Polymer-Based Poly Aniline Cobalt Oxide Nanocomposites Characterization, Synthesized by Sol-Gel method and their photocatalytic Activity

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Abstract:

The polyaniline cobalt oxide nanocomposite was synthesized using in-situ chemical oxidation polymerization and the Sol-Gel method, employing $CoCl_2$ as a precursor. In this process, $K_2S_2O_8$ is used as an oxidant in an acidic medium containing different concentrations of CoO (0.1, 0.2, 0.3, 0.4, and 0.5 moles at room temperature). The synthesized PACoO nanocomposites were characterized by XRD, FTIR, SEM, and TEM. The polymer CoO nanocomposite showed more promising photocatalytic activity than nitrogen-doped TiO_2 for photocatalytic degradation under a UV-visible light source via the photo-degradation of KMnO4 dye. The synthesized nanocomposites demonstrated semiconducting and ferromagnetic characteristics. Based on the results obtained, the nanocomposite that was developed has the potential to be an effective photocatalyst.

Keywords: Polyaniline-Cobalt Oxide nanocomposites, Sol-Gel method, Spectroscopic methods, and Photocatalytic activity.

1. Introduction:

Polymer nanocomposites, which are composed of hybrid organic and inorganic materials, contain at least one filler phase dimension that is less than 100 nm [1]. The four primary methods for producing polymer nanocomposites are exfoliation adsorption, melt intercalation, template synthesis, and in situ polymerization intercalation [2–6]. Recently, a great deal of interest has been in studying the modification of basic polymers, such as polyaniline-cobalt oxide nanocomposites. Polyaniline-cobalt Oxide-based nanocomposites offer excellent sensitivity and a quick reaction time due to their high surface area to volume ratio and decreased target analyte diffusion distance. These properties are crucial for various applications, including photocatalytic activity [7].

Polyaniline-cobalt oxide nanocomposites are the most widely utilized conductive polymers due to their easy manufacture, reversible redox, pH-switching, and sensing capabilities [8]. Like most of these materials, it has poor thermal and physical stability. Since melt methods like extrusion cannot be used in a spinning process, Polyaniline Cobalt Oxide nanocomposites can only be made from spinning solutions [9]. Because of the PACoO's desirable properties and wide range of application possibilities, researchers have taken an interest in it [10–12]. The PACoO nanocomposite has been successfully produced utilizing the Sol-Gel technology to produce polymer-based nanocomposites that may be used as filters, photocatalysts, protective textiles, and pharmaceutical substrates [13]. As far as we are aware, this research is the first to provide helpful information.

2. Experimental:

2.1 Materials:

After two distillations, aniline (S. D. Fine-Chem Ltd., 99.5%) was utilized. Other chemicals, including Cobalt Chloride (99%), potassium persulfate (99%), ethanol (99.9%), sulfuric acid (98.9%), starch, and N- and N-Dimethyl formamide, were purchased from a local business. The ammonia used was of AR quality. De-ionized water was utilized in this experiment.

2.2 Synthesis method of PACoO nanocomposites:

Step-1(Preparation of Cobalt Oxide Nanoparticles)

Drop by drop, ammonia was added to the mixture of 100 ml of starch solution and 0.1 M cobalt chloride while being constantly agitated. After the ammonia was completely mixed, the sample's colour changed from blue to dark grey, and the solution was allowed to settle for the whole night. The obtained sample is filtered using Whatman filter paper. To remove any impurities, get a sample of CoO nanoparticles and wash it in ethanol and deionized water. After that, it was dried at 750°C in hot air furnaces.

Step-2(Preparation of Poly aniline Cobalt Oxide Nanocomposites)

The PACoO matrix is produced by in-situ chemical oxidative polymerization of synthesized CoO nanoparticles. Dissolve 1 ml of aniline in 1 ml of H_2SO_4 solution. One Hr is spent stirring the mixture using a magnetic stirrer. Add the 10 ml of sonicated CoO solution that was in step 1 made. For three hours, heat the reaction mixture while stirring constantly. Drop by drop, add the potassium persulfate and stir constantly for three hours. We can determine that polymerization has occurred and PACoO nanocomposites have been formed when the colour shifts from gray to dark grey. After the solution was kept overnight in a dark place, the sample was collected. Clean the sample with deionized water and ethanol. It was feasible to produce polyaniline and cobalt oxide nanocomposites at various mole

3. RESULT AND DISCUSSIONS:

3.1.Transmission Electron Microscopy (TEM):

300 kV of TEM and an acceleration potential of around 2 A◦ are used, along with point resolution. The condenser, objective, and projection lenses of the microscope column among its many lenses were used. The electrons are typically produced by an electron cannon mounted atop a TEM and then accelerated to a high energy chosen by the user, often between 100 and 300 kV. A substance with a low work function for electron emission is the source of an electron. The dimensions and shapes of the PACoO Nanocomposites were examined using TEM. **Fig.1 and 2** provide images of the different morphologies of the produced PACoO Nanocomposites. The rate of polymerization of the aniline affects the production of spherical nanoparticles from PACoO nanocomposites. To find out how efficiently nanocomposites diffused light, we used the TEM technique. The most direct method for finding the exfoliation states of polyaniline Cobalt oxide nanocomposites is TEM imaging, as shown in **Fig.1** by its ability to image materials at the nanoscale. We found a few small agglomerates mixed in with a homogeneous distribution of polymer nanocomposites in the matrix. Based on the TEM images in **Fig.1 and 2,** we calculate that the uniform size of the polyaniline Cobalt oxide nanocomposites is 200 nm.

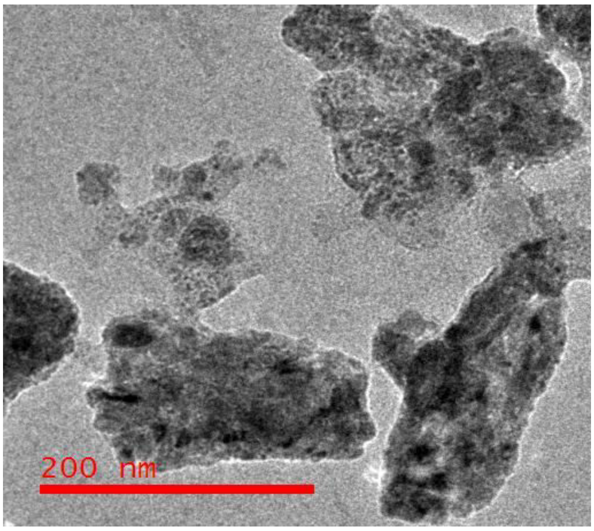

Figure.1

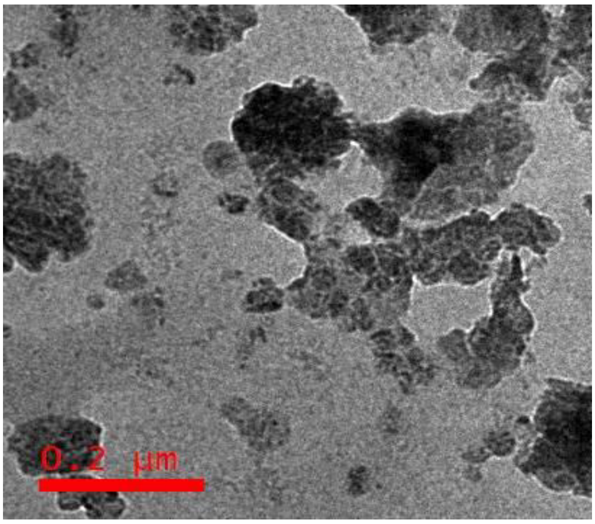

Figure.2

TEM-images of Polymer-based Polyaniline Cobalt Oxide (PACoO) nanocomposites.

3.2.Scanning Electron Microscopy (SEM):

The size and shape of Polyaniline-CoO nanocomposites were investigated by SEM analysis, and the results are shown in **Fig. 3.** Furthermore, as can be shown in the SEM image of the as-synthesized Polyaniline-CoO nanocomposites, the Polyaniline CoO takes on the NaCl structure, including octahedral Co^{2+} and O_2 sites. The most common theoretically simple structure is "Rock Salt structured and crystallizes in the trigonal $\overline{R}3m$ space group" **Fig.4** displays the surface of the PACoO nanocomposites, illustrating their efficient synthesis. The surface morphology of 200 nm polymer-based PACoO nanocomposites was investigated using SEM. SEM micrographs of a polymer-based nanocomposite consisting of polyaniline Cobalt oxide are shown in **Fig. 5.** SEM examinations show that the bright white nanocomposite particles in PACoO nanocomposite are in their purest form. The Cobalt Oxide nanocomposites based on polymers exhibit a uniform size and structure as shown in the Scanning Electron Microscopy picture. **Fig. 6** shows that the samples with medium concentrations had a better size distribution. This is because the medium concentrations of the samples had fewer aggregated particles.

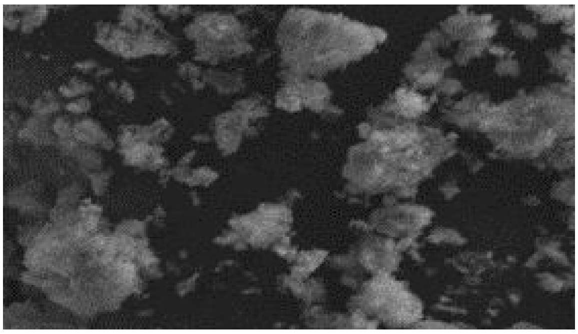

Figure.3

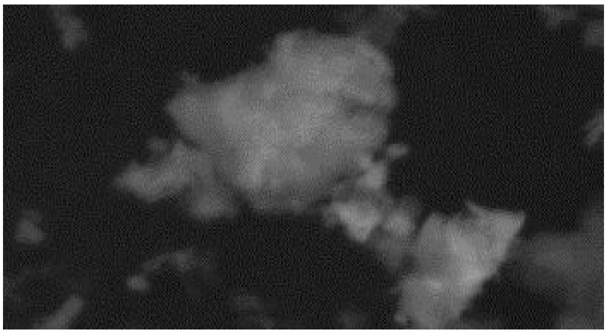

Figure.4

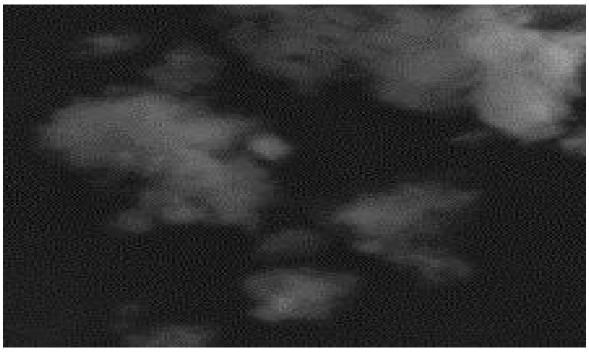

Figure.5

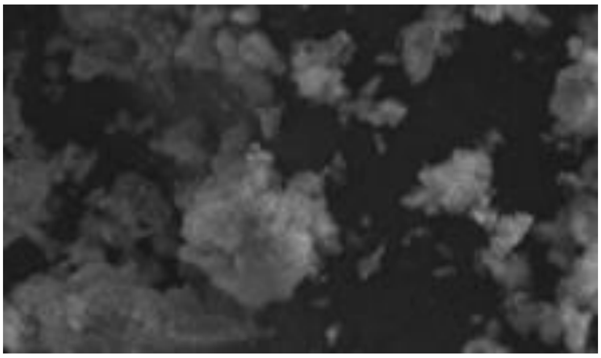

Figure.6

SEM-Images of Polymer-based Polyaniline Cobalt Oxide (PACoO) nanocomposites.

3.3.X-ray Diffraction Studies (XRD):

The X-ray diffraction patterns of PACoO nanocomposites are shown in **Figs. 7 and 8.** The PACoO nanocomposites show two unique broad peaks at 2θ = 21.31° and 26.23° when contrasted to Cobalt oxide, which shows crystalline peaks at 2θ = 37·53° and 43·60° that have been identified as peaks from the single-phase cubic structure of CoO. It was found that the crystallite size of the PACoO nanocomposites was 200 nm. The locations of the two broad peaks are 2θ = 21.31° and 26.23°. The partly crystalline structure of PACoO is shown by the XRD pattern seen in **Fig.7** Interfacial interactions between the XRD patterns of PACoO and the CoO crystallites are shown by the crystalline peaks of PACoO nanocomposites and the crystalline peaks of CoO.

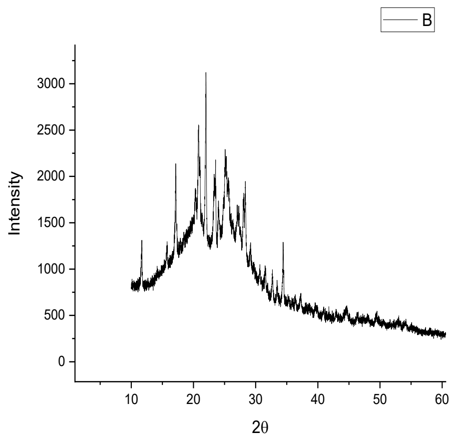

Figure.7

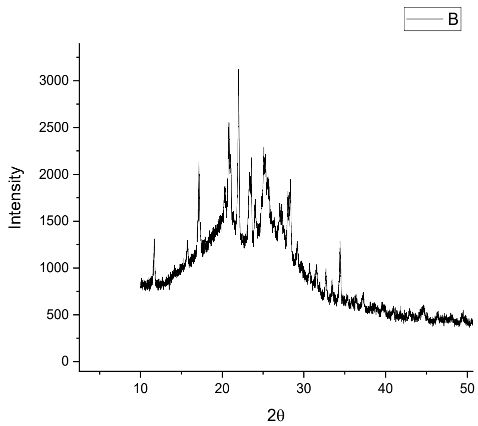

Figure.8

XRD-Spectra of Polymer-based Polyaniline Cobalt Oxide (PACoO) nanocomposites.

3.4. Fourier Transform Infrared Spectroscopy (FTIR):

Table-1 lists the FTIR peaks for the polymer-based polyaniline Cobalt oxide nanocomposites and their likely designations. FTIR peaks at 2975 cm-1, 2923 cm-1, and 2852 cm-1, which matched the published FTIR spectra for PACoO nanocomposites in **Fig. 9**, corroborated the existence of polyaniline in the PACoO nanocomposite recovered following repeated extraction with DMF.

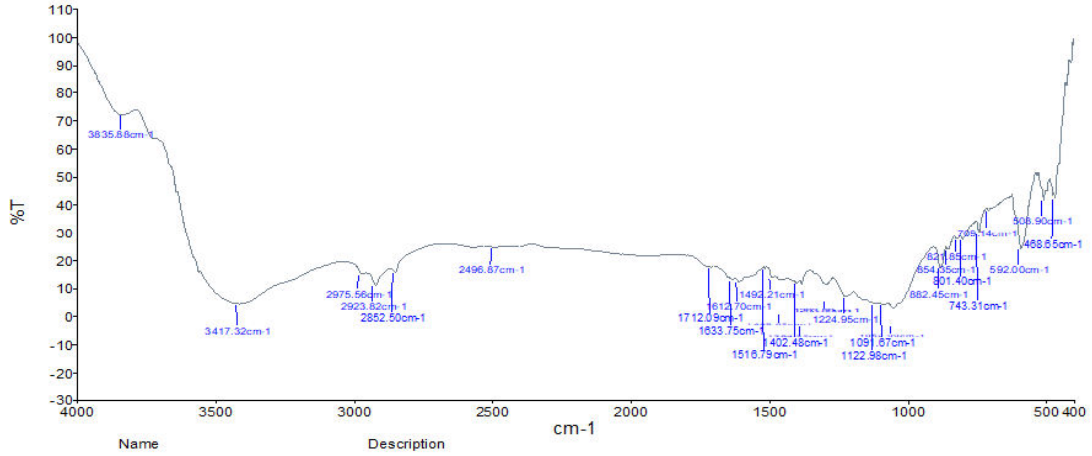

Figure.9

FTIR-Spectra of Polymer-based Polyaniline Cobalt Oxide (PACoO) nanocomposites.

Table-1. FTIR absorption characteristics of PACoO nanocomposites

PAN	PACoO Nanocomposite in cm^{-1}	Assignments
2939	2975	C-H Stretching vibration
2245	2923	C=N Stretching vibration
1454	2852	C-H bending vibration

3.5. The photo-catalytic activity of PACoO nanocomposites:

Potassium permanganate was used in the photo-degradation of a concentration of 0.5 M dye to create PACoO nanocomposites, which demonstrated strong photocatalytic activity, as it is a common chemical that is affordable and available in laboratories. To dissolve the potassium permanganate in the distilled water, it was taken and put on a Petri plate. Samples of PACoO nanocomposite at different concentrations should be prepared, added to a ($KMnO_4$) solution, and exposed to UV light sources with a wavelength between 300 and 800 nm. The relationship between $KMnO_4$ absorbance and UV radiation, broken down by minute length (20, 40, 80, and 100 minutes), is shown in **Fig. 10.** The absorbance of the sample after annealing with PACoO nanocomposites at 550 °C. The polymer nanocomposite catalyst, which has poor crystallinity and an imperfect crystal structure, produced fewer electron-hole pairs as a result of photogeneration. This result demonstrated in **Fig. 10** that the integrity and regularity of the PACoO nanocomposite's structure have critical influences on the photo-degradation rate.

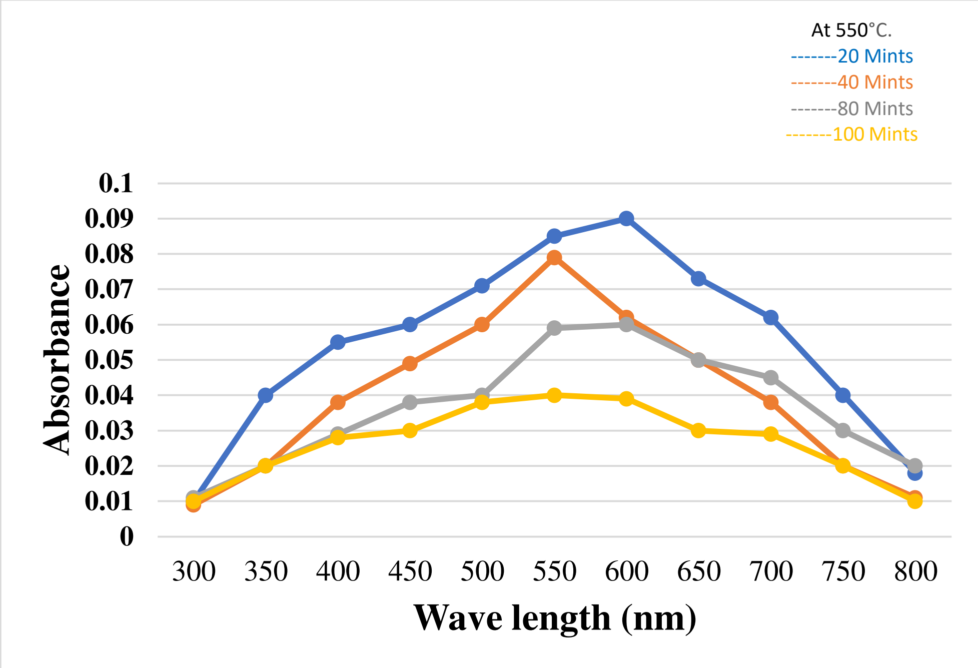

Figure.10

Photocatalytic analysis data of PACoO nanocomposites

Table-2. Experimental values for photocatalytic application of PACoO nanocomposites

S.No	Wavelength(nm)	Absorbance (100 mins)	Absorbance (80 mins)	Absorbance (40mins)	Absorbance (20 mins)
1.	300	0.01	0.011	0.009	0.01
2.	350	0.02	0.02	0.02	0.04
3.	400	0.028	0.029	0.038	0.055
4.	450	0.03	0.038	0.049	0.06
5.	500	0.038	0.04	0.06	0.071
6.	550	0.04	0.059	0.079	0.085
7.	600	0.039	0.06	0.062	0.09
8.	650	0.03	0.05	0.05	0.073
9.	700	0.029	0.045	0.038	0.062
10.	750	0.02	0.03	0.02	0.04

11.	800	0.01	0.02	0.011	0.018

Conclusion:

Oxidatively induced polymerization using the bottom-up Sol-Gel methodology has effectively produced polymer-based PACoO nanocomposites and CoO nanoparticles. We have described the morphology, and topology, size of PACoO nanocomposites 200 nm by TEM, SEM, whereas in the case of the cubic crystalline structure of nanocomposites illustrated by XRD. The polymer PACoO nanocomposite's chemical grafting was validated by FTIR. UV-visible spectral data reveals that at wavelength 600 nm with good absorbance at 20 mins and found to be photocatalytic activity and suitable for semiconducting material.

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