ABSTRACT: Mixed metal oxide nanocomposites (NCs) comprising Cu–Sr (CS), Sr–Cd (SC), and Cd–Cu (CC) were fabricated via a sol–gel method. Structural investigations of fabricated samples were carried out via X-ray diffraction (XRD), scanning electron microscopy (SEM), diffuse reflectance spectroscopy (DRS), and X-ray photoelectron spectroscopy (XPS). The Maxwell–Wagner model, attributing to poor conducting layers around the conducting grains, was indicated to be followed by all of the NCs while investigating the dielectric properties. The Space-charge polarization and hoping mechanism contributed to low AC conductivity at lower frequencies and high AC conductivity at higher frequencies. The as-synthesized NCs effectively degraded two toxic water contaminants, such as crystal violet (CV) and Congo red (CR). Furthermore, the NCs were also evaluated for humidity sensing measurements. All of the NCs indicated efficient response/recovery time with better stability. The extensive investigation suggested the synthesized NCs, well suited for various optical and microelectronic applications.

1. INTRODUCTION

The versatile nature of metal oxide NCs allows them to be utilized in various industrial applications such as optoelectronics, catalysis, medicinal uses, and humidity sensing. Microelectronic industries are usually involving the lead-based dielectric materials, which are generally hindered by high working temperatures, nonstoichiometric ratios, and environmental contaminants. The best remedy for all of these issues is to replace them with lead-free materials such as metal oxides. Metal oxide composites having a narrow band gap can be obtained with extensive physicochemical properties when they are mixed with larger band gap nanomaterials. Until now, Cu, Sr, Al, Zn, Ni, and Ni mixed with various elements such as Li, Na, Ti, and Al have been successfully used for various applications. The nanostructures such as CuO, ZnO, SnO2, SrO, NiO, CdO, and TiO2 exhibit novel and advanced optical and electronic properties due to the quantum confinement effect. Despite all outstanding properties of metal oxides, the carrier recombination rate and resistivity limit their utilization on the industrial level. The suitable alternative is to modify the microstructure to obtain a uniform particle distribution, surface decoration with nanoparticles, and mixing with suitable elements. The separation distance, junction formation, lifetime of charge carriers, and improved degradation ability of metal oxides are the major concerns to decompose toxic organic effluents from dye and paper industries. Transition metal oxides are frequently used in heterogeneous catalysis, solar cells, gas sensors, photovoltaic devices, and lithium-ion batteries based on their chemical stability and electrical properties.

In a similar way, various science and technology industries such as textile, food, and gas purification are mainly dependent on sensing materials and devices. Various organic materials, polymers, ceramics, and metal oxides have been fabricated as promising humidity sensing devices, but to operate them at unstable and varying environments is still a challenging task for

Figure 1. Schematic diagram for the synthesis of NCs.

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researchers. Once again, the NCs comprising metal oxides are devised and analyzed with improved sensing and recovery behavior, low hysteresis, and high stability operable in a wide range of humid environment.\textsuperscript{16,17} The particular advantageous aspects and versatile microstructural properties of metal oxides such as low cost, intermediate working temperature, porosity, enlarged surface area, uniform size distribution, and operability over a wide humidity range make them preferable.\textsuperscript{18} The

Figure 2. (a) XRD patterns; (b–e) UV spectra and DRS Tauc plots; and (f–o) low- and high-resolution XPS spectrum for CSNC, SCNC, and CCNC.
frequent response of transition metal oxides to the surface phenomenon, such as adsorption, is considered favorable for humidity sensing applications.19,20

The present work describes a cost-effective and mild synthesis of three NCs such as CSNC, SCNC, and CCNC. Structural characterization is carried out via X-ray diffraction (XRD), diffuse reflectance spectroscopy (DRS), X-ray photo-electron spectroscopy (XPS), and scanning electron microscopy (SEM). The dielectric properties of the fabricated NCs are discussed with respect to frequency and temperature. The NCs are catalytically evaluated against two hazardous dyes, such as crystal violet (CV) and Congo red (CR). The last part is investigating the resistive humidity sensing properties of these NCs at room temperature with a humidity range of 11−97% RH.

2. EXPERIMENTAL SECTION

2.1. Synthesis Route and Characterization Techniques. All of the chemicals utilized in this research work were purchased from Sigma-Aldrich. Raw materials such as Sr(NO3)2, Cu(NO3)2, and Cd(NO3)2 were mixed together in equimolar ratios for the preparation of CSNC, SCNC, and CCNC. Solutions were prepared with triple distilled water at constant stirring. The pH was adjusted by the dropwise addition of 0.1 M NaOH. At a pH value of 9, a precipitate appeared, which was further stirred at 300 rpm, at a temperature of 80 °C for 24 h. The precipitate was then filtered and washed with distilled water and ethanol. It was then dried in an oven and ground. To enhance crystallinity, the powder was further calcined at 400 °C for 2 h. Calcined powder was stored and utilized for subsequent analysis. The schematic diagram for the synthesis of the NCs is described in Figure 1. The calcined NCs were thoroughly investigated via XRD (Bruker-D8), DRS (Wincom-UV2800), XPS (UK-250Xi), and Scanning Electron Microscopy (QUAN- TA-450).

2.2. Electrical Properties and Humidity Sensing Measurements. Pelletization (with 2−3 mm of thickness and 10 mm of diameter) for the calcined powder was carried out at a pressure of 100 MPa. The two faces of pellet were silver coated and then was red at a temperature of 600 °C for 2 h. Frequency and temperature-dependent electrical measurements were carried out by utilizing an LCR meter (4284-Agilent). A digital hygrometer (RH-101) and an LCR meter were utilized to indicate humidity sensing and variations in resistance at different humidity levels. The water vapor was injected into a humidity chamber at a steady rate to change the humidity level from 11 to 97% RH.

2.3. Photocatalytic Activity. The photocatalytic abilities of the synthesized NCs were evaluated by color changes of the organic pollutants, i.e., CV and CR. Stock solutions of 0.05 mM for CV and CR dyes and 1 g/L each for SCNC, SCNC, and CCNC were used during catalytic experiment. A decrease in the concentration for CV and CR dyes was measured at regular intervals via a 105 UV−vis spectrophotometer in a wavelength ranging from 200 to 800 nm. To specify the adsorption phenomenon, the reaction was initially carried out in the dark for half an hour and then was shifted to solar light. Equations 1−3 represent the complete degradation phenomenon during photocatalytic experiments20

\[
A_i/A_o = \exp(-kt)
\]

\[
D(\%) = \frac{(A_o - A_i/A_o) \times 100}{A_o}
\]

3. RESULTS AND DISCUSSION

3.1. XRD, DRS, and XPS. XRD patterns for the synthesized NCs are shown in Figure 2a. For CSNC, the monoclinic phase of CuO was revealed by the peaks at 2θ values of 38.9, 48.7, 58.3, 68.9, 79.7, and 89.8°, indicating the planes as (200), (−202), (202), (−221), (023), and (−131). Peaks at 29.6, 50.0, 59.3, 60.0, and 74.0° representing the planes (102), (321), (132), (610), and (215) may be attributed to SrO as matched with (JCPDF No. 72-0629) and (JCPDF No. 06-0520), respectively. Similarly, for SCNC, the peaks at 33.4, 38.4, 55.3, 65.9, and 69.3° with the corresponding planes as (111), (200), (220), (311), and (222) were due to the FCC phase of CdO (JCPDF No. 75-0592, and JCPDF No. 75-0594), while the remaining peaks represented SrO. For CCNC, the peaks at 33.0, 38.8, 55.5, 65.7, and 69.4° with crystal planes as (111), (200), (220), (311), and (222) revealed CdO (JCPDF No. 65-2908), while the other peaks at 38.9, 48.7, 58.3, 68.9, 79.7, and 89.8° with the corresponding planes as (200), (−202), (202), (122), (320), and (131) were for CuO (JCPDF No. 65-6186). The grain size calculated from XRD measurements was ~79.97, 60.36, and 82.62 nm for CSNC, SCNC, and CCNCs, respectively.

\[
d = \frac{KL}{\beta \cos \theta}
\]

UV spectra and DRS Tauc plots were measured to obtain the values for band gap energies. Decreased band gap energies of ~1.76 eV (CSNC), 1.66 eV (SCNC), and 1.85 eV (CCNC) were due to various metals mixed together, as shown in Figure 2b−e.21 XPS was utilized for the oxidation states of constituent elements within the synthesized composites.22 Peaks of Cu, Sr, Cd, and O indicated the presence of these elements within the prepared NCs, as shown in Figure 2c. The detailed spectra collected for each element, as shown in Figure 2g−o, are summarized in Table 1.

3.2. SEM Micrographs and EDX Spectra. SEM images suggested small size dense and agglomerated structures for all of the synthesized NCs with the average particle size ranging from ~50 to 100 nm. The particle microstructure and shape are shown in Figure 3a−c. EDX spectra showed that the NCs are composed of Cu, Sr, Cd, and O elements, as shown in the inset of Figure 3d−f.
3.3. Dielectric Properties. Electrical and electronic devices are operated on the basis of dielectric characteristics of materials. NC powders were pressed into pellets with 2 × 10 mm of thickness × diameter, using a hydraulic press to investigate the dielectric properties. The pellets were silver coated, then sintered at 600 °C for 2 h and were measured at different frequencies and temperatures, as shown in Figure 4a–d. The dielectric constant decreased with the increase in the frequency.
for all of the NCs, attributed to the Maxwell–Wagner polarization model, as depicted in Figure 4a.22 The conductive grains insulated by grain boundaries of dielectric materials generated polarization and internal barrier and hence a high dielectric constant.23 However, with the increase in the frequency, the lagging behavior of the hoping mechanism with the field caused a decrease in polarization and dielectric constant at higher frequencies.24 CSNC exhibited higher dielectric polarization (≈8611) compared to SCNC (≈6517) and CCNC (≈3890). The space charge at grain boundaries and polarization effect at the electrodes caused a higher dielectric constant at a temperature of ≥50°C, as shown in Figure 4b. Polarization and
dielectric constant were also enhanced due to the additional thermal energy released to the bound charges.25 However, with the increase in temperature beyond a certain limit, the dielectric constant was decreased. This decrease could be attributed to freezing and stopping of the excessive charges as well as the complete spin ordering of the constituent’s elements.26 The frequency-dependent dielectric loss for the synthesized NCs is shown in Figure 4c. The dielectric loss initially decreased with the increase in the frequency, but with further increase, it entered into a constant region. A high dielectric loss was measured for CSNC and SCNC due to the loss of heat energy with increased charge density. 27 The increased dielectric constants and decreased dielectric losses make these fabricated NCs suitable for high-frequency device applications. In low-frequency regions, AC conductivities of the NCs remained constant, but an increase was recorded at higher frequencies, as depicted in Figure 4d.28 Maximum electrical conductance calculated for SCNC, CCNC, and CSNC were 0.2761, 0.1596, and 0.0924 S/cm, respectively. Low conductivity at lower frequencies and greater conductivity at higher frequencies are due to the space-charge polarization and hoping mechanism.29

### 3.4. Catalytic Performance.
Catalytic experiments for CSNC, SCNC, and CCNC were carried out on the basis of change in color for toxic organic pollutants such as CV and CR. Stock solutions of 0.05 mM for CV and CR dyes and 1 g/L each for SCNC, SCNC, and CCNC were utilized during the experiment. Solar light of 100 mW/cm² was used for catalytic reactions. The decrease in the concentration and hence the decrease in the intensity of UV–vis absorbance bands for CV and CR dyes measured at regular intervals represented the degradation phenomenon within 80 min, as shown in Figure 5a–h. Figure Si—n represents the linear pseudo-first-order kinetics between the concentration decrease and irradiation time for CV and CR dyes.30 Numerical data about the detailed degradation process is shown in Table 2.

#### 3.4.1. Photocatalytic Mechanism and Intermediate Analysis.
During the degradation reaction, electrons received solar energy and shifted from its stable band to excited band, as shown in Figure 6. Ethanol and H₂O₂ scavengers prevented the recombination process of the carriers, hence exciting more electrons. These electrons reacted with O₂ and generated O₂⁺, H⁺, and OH⁻. These OH⁻ radicals were split into hydrogen peroxides (H₂O₂), combined with O₂⁺, and *OH was generated. In a similar way, H₂O₂ was also decomposed into *OH. The whole degradation reaction can be explained mathematically as follows

\[
h\nu (\text{energy}) + \text{NCs} \rightarrow \text{NCs}(e^- + h^+) \quad (5)
\]
\[
e^- + O_2 \rightarrow O_2^- \quad (6)
\]
\[
O_2^- + H^+ \rightarrow *OH \quad (7)
\]
\[
2*OH \rightarrow O_2 + H_2O \quad (8)
\]
\[
H_2O_2 + O_2^- \rightarrow *OH + OH^- + O_2 \quad (9)
\]
\[
H_2O_2 \rightarrow *OH \quad (10)
\]

These results revealed that when the water molecule reacted with holes, H⁺ and H₂O₂ were produced. Dyes were degraded with the generation of two *OH ions and splitting of the H₂O₂ molecule.31–33 Mathematically we can express the reactions as follows

\[
[\text{NC}]h^+ + (\text{CV/CR}) + H_2O \rightarrow H^+ + [\text{NC}](*OH) - (\text{CV/CR}) \quad (11)
\]
\[
[\text{NC}]h + (\text{CV/CR}) + *OH \rightarrow [\text{NC}](*OH) - (\text{CV/CR}) \quad (12)
\]
\[
2H_2O + 2h^+ \rightarrow 2H^+ + (H_2O_2) \quad (13)
\]
\[
(H_2O_2) \rightarrow (*OH)_2 \quad (14)
\]
\[
[\text{NC}]e^- + (\text{CV/CR}) + O_2 \rightarrow [\text{NC}]O_2^- - (\text{CV/CR}) \quad (15)
\]
3.5. Humidity Sensing Measurements. The silver-coated calcined pellets were investigated to determine the electrical resistance at different humidity levels ranging between 11 and 97% via an LCR meter and digital hygrometer.

$$\text{O}_2^- + (\cdot \text{OH})_2 + \text{H}^+ \rightarrow \text{O}_2 + (\text{H}_2\text{O}_2), \text{H}_2\text{O}_2$$

$$\rightarrow (\cdot \text{OH})_2$$

$$[\text{NCs}]\text{O}_2^- \text{or HO}^+ + (\text{CV}/\text{CR}) \rightarrow \text{H}_2\text{O} + \text{CO}_2$$

(16)  

(17)  

The silver-coated calcined pellets were investigated to determine the electrical resistance at different humidity levels ranging between 11 and 97% via an LCR meter and digital hygrometer.

3.5.1. Resistive Response of Humidity. The resistive response of humidity is specified by a change in resistance with respect to various humidity levels, which can be calculated by utilizing the following mathematical equation:

$$S_H = \frac{\Delta R_o}{\Delta R_{RH}}$$

(18)

In this equation, $\Delta R_o$ is representing air at the lowest RH level, and $\Delta R_{RH}$ is utilized for the resistance at higher RH%. The decrease in resistance with an increased humidity level (attributed to the surface adsorption of water) was revealed by all of the NCs, as shown in Figure 8a. The enlarged surface area, intergranular pores, and existence of defects and vacancies due to sintering and nonstoichiometric composition caused a high-sensing response (i.e., greater sensitivity) of the materials. Maximum sensitivity was recorded for CCNC as shown in Figure 8b. The redox properties and oxygen vacancies can also facilitate the adsorption of the water molecules in pores existing in the materials.

3.5.2. Response and Recovery Behavior. The active and efficient response and recovery time are the important and critical parameters for the performance evaluation of any humidity sensor. To evaluate the accurate response and recovery behavior of the sensor, two chambers, one with a low humidity level of ~11% and another with a higher level of...
~97%, were maintained. The characteristic response and recovery curves for the fabricated nanocomposites are shown in Figure 8c. The response and recovery time calculated were (68 s, 70 s), (40 s, 69 s), and (59 s, 70 s) for CSNC, SCNC, and CCNC, respectively. The greater value of the heat of adsorption compared to the heat of desorption contributed to the small difference of response and recovery time. Furthermore, a higher value of bonding energy between the adsorbed water and material surface delayed the desorption phenomenon.32−34

3.5.3. Hysteresis. Humidity hysteresis involves the adsorption curve at a humidity level ranging from 11 to 97% and the desorption curve in the backward retracing of the system from 97 to 11%. The exothermic and endothermic processes during the humidity sensing technique have different speeds; therefore, the adsorption process was found to be a bit faster than the desorption process, as shown in Figure 8d−f.35

3.5.4. Stability. Stability is another important characteristic of the practical and commercial utilization of any humidity sensing device.36 Therefore, a stability test was carried out for all of the NCs for a time span of 60 days at 11, 34, 75, and 95% RH levels, as shown in Figure 8g−i. All of the NCs were found to be stable for the tested time period.

4. CONCLUSIONS

Mixed metal oxides such as CSNC, SCNC, and CCNC were successfully synthesized through the sol−gel method. Advanced characterization techniques such as XRD, SEM, DRS, and XPS were carried out to investigate the crystallinity, microstructure, band gap energy, and co-existence of the constituent elements. The synthesized NCs were evaluated for dielectric, catalytic, and humidity sensing applications. The NCs performed well and effectively in all of the investigated areas such as conductivity, degradation, and humidity response/recovery. This research indicated that the NCs well suited for practical applications in various fields of optics and microelectronics.

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Notes

The authors declare no competing financial interest.

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