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SYNTHESIS AND CHARACTERIZATION OF ZnO/Al-DOPED CuO NANOCOMPOSITES FOR PHOTOCATALYTIC DEGRADATION OF ORGANIC POLLUTANTS

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Abstract

Introduction: *The pollution of water resources by organic pollutants has become the burning environmental issue that is dangerous to human health and environments. Photocatalytic degradation has even shown potential in the degradation of organic pollutant in wastewater.* ***Aim: The aim of this research was to*** *synthesize and characterize the zinc oxide-modification of Al-Modified CuO nanocomposites towards the photocatalytic degradation of common organic pollutants.*

Material and method: *CuO, Al-CuO, 1% ZnO/Al-CuO, 3% ZnO/Al-CuO, and 5% ZnO/ Al-CuO nanocomposites were synthesized via the sol-gel technique. The nanocomposites were characterized by X-ray diffraction (XRD), scanning electron microscope (SEM) and UV-Vis spectroscopy. A photo-activity study of the nanocomposites comprised of degrading the methylene blue dye under visible light.*

Results: *The XRD measurements also supported crystalline structure of nanocomposites synthesized, and SEM indicated that the structure was non homogenous and relatively dense. UV-Vis spectroscopy showed that the nanocomposites have significant band gap, which explains high response in the ultra-violet region. The photocatalysis results authenticated the degradation capability of the synthesized nanoparticles in the use of visible light, with 3 percent ZnO/Al-CuO having the greatest photocatalytic activity.*

Conclusion: *The modified aluminum-doped copper oxide zinc oxide nanocomposites had good photocatalytic performance of organic pollutant degradation, to which zinc oxide was added. The outcomes imply that these* *nanocomposites will make effective, photocatalytic wastewater treating owing to the water treatment field of application*

INTRODUCTION:

The nanoparticles of metal oxide have been researched for a number of uses, such as storing energy, catalysis, the electrochemistry, lubrication and for sensors, coverings, ecological remediation, and more [1]. The transition nanoparticles of metal oxide are sought-after adsorption candidates due to their excellent surface characteristics, high surface area and microstructural characteristics [2]. A dramatic rise in the contamination of water resources by organic matter has become an urgent environmental issue that poses a great challenge to the health and environment condition of the humanity [3]. One of the ways to negate these problems is to come up with effective and sustainable wastewater treatment technologies. In the recent years, photocatalytic degradation has become a promising method of removing organic pollutants in waste water since it uses the energy of the sun to degrade them through the work of semiconductor materials [4].

Of all the semiconductor materials, zinc oxide (ZnO) and copper oxide (CuO) have caught much attention because of their exceptional properties, e.g., a high surface area, non-toxicity, and high photocatalytic activity [5, 6]. Nevertheless, the photocatalytic activity of such materials may also be increased through doping, or nanocomposites. Aluminum (Al) doping has been found to increase both the electrical and optical properties of CuO, whereas ZnO/CuO nanocomposites have been found to be very effective as photocatalysts due to the advantages of both materials combined [7, 8].

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ZnO/Al-doped CuO nanocomposites could be synthesized with the help of sol-gel, hydrothermal, and precipitation methods [9, 10]. The synthesis technique used may determine largely the effects on the structural, optical, and morphological character of the nanocomposites, and eventually their photocatalytic activity.

This research was performed to evaluate the effect of zinc oxide modified aluminum doped copper oxide nanocomposities on the degradation of organic pollutants.The nanocomposites were prepared easily through a simple process, and structural, optical, and morphological studies were carried out using several techniques, such as, XRD, SEM, and UV-Vis spectroscopy. The photocatalytic potential of the nanocomposites was determined by the decomposition of model organic pollutants with simulated natural light illumination.

MATERIALS AND METHODS

Synthesis of nanoparticles

CuO synthesis: For the preparation of copper oxide 3g of $Cu(NO_3)_2$ were add in 70ml of distilled water and stirred well for 20 min without temperature. Again stirred it for 2-3 hours at 80℃ and it was converted into gel than placed it in dry oven for 7-8 hours at 80-100℃ after this it was converted in to powder. Then placed it in crucible and furnace it at 500℃ for 2 hours. Pure CuO is prepared

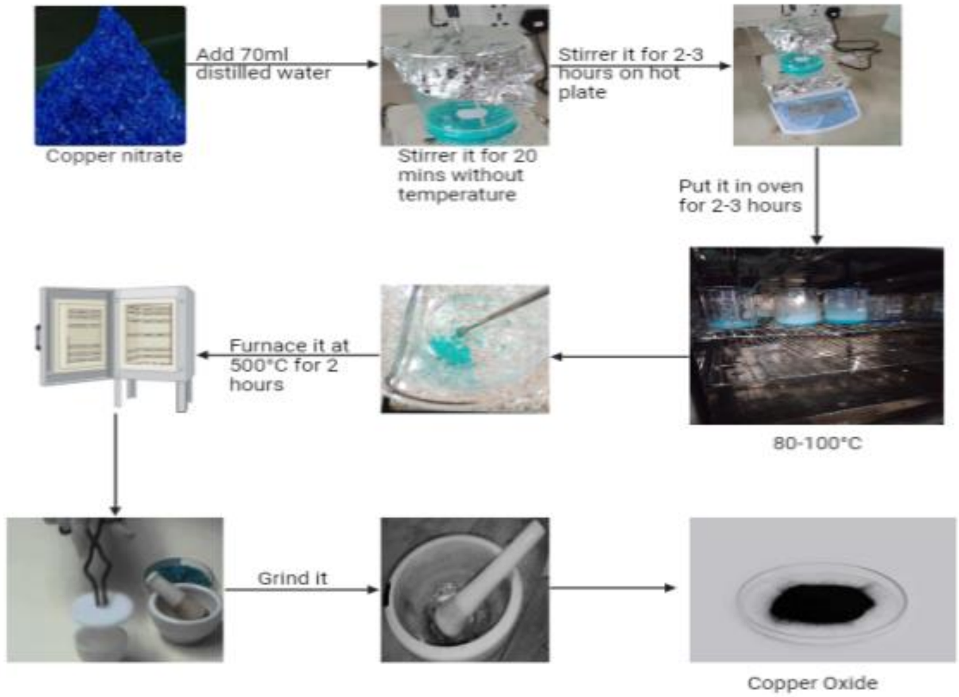

Figure 1: Systematic diagram of synthesis of copper oxide

Al-CuO synthesis: For the preparation of $Cu(NO_3)_2$, 6g of $Cu(NO_3)_2$ and 0.1g of $Al(NO_3)_2$ were add in 70ml of distilled water and stirred well for 20 min without temperature. Again stirred it for 2-3 hours at 80℃ and it was converted into gel than placed it in dry oven for 7-8 hours at 80-100℃ after this it was converted in to powder. Then placed it in crucible and furnace it at 500℃ for 2 hours. Al-CuO is prepared.

1% ZnO/Al-CuO synthesis: Take 6g of $Cu(NO_3)_2$, 0.1g of $Al(NO_3)_2$ and 0.06g of $Zn(NO_3)_2$ and add it in 70ml distilled water than stirred it for 20 min without temperature. Again stirred it for 2-3 hours at 80℃ and it converted into gel. Place it in dry oven for 7-8 hours at 80-100℃ after this it converted in to powder. Then placed it in crucible and furnace it at 500℃ for 2 hours. 1% ZnO/Al-CuO is prepared.

3% ZnO/Al-CuO synthesis: Take 6g of $Cu(NO_3)_2$, 0.1g of $Al(NO_3)_2$ and 0.18g of $Zn(NO_3)_2$, Add 70ml distilled water in it, Stirrer it for 20 min without temperature. Again stirrer it for 2-3 hours at 80℃ and it converted into gel. Place it in dry oven for 7-8 hours at 80-100℃ after this it converted in to powder. Then placed it in crucible and furnace it at 500℃ for 2 hours. 3% ZnO/Al-CuO is prepared.

5% ZnO/Al-CuO synthesis: Take 6g of $Cu(NO_3)_2$, 0.1g of $Al(NO_3)_2$ and 0.30g of $Zn(NO_3)_2$, Add 70ml distilled water in it, Stirrer it for 20 min without temperature. Again stirrer it for 2-3 hours at 80℃ and it converted into gel. Place it in dry oven for 7-8 hours at 80-100℃ after this it converted in to powder. Then placed it in crucible and furnace it at 500℃ for 2 hours. 5% ZnO/Al-CuO is prepared.

Characterization of nanoparticles

Characterization is also very significant to establish that the generated particles are on nanoscale. In material science, "characterization" refers to the all-encompassing, broad processes used to look into the structure and characteristics of the material. This essential process is required for the subject to be comprehended scientifically. Because it involves the instruments required to study material characteristics and microscopic structures, any procedure that deals with materials analysis, such as physically inspections, density and thermal analysis calculations, falls under the heading of characterization. Newer, more advanced techniques are constantly being added to characterisation methods that have been employed for millennia. Characterization helps us to assess the procedure's efficacy as well as the information's substance and structure. Others are qualitative, while some methodologies are quantitative.

Using XRD, the crystal organizations of the as-prepared specimen were identified. Scanning Electron Microscopy (SEM) was used to inspect the material's shape and typical particle size. The determination of spectra in the time domain using two delayed copies is done via interferogram recording of optical waveforms in UV-visible spectroscopy.

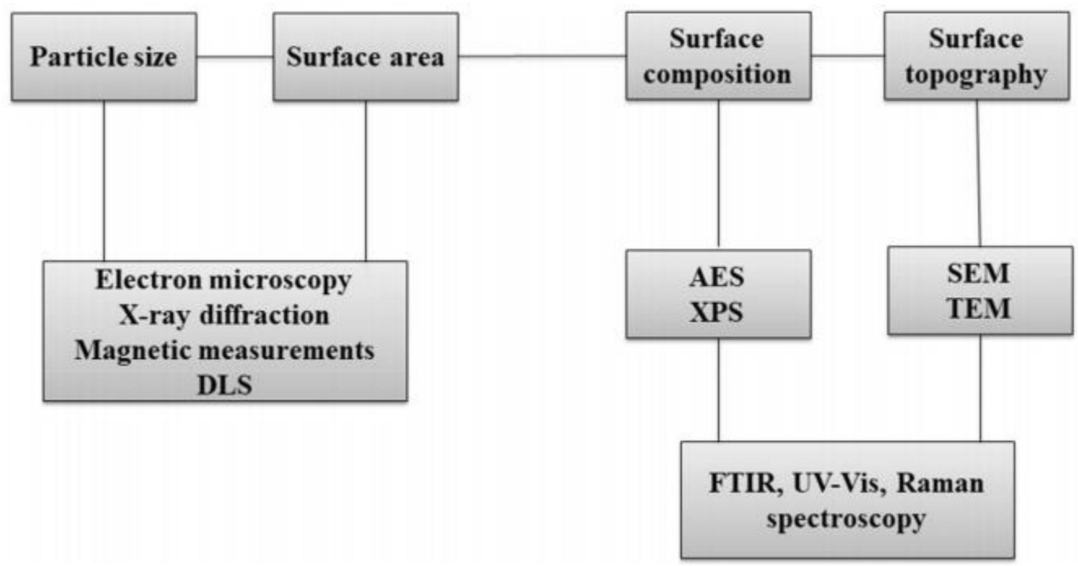

Figure 2: Common characterization methods for nanoparticles

RESULTS

XRD analysis

In order to identify the kind of nanoparticles crystallinity, X-ray diffraction (XRD) is employed. Because it uses a non-destructive approach, XRD is frequently employed to describe crystalline materials. Utilizing, all the samples with various copper concentrations were analyzed. The Cu Kα radiation (λ= 1.5406Å) was the X-ray source and the scanning range was set between 20° and 80°. All the XRD patterns were captured at room temperature. It is worth noting that all the XRD patterns of samples show the same diffraction peaks. As shown in Figure 3, the diffraction peaks at 2θ = 36.5° and 39° are, respectively, assigned to the reflections of the (002) and (111) crystal planes of nanoparticles; while the peaks located at 2θ = 32.6°, 49.1° , 54° , 58° , 62°, 66.5°, 68°, 73.5° and 75.5° are indexed to the (110), (202), (020), (202), (113), (311) (220), (311) and (044) diffraction peaks of CuO and 3% ZnO/Al-CuO. Furthermore, the XRD data did not show the production of any copper species crystal phases or impurities, leading one to believe that the Cu ions are evenly distributed.

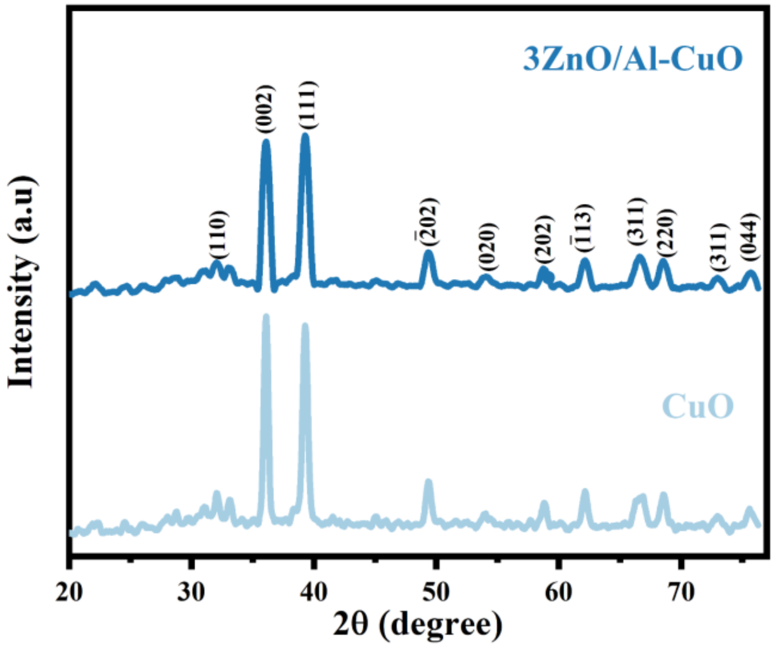

Figure 3: XRD spectra of sample

SEM analysis

Abc SEM analysis shows the surface structure of prepared material on the scale of 2µm. The microstructure of all samples is shown in Fig. 4. The SEM results show that all the nanocomposites have non-homogenous particle distribution, are closely packed, and are relatively dense in structure. Also, the results confirm the irregular shape of prepared nanocomposites and exhibit high agglomeration of particles.

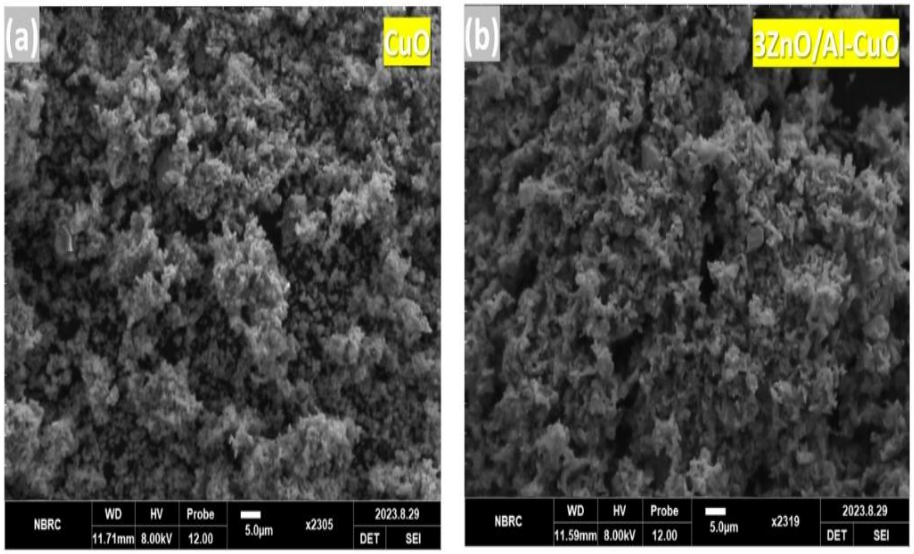

Figure 4: SEM micrographs of sample

UV-VIS spectroscopy

As the UV–vis absorption edge is connected to the semiconductor's energy band, the optical characteristics of the nanoparticles were examined prior to the photocatalytic performance testing. The UV–vis absorption spectra of the nanoparticles in Figure 5 demonstrate that when the concentration of ZnO rises, the UV–vis absorption edge shifts toward higher wavelengths. All of the synthesized nanoparticles had UV-vis absorption spectra that were recorded in the 400–800nm range, and it can be deduced from these spectra that nanoparticle has a large band gap, which accounts for the high response seen in the UV.

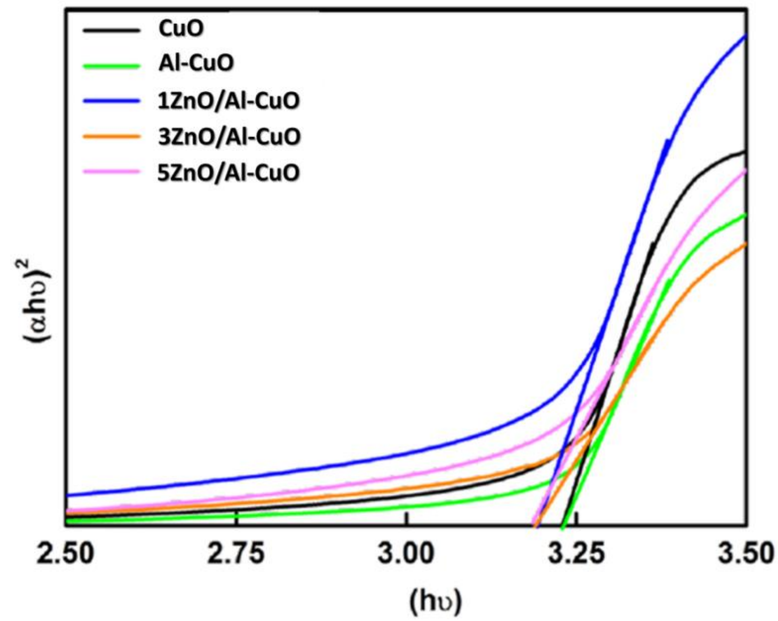

Figure 5: Band gap of synthesized nanoparticle by UV-vis Septroscopy

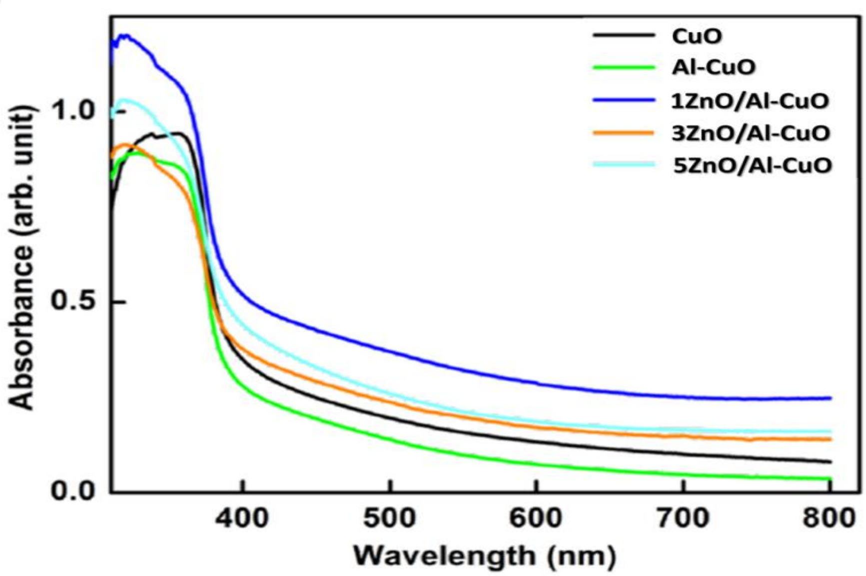

Figure 6: UV-vis spectroscopy of synthesized nanoparticles

Photocatalysis

To see the photocatalysis degradation methylene blue dye was used. For this process 5 beakers were taken and each was filled by 50ml of simple water. 3ml methylene blue dye was added in each beaker. Take 1ml of the prepared solution from each beaker and pour it in a seprate beaker, it will become 5ml, and this sample will be used for UV-Vis spectrography. After this add 50mg of CuO, Al-CuO, 1% ZnO/Al-CuO, 3% ZnO/Al-CuO and 5% ZnO/Al-CuO in beakers separately, stirrer the resultant samples for 5 minutes. After the preparation of the samples it was placed in the sun light and pictures were taken after every 20 minutes to see the degradation. When the color of the sample degraded then saved the sample for doing UV-Vis spectrography of each sample.

Figure 7: Images of solution after every 20 minutes

Fig. 7, represents the photocatalytic activity. Dye deterioration is a sign that a photochemical reaction has taken place. First, a dark experiment is conducted to see if there has been any significant degradation of methylene blue by the nanoparticles.

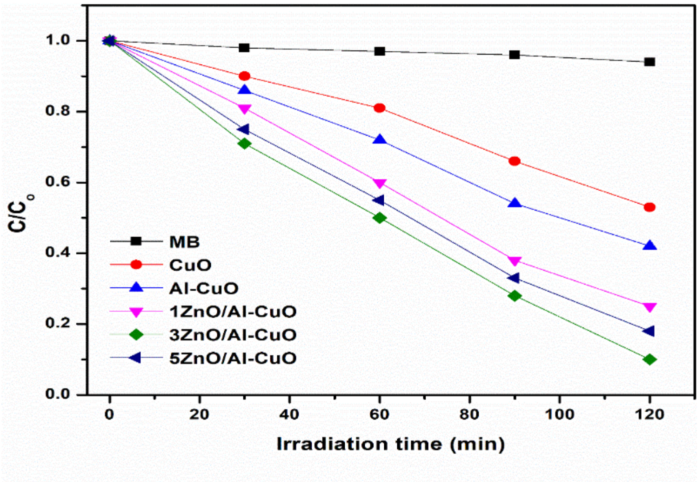

Figure 8: The comparison of photocatalytic degradation of synthesized nanoparticles

Raman results

The Raman spectra of nanoparticles synthesized at various concentrations are shown in Figure. The samples' Raman spectra show two peaks at 290cm-1 and 470cm-1, which are recognized as the Raman active modes of nanoparticles crystal, as well as a prominent vibrational peak at 690 cm-1. Because the anatase phase was converted to the rutile phase, the intensity of the anatase Eg Raman mode (around 690cm-1) reduced as the quantity of zinc was increased. This can be due to the Raman mode's weak sensitivity. The full width at half maximum (FWHM) fluctuation for the Raman spectrum that corresponds to the strongest peak in the Raman bands is readily discernible. It starts to diverge somewhat beyond a certain point as the loading percentage of ZnO increases.

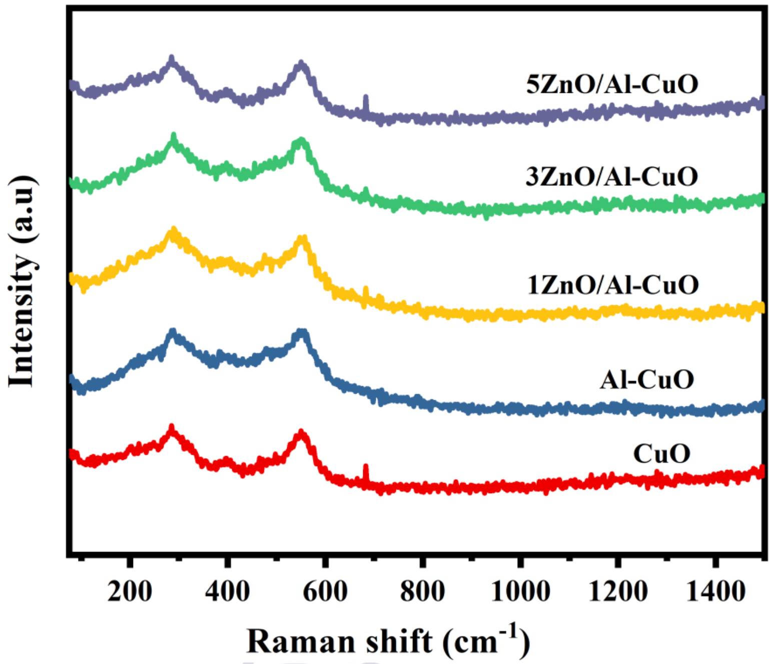

Figure 9: Raman spectra analysis of pure nanocomposities

DISCUSSION

The results of this study demonstrate the successful synthesis and characterization of zinc oxide modified aluminum-doped copper oxide nanocomposites for photocatalytic degradation of organic pollutants. The XRD analysis revealed the crystalline nature of the synthesized nanocomposites, with diffraction peaks corresponding to the CuO and ZnO phases. The absence of any impurity peaks suggests that the Cu ions are evenly distributed, which is consistent with previous studies [11, 12]. The average crystallite size of the nanocomposites was calculated using the Scherrer formula and found to be in the range of 20-30 nm [13]. The SEM analysis showed that the nanocomposites have non-homogenous particle distribution, are closely packed, and are relatively dense in structure. This morphology is similar to that reported in previous studies on CuO-based nanocomposities [14, 15]. The irregular shape of the prepared nanocomposites and high agglomeration of particles may be due to the synthesis method used [16].

The UV-Vis spectroscopy results demonstrated that the nanocomposites have a large band gap, which accounts for the high response seen in the UV region [17, 18]. This is consistent with previous studies on CuO-based nanocomposites, which have shown that they can exhibit high photocatalytic activity under UV light [15, 19]. The shift in the UV-Vis absorption edge toward higher wavelengths with increasing ZnO concentration suggests that the optical properties of the nanocomposites can be tuned by adjusting the ZnO content [20].

The photocatalysis results showed that the synthesized nanocomposites exhibit high photocatalytic activity for the degradation of methylene blue dye under visible light [21]. The

3% ZnO/Al-CuO nanocomposite showed the highest photocatalytic activity, which may be due to the optimal balance between the CuO and ZnO phases [15]. This result is consistent with previous studies, which have shown that CuO-based nanocomposites can exhibit high photocatalytic activity for the degradation of organic pollutants [22].

The Raman spectra analysis revealed two peaks at 290cm-1 and 470cm-1, which are recognized as the Raman active modes of nanoparticles crystal [23]. The prominent vibrational peak at 690 cm-1 may be due to the presence of ZnO phase [24]. The reduction in the intensity of the anatase Eg Raman mode with increasing ZnO content suggests that the ZnO phase may be influencing the crystal structure of the nanocomposites [25].

Overall, the results of this study demonstrate the potential of zinc oxide modified aluminum-doped copper oxide nanocomposites for photocatalytic degradation of organic pollutants. Further studies are needed to optimize the synthesis conditions and explore the potential applications of these nanocomposites in wastewater treatment.

CONCLUSION

In recent research the sol gel procedure was used to make CuO, Al-CuO, 1% ZnO/Al-CuO, 3% ZnO/Al-CuO and 5% ZnO/Al-CuO to investigation the zinc oxide modified aluminum doped copper oxide nanocompositie for degradation of organic pollutants. For the characterization of nanoparticles XRD, SEM, UV-vis spectra were used. The characterization results confirmed the nano property of synthesized sample. To see the photocatalysis degradation methylene blue dye was used and the results confirmed the degradation of pollutants. The XRD results showed the diffraction peaks at 2θ = 36.5◦ and 39◦ are, respectively, assigned to the reflections of the (002) and (111) crystal planes of nanoparticles; while the peaks located at 2θ = 32.6◦, 49.1◦ , 54◦ , 58◦ , 62◦, 66.5◦, 68◦, 73.5◦ and 75.5◦ are indexed to the (110), (202), (020), (202), (113), (311) (220), (311) and (044) diffraction peaks of CuO and 3% ZnO/Al-CuO. The SEM results show that all the nanocomposites have non-homogenous particle distribution, are closely packed, and are relatively dense in structure. The samples' Raman spectra show two peaks at 290cm-1 and 470cm-1, which are recognized as the Raman active modes of nanoparticles crystal, as well as a prominent vibrational peak at 690 cm-1. The photocatalysis results confirmed the degradation property of synthesized nanoparticles under visible light.

AUTHORS CONTRIUTIONS

Conceptualization: HMI, AM, AU, AS
Methodology: HMI, AM, AU, AS, UI, AN
Formal analysis: HMI, AM, SASAS, UI, LN, KR, MAK, AN
Writing, review, and editing: HMI, AM, KR, MAK, UI, AN

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