Figure 8 compares the XRD patterns of pure PANI and the PANI-Cellulose nanocomposite. Pure PANI displays a main characteristic peak at 25.2° due to scattering from the periodicity perpendicular to PANI strands, and one at 20.3° due to the alternating distance between layers of polymer chains [23].

The diffraction peaks of PANI Cellulose composite are comparable to PANI, and there is no noticeable diffraction peak at around (200) of cellulose because the cellulose content in the composite is relatively low and uniformly spread throughout the composite materials [24].

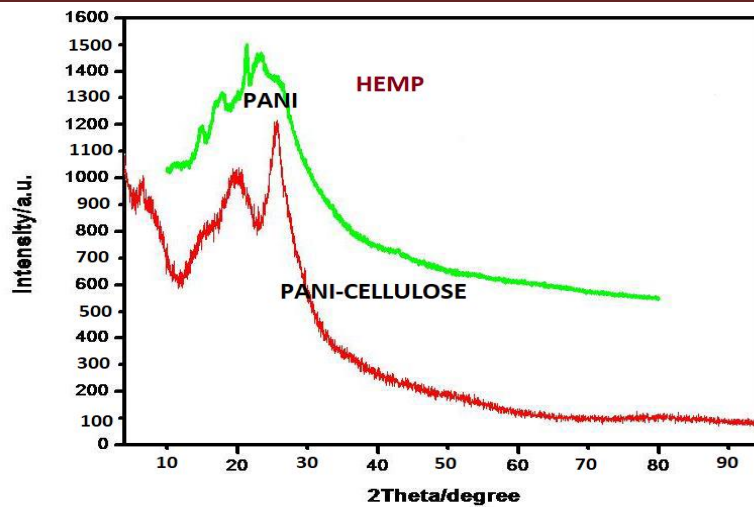


Figure 8 X-ray diffraction of PANI and PANI -cellulose hemp nanocomposite

THERMAL STUDIES (TGA/DTA)

Figure 9 shows the TGA and DTA curves for pure PANI and PANI-Cellulose (Hemp). The first weight losses recorded between 38-137°C and 28-146°C are attributable to moisture losses in the processed components of PANI and composite, respectively. The second weight loss occurred from 132 to 543°C and 136 to 598°C, owing to the degradation of acid dopant and oligomers in the PANI and its composite, respectively. The third stage of deterioration is found at temperatures ranging from 548 to 800°C and 598 to 800°C, probably due to the degradation of PANI and PANI-Cellulose.

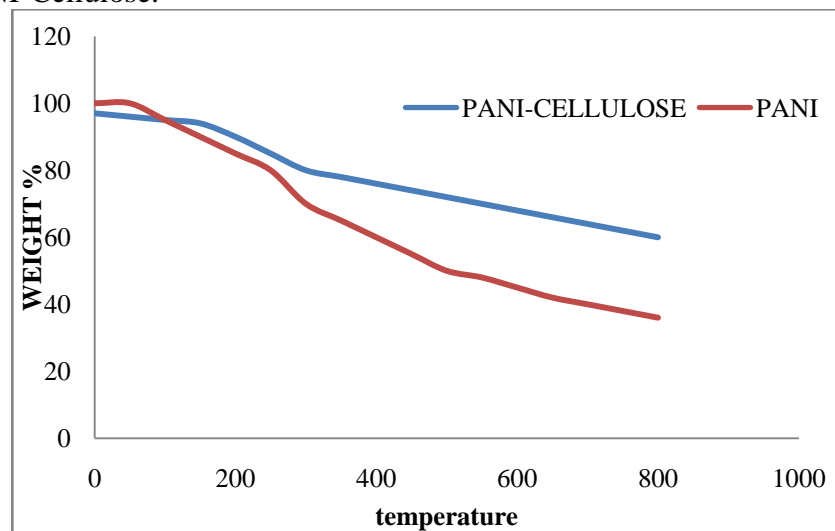


Figure 9: TGA spectra of PANI and PANI -cellulose (hemp) Nanocomposite

DTA OF PANI AND PANI-CELLULOSE

The DTA curves clearly show that moisture causes the earliest losses, followed by the breakdown of dopant HCl in the backbone (Figure 10). In the PANI-Cellulose system, the gradual weight loss observed at 328°C corresponds to the the decay of protons of acid dopant from the PANI backbone, and the maximum weight loss observed at 443°C is due to the degradation of cellulose. The DTA spectrum of PANI-Cellulose (Hemp) showed a maximum

weight loss at 443°C, which is due to cellulose degradation. So it is obvious that the thermal stability of PANI-Cellulose composites is higher than that of pure PANI.

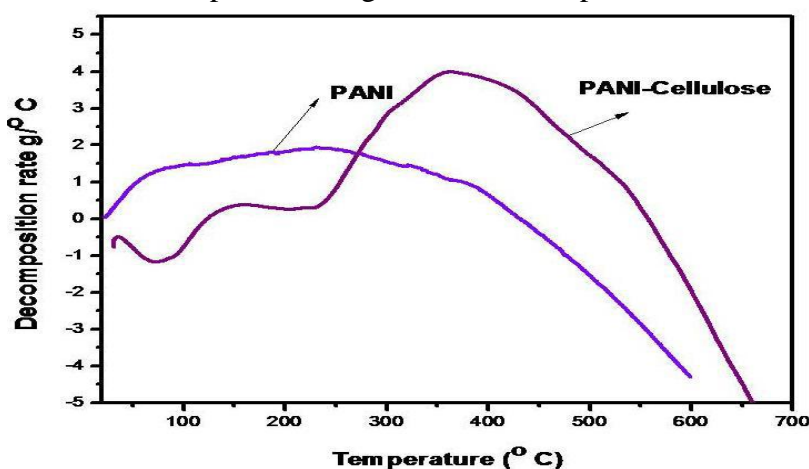


Figure 10: DTA study of PANI and PANI -cellulose hemp nanocomposite

CONCLUSION

The conducting polymers, Polyaniline (PANI) and Polypropylene (PPY), were created using a chemical oxidative polymerization process. Additionally, the presence of ascorbic acid in fruit juices was evaluated electrochemically utilizing PANI/Cellulose and PPY/Cellulose composites. The investigation's findings were reviewed and interpreted appropriately in this study. FT-IR spectrum investigations were also used to characterize the structure of the produced conducting polymers, PANI as well as their encapsulating cellulose composites. The FT-IR spectra reveal that the peaks in PANI were preserved in the cellulose nanocomposite. The crystalline nature of PANI and their composites was investigated using the X-ray diffraction (XRD) technique. The diffractogram of composites made from hemp material reveals a considerable shift in the diffractograms PANI, confirming the interaction of cellulose with PANI conducting polymers. The thermal stability of synthesized PANI and cellulose, as well as the composites made from them, was investigated using thermogravimetric and differential thermal analysis (TGA/DTA). Thermal stability variables were measured for cellulose, PANI, PANI-C (hemp) composites, indicating that cellulose-containing conducting polymer composites have higher thermal stability than pure conducting polymers.

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