




# Highly sensitive humidity sensor based on cadmium selenide quantum dots-polymer composites: synthesis, characterization, and effect of UV/ozone treatment

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## ABSTRACT

This work describes the rational design of thin film-based cadmium selenide quantum dots (CdSe) mixed with conductive polyvinylidene fluoride (PVDF), inducing PVDF-CdSe composite for potential resistive humidity-sensing applications. The effect of UV/ozone treatment on surface hydrophilicity and sensing properties was investigated. AFM has been performed to examine the prepared films' texture, distribution over the surface, and size. Overall, the hydrophilicity of the developed films increases with UV radiation exposure time, leading to enhanced water vapor absorption without deforming the film surface. The sensor's sensitivity is improved with increasing UV radiation exposure. The electrical response of the PVDF-CdSe humidity sensors after 30 min of UV/ozone treatment reveals that at higher humidity levels (i.e., > 80% RH), the sensors exhibit an irregular response. However, at 20 min, treatment increases sensitivity and a linear change in impedance response concerning humidity level change compared to other samples. The hysteresis response was divided into two regions: the lower region, between 30 and 60% RH—where the maximum hysteresis loss was calculated to be 3%. While the higher area between 60 and 90% RH, where the maximum estimated hysteresis loss of the PVDF-CdSe sensor is around 14%, the UV/ozone treatment of the PVDF-CdSe nanocomposite film was found to enhance the sensing film's hydrophilicity without deforming the surface of the as-prepared PVDF-CdSe as well as the UV-treated films validates a potential for novel humidity sensors.

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## 1 Introduction

Humidity sensors widely monitor and detect humidity levels in industrial manufacturing, packing processes, and quality control. Most related research focuses on developing humidity sensors with fast response, recovery time, and high stability [1]. Various humidity-sensing materials have been explored in order to achieve these, along with cost-effective production [2], which include nano- and mesoporous materials [3], clays [4, 5], polymer composite [6], and quantum dots [7].

Quantum dots (QDs) have received much attention due to their excellent optical and electronic properties. The hybrid mixture of quantum dots and polymer matrix is highly recommended to improve the electrical and mechanical properties of the polymeric sensing film. The small size and large surface area of QDs may enhance the sensitivity of the nanocomposite film [8]. QDs with low toxicity and water solubility, photostability, high fluorescence intensity, and narrow emission spectra are helpful for bio-compatible humidity-sensing applications. Cadmium selenide (CdSe) quantum dots [9] have gained potential interest due to their profound electronic properties [10], optoelectronic properties [11], low toxicity, and water solubility [12]. CdSe QD has also been used with other materials, particularly in sensing applications; thus, once mixed with other polymers, the composite materials provide better stability and may be used to prepare a flexible sensing platform [13]. Among the polymers, polyvinylidene fluoride (PVDF) is a piezoelectric polymeric material that is flexible [14], thermally stable, chemically resistive, and dielectric constant [15]. Accordingly, the PVDF polymer has been extensively investigated as a sensing polymeric film. Incorporating QDs within the polymeric film has been previously used in sensors preparation.

Two methods are commonly used: acetone treatment of the polymeric surface and plasma treatment of the character, which were used to enhance the surface wettability of polymeric film. In both ways, surface roughness increases, which adsorbs more water, resulting in a decrease in the stability of the humidity sensor. The modification of the nanocomposite film surface without affecting surface

roughness has not been reported yet. To the best of our knowledge, no reports on developing PVDF-CdSe-based materials using UV/ozone treatment for humidity sensors have been published.

This work outlines the preparation of PVDF-CdSe-based sensing material—2 wt% only of CdSe was incorporated into the PVDF matrix. We then studied the impact of UV/ozone treatment on the hydrophilicity of the PVDF-CdSe nanocomposite film by subjecting the latter to UV/ozone light for up to 30 min. The UV/ozone-treated PVDF-CdSe nanocomposite film improves the sensing film's hydrophilicity to evaluate the composites' physico-chemical changes.

## 2 Experimental section

### 2.1 Materials and chemicals

PVDF, oleic acid, octadecene, cadmium, and selenide was purchased from Sigma-Aldrich and ITO/glass electrode (S161) from Ossila UK.

### 2.2 Synthesis and purification of CdSe

CdSe were typically prepared following the procedure previously described in this work [16]. Briefly, CdO (0.51 g) was dissolved in 1-octadecene solution (80 mL) and oleic acid (6 mL) at 150 °C under N<sub>2</sub> by vigorous stirring till the formation of a clear solution. Then Cd<sup>2+</sup> oleates in 1-octadecene solution were heated to 180 °C under N<sub>2</sub>. Meanwhile, the Se powder was dissolved in trioctylphosphine (TOP) solution using an ultrasonic bath and under N<sub>2</sub> gas. The clear Se-TOP solution was swiftly injected into the cation precursor solution (Cd-oleates) at 180 °C. The obtained material was purified twice using the solvent mixture technique (mixture of Acetone and IPA) and finally dissolved hexane or toluene.

### 2.3 Preparation of humidity sensor-based PVDF-CdSe

Primary, the ITO electrodes were cleaned using acetone, then distilled water under sonication, distilled water, and nitrogen gas for 10 min each. A nanocomposite solution using 2 wt% of CdSe was prepared by dissolving g in PVDF. The spin-coating

technique deposited the PVDF-CdSe solution on the interdigitated ITO/glass substrates.

### 3 Characterization

#### 3.1 Electrical characterization

The PVDF-CdSe impedance sensor was placed in a sealed chamber to investigate the electrical response of the humidity sensor, as shown in Fig. 1. A humidifier was connected through a control valve to the sealed chamber to raise the humidity level. The Drierite-based desiccant was attached to the humidity chamber through an inlet valve to decrease the humidity level. The PVDF-CdSe-based humidity sensor impedance response was measured by an MS5308 LCR meter. The humidity meter RS-6109 was used as a reference humidity to observe the humidity level. Fig. 1 shows the complete fabrication process and the experimental setup employed to analyze the humidity-sensing properties of the PVDF-CdSe-based humidity sensor.

#### 3.2 Morphological, chemical, and surface analyses

Morphology changes of the coated films were tested using an atomic force microscopy (AFM, MFP-3D, Asylum Research, the USA) machine. The X-ray photoelectron spectra (XPS) analysis was carried out to check the surface and chemical composition of the pristine and prepared films using ESCALAB 250

instrument fitted with a monochromatic Al K $\alpha$  X-ray source. UV-Vis absorption spectra were determined using a UV-Vis spectrophotometer (Gold Spectrum-lab 54, Shanghai Lengguang Technology Co. Ltd., and China). The fluorescence spectra were monitored for three samples of CDs prepared at different periods using a HORIBA SPEX Fluorolog-3-11 spectrofluorometer. No significant shifts in the fluorescence peaks were noted from batch to batch.

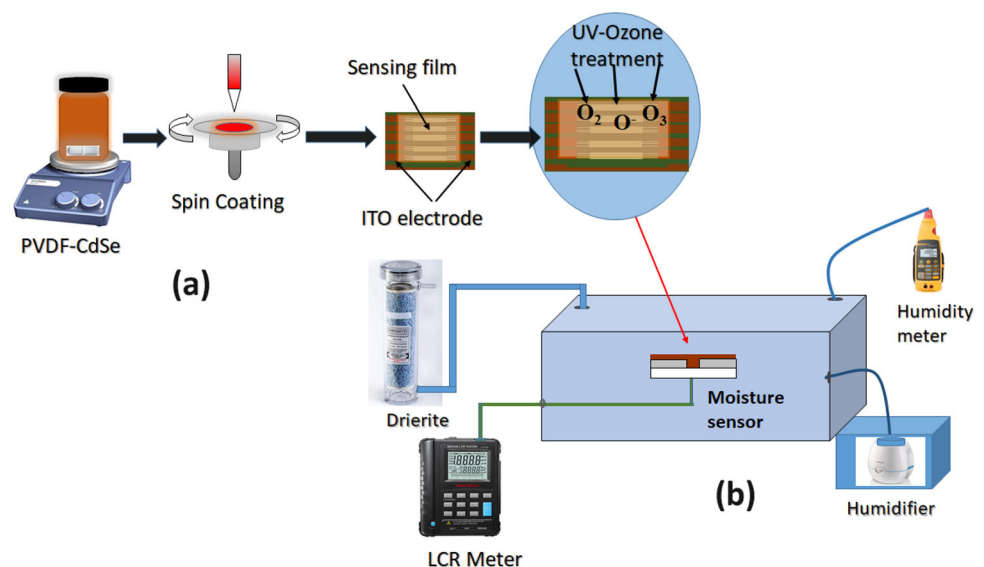
## 4 Results and discussion

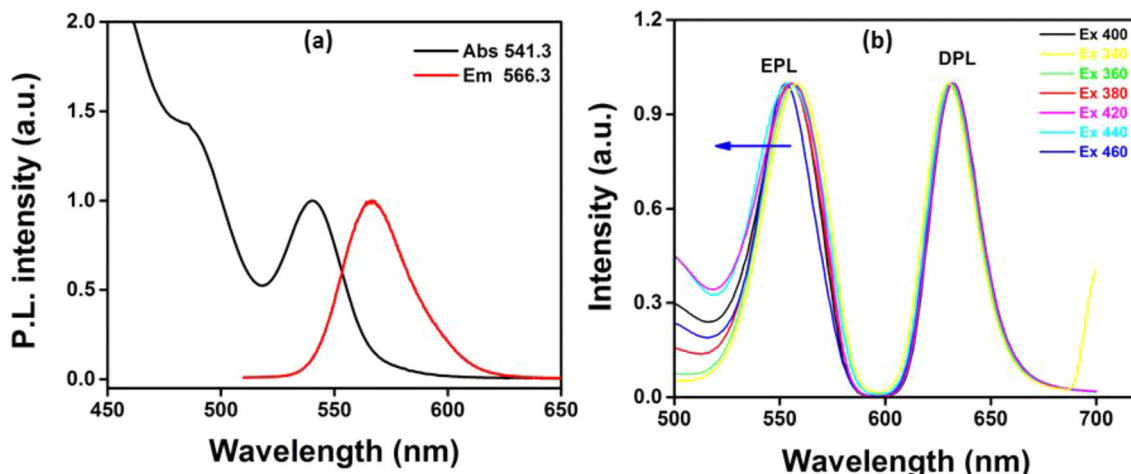
### 4.1 Optical properties of CdSe

Figure 2a presents the steady-state absorption (black line) and emission (red line) spectra for as-prepared oleic acid-capped CdSe QDs in toluene. From the absorption spectrum, the first excitonic peaks are well defined for the CdSe QDs [17]. While the steady-state emission spectrum for the QDs on the low-energy side of the absorption ones with  $\sim 33$ -nm FWHM. Narrow emission bands indicate the less size distribution of our QDs; as shown in Fig. 2b, the exciton PL peak underwent a blue shift and a narrowing with subsequent broadening.

The surface roughness of the PVDF-CdSe humidity sensors was studied using the AFM analysis, and the results for UV exposure time 0–30 min are shown in Fig. 3a–d, respectively. The root mean square roughness (RMS) of the composite film without UV/Ozone treatment is 23.633 nm; after applying the

**Fig. 1** a Graphical illustration of PVDF-CdSe-based humidity sensor prepared by the spin-coating method. b A humidity sensor setup was used during this work

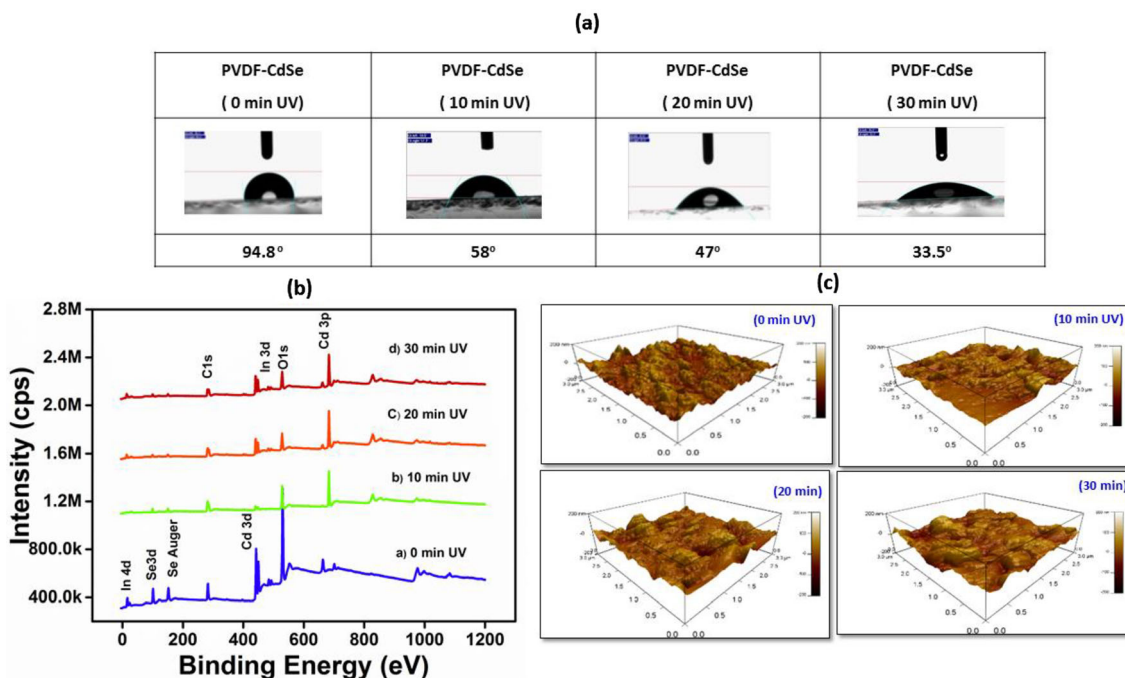




**Fig. 2** **a** Steady-state absorption (black line) and emission (red line) spectra for as-prepared oleic acid-capped CdSe QDs in toluene; **b** excitation-dependent emission spectra of CdSe in toluene (Color figure online)

UV/Ozone treatment on PVDF-CdSe composite film, the RMS roughness is 26.06 nm, 32.34 nm, and 33.90 nm with 10 min, 20-min UV, and 30-min UV exposure time, respectively. The AFM analysis of the PVDF-CdSe composite film with an increase in UV exposure time from 0 to 30 min shows no significant impact on the roughness of the sensing film, and the composite film remains homogenous.

Figure 3a shows the contact angle tested on PVDF-CdSe films exposed to different (0, 10, 20, and 30 mins) UV radiation. The contact angle of PVDF-CdSe composite film with no exposure to UV/Ozone radiation has a contact angle of 94.8°, which shows the hydrophobic nature of the composite film. By increasing the UV/Ozone radiation exposure time to 10 min, the contact angle of the film decreases to 58°, and this decrease in contact angle indicates the



**Fig. 3** **a** Contact angle measurements, **b** XPS general survey, and **c** AFM image of CdSe-PVDF nanocomposite films (2 wt%) after (0, 10, 20, and 30 min of UV exposure)



increase in hydrophilicity of the composite film. As the UV exposure time increased to 20 min, the contact angle decreased to 47°, and the composite film became more hydrophilic than the previously mentioned samples. After 30 min of UV exposure, the film becomes super hydrophilic, and the contact angle decreases to 33.5°. Overall, the hydrophilicity increases with UV radiation exposure time, which leads to enhanced water vapor absorption on the surface of the prepared film. The sensor’s sensitivity is improved with increasing UV radiation exposure time. Hysteresis significantly decreased for PVDF-CdSe films at higher UV exposure time. X-ray photoelectron spectroscopy was used to study the chemical and structural composition and the chemical states of the prepared PVDF-CdSe films under different UV exposure times, as shown in Fig. 3. The hybrid nanostructure displays C1s, O 1s, Cd 3d, and Se 3d signals in the survey spectrum. The general survey indicates the first peak at 280.6 eV in the C1s range, Cd 3d is confirmed by its characteristic peaks at 411.6 eV (Cd 3d3/2) and 404.90 eV (Cd 3d5/2), and the chemical structure and surface composition of the as-prepared CdSe are similar to the previously reported CdSe based materials [17].

Further, the increasing UV exposure time fills in the voids in the prepared films. Upon UV exposure, the PVDF-CdSe solution probably reacts covalently to the ITO surface, thus reducing surface asperities. Hence, a combination of morphology and composition could explain the decreasing contact angle and the disappearance of hysteresis. Indeed, voids in the surface induce hydrophobic surface character, whereas smooth surface containing CdSe interacts favorably with water, hence the improved wettability of all examined surfaces. Concomitantly, contact angle hysteresis is reduced. Moreover, the UV effect was correlated to the calculated sensitivity of resistive humidity sensors 0-min UV, 10-min UV, 20-min UV, and 30-min UV are 0.001352, 0.002, 0.0107, and 0.0105, respectively (Table 1). [18, 19]

#### 4.2 Electrical response

Figure 4 shows that without UV/Ozone treatment, the PVDF-CdSe composite film-based humidity sensor has low sensitivity to changes in humidity level. Figure 4 depicts the impedance response of the PVDF-CdSe composite humidity sensor to changes in humidity level during different UV/Ozone exposure

**Table 1** Regression analysis of the PVDF-CdSe (20-min UV)-based resistive humidity sensor

Regression statistics	
Multiple R	0.8870482
R Square	0.786854509
Adjusted R square	0.744225411
Standard error	1.964910576
Observations	7

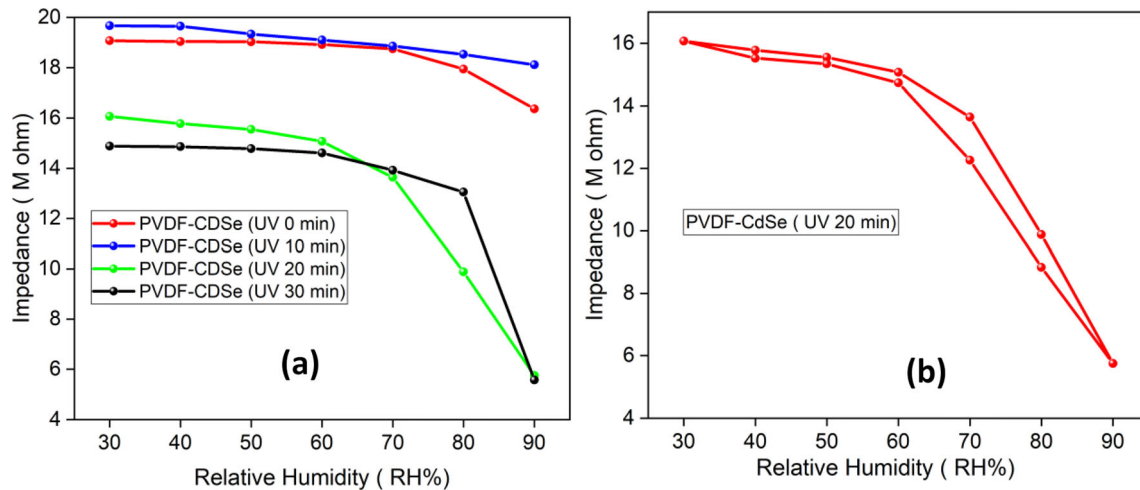
times. By increasing the UV/Ozone exposure time to 10 min, the surface of the composite film becomes hydrophilic, and the sensitivity of the PVDF-CdSe-based humidity sensor increases. By raising the exposure time to 20 min, the hydrophilicity of the hybrid movie rises even more, improving the impedance response of the PVDF-CdSe composite humidity sensor as a function of the change in humidity level.

When the UV/Ozone exposure time is increased to 30 min, the composite film becomes super hydrophilic (as shown in Fig. 4a). The electrical response of a PVDF-CdSe combined film sensor after 30 min of UV/Ozone exposure shows that at higher humidity levels above 80% RH, the sensor offers an irregular response, which may be caused by a large amount of moisture collected on the film surface, causing the conductance to increase at higher humidity levels. The PVDF-CdSe (20 min UV) exhibits the most heightened sensitivity and a linear change in impedance response compared to other samples. The sensitivity of the resistive humidity sensors at different UV/Ozone exposure times is calculated using Eq. (1)

$$S = \left( \frac{1}{RT_i} \right) \left( \frac{RT_i - RT_f}{RH_{final} - RH_{initial}} \right). \tag{1}$$

RT<sub>i</sub> and RT<sub>f</sub> are the initial and final impedance values at initial and final humidity levels. The calculated sensitivity of resistive humidity sensors 0-min UV, 10-min UV, 20-min UV, and 30-min UV are 0.001352, 0.002, 0.0107, and 0.0105, respectively.

The hysteresis response of the PVDF-CdSe (20-min UV) impedance sensors is shown in Fig. 4b; hysteresis is an essential parameter for analyzing the sensor’s performance and stability. The hysteresis response of the PVDF-CdSe (20-min UV) composite film exhibits a clear division into two regions. In the lower relative humidity (RH) range, specifically from



**Fig. 4** **a** Impedance vs. Relative Humidity level for fabricated PVDF-CdSe (UV 0 min, 10 min, 20 min, 30 min) nanocomposite. **b** Hysteresis response of PVDF-CdSe (UV 20 min)

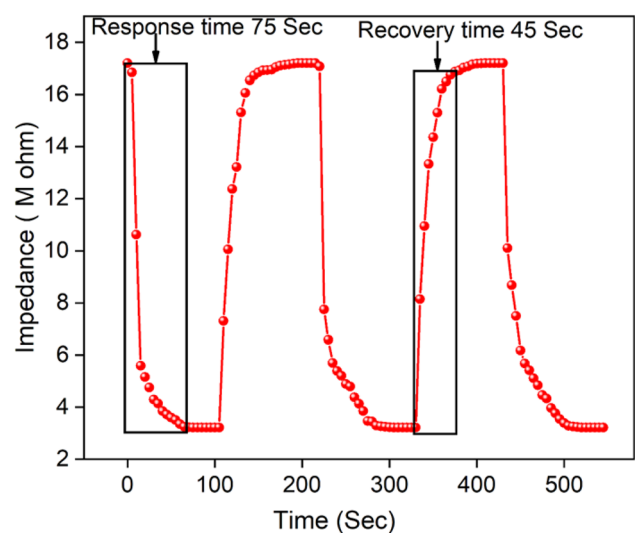
30% RH to 60% RH, the maximum calculated hysteresis loss of the PVDF-CdSe (20-min UV)-based humidity sensor was approximately 3%. Conversely, in the higher RH region spanning from 60% RH to 90% RH, the maximum calculated hysteresis loss was around 14%.

Table 1 shows the Regression analysis performed on the electrical response of the PVDF-CdSe (20-min UV)-based resistive humidity sensor.

Based on the regression analysis, the electrical response of the PVDF-CdSe (20-min UV)-based resistive humidity sensor demonstrates a robust linear relationship. The high R-squared value of 0.7869 indicates that approximately 78.7% of the variability in the y-axis values can be explained by the linear relationship with the x-axis values. The adjusted R-squared value of 0.7442 suggests that the linear relationship is robust and not heavily influenced by outliers or excessive complexity. The low standard error of 1.9649 further supports the conclusion of a well-fitted linear regression model. Therefore, it can be concluded that the PVDF-CdSe (20-min UV)-based resistive humidity sensor exhibits a significant linear relationship.

The response and recovery durations of impedance humidity sensors are other essential factors to consider when considering a sensor's performance. The humidity sensor's response time is defined as the time it takes for the sensor to reach 95% relative humidity (in the current study: 30–95%). The recovery time is when a sensor returns to its starting RH level (in this case, 30 %RH) after being exposed to 95

%RH. PVDF-CdSe (20-min UV)-based impedance sensors provide reliable and reproducible response and recovery curves, as shown in Fig. 5. At room temperature of 25°C, the response and recovery times of the PVDF-CdSe (20-min UV)-based impedance sensor was determined to be 75 and 45 s, respectively. In addition, the stability of a PVDF-CdSe (20-min UV)-based impedance humidity sensor was also performed. The electrical response of the prepared sensor was initially tested and then after 2, 4, and 6 months to analyze sensor stability. Figure S1 depicts the sensors' performance in response to changes in humidity level. The impedance response of PVDF-



**Fig. 5** Response and recovery cycles of PVDF-CdSe (20-min UV)-based resistive humidity sensors response taken at  $25 \pm 1$  °C

**Table 2** Summary of the carbon-based Humidity sensors

Material experimental details	Sensing range	Response/recovery	References
1:2 concentration (PVDF: CdSe) was prepared and coated on ITO 20 min of UV exposure	30%RH to 90%RH	Response: 75 s Recovery: 45 s	This work
Graphene quantum dots (GQDs) Pyrolysis of citric acid	15 and 80% of RH	Response: 5 s Recovery: 90 s	[20]
N-S-co-doped graphene quantum dots (GQDs) From graphite waste dissolved in H <sub>2</sub> SO <sub>4</sub> /HNO <sub>3</sub> 12 h and at 120 °C; Deep yellow supernatant N-GQDs	0.79% at 60%RH	Response: 15 s Recovery: 55 s	[7]
Polymeric Graphene Quantum Dots-Based Nanocomposites From graphite flake (GF), KMnO <sub>4</sub> , and HNO <sub>3</sub>	60 to 80%	Response: 20 s Recovery: 45 s	[21]
Graphene quantum dots From citric acid warmed to 200 °C	0–52% 52–97%	Response: 10 s Recovery: 400 s	[22]
Zinc oxide nanoparticles Polypyrrole polymer	11%RH–75%RH	Response: 180 s Recovery: 60 s	[23]

CdSe (20-min UV) humidity sensors remains stable over time. Table 2 is synoptic table summarizing recent Progress in humidity-sensing quantum dots-based materials and comparing them with the current work. One can note that the present work has shown a good response and recovery times compared to the previous study.

## 5 Conclusion

The effect of UV/ozone treatment time on surface hydrophilicity and sensing properties was tested. The prepared CdSe are fluorescent and rich in oxygen, cadmium, selenide, and carbon. The prepared CdSe were then mixed with PVDF polymer matrix, coated on ITO surfaces, and a resistive humidity sensor was prepared. AFM was used to monitor the prepared films' texture, distribution over the surface, and size. Overall, the hydrophilicity of the developed films increases with UV radiation exposure time, leading to enhanced water vapor absorption on the surface of the sensing film. Due to the enhanced water vapor absorption, sensitivity is improved and water vapor can be detected even at low humidity levels. The PVDF-CdSe sensors' electrical response after 30 min of UV/ozone exposure represents an irregular response at higher humidity levels (over 80% RH). The sensors' electrical response seems linear after 20 min of UV/ozone exposure. The PVDF-CdSe (20 min UV) sensor's highest calculated hysteresis loss is roughly 3% between 30 and 60% RH and

around 14% between 60 and 90% RH. The response and recovery times of the PVDF-CdSe (20-min UV)-based impedance humidity sensor was determined to be 75 and 45 s, respectively. These results indicate that UV/ozone treatment of the PVDF-CdSe nanocomposite film enhances the sensing film's hydrophilicity without deforming the surface of the sensing film; however, an optimum UV/ozone exposure time of 20 min is recommended.

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## Author contributions

Conceptualization: KJ and ZA; Methodology: KJ, SM, HM, and SAS; Data curation and writing of the original draft: KJ, SM, HM, and SAS; Validation and writing, reviewing, and editing of the manuscript: MC, AMA, ZA, MFA, and MA.

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## Data availability

Data will be provided on reasonable request.

## Declarations

**Conflict of interest** The authors have no conflict of interest.

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