

Article

Humidity Sensors Based on ZnO-BiFeO₃ Nanocomposites

Rachida Douani ¹, M'Hand Oughanem ¹ , Hayat Hammouche ², Malika Saidi ¹, Nouara Lamrani ¹ ,
Yannick Guhel ³, Ahcène Chaouchi ¹, Bertrand Boudart ^{3,*} and Saliha Rabehi ²

¹ Laboratoire de Chimie Appliquée et Génie Chimique, Faculté des Sciences, UMMTO de Tizi Ouzou, Tizi-Ouzou 15000, Algeria; rachida.douani@ummto.dz (R.D.); mhand.oughanem@ummto.dz (M.O.); malika.saidi@ummto.dz (M.S.); nouara.lamrani@ummto.dz (N.L.); ahcene.chaouchi@ummto.dz (A.C.)

² Laboratoire de Chimie Appliquée et Génie Chimique, Faculté de Génie Electrique, UMMTO de Tizi Ouzou, Tizi-Ouzou 15000, Algeria; hayat.hammouche@ummto.dz (H.H.); saliha.rabehi@ummto.dz (S.R.)

³ Université Caen Normandie, ENSICAEN, CNRS, Normandie University, GREYC UMR6072, F-14000 Caen, France; yannick.guhel@unicaen.fr

* Correspondence: bertrand.boudart@unicaen.fr

Abstract

The aim of this study was to investigate the impact of ZnO nanoparticles on the humidity-sensing properties of BiFeO₃ nanoparticles. BFO, ZnO, and x% ZnO-BiFeO₃ nanoparticles were synthesized using chemical processes and then analyzed by Scanning Electron Microscopy (SEM), X-Ray Diffraction (XRD), and Raman spectroscopy. The electrical capacitance of the sensors was measured using an impedance meter over a Relative Humidity (RH) range of 17 to 94% at room temperature and at an applied frequency of 100 Hz. This paper demonstrates that adding zinc oxide (ZnO) to bismuth ferrite (BFO) materials significantly improves the humidity response of BFO-based sensors. Indeed, a response of $2.8 \times 10^6\%$ was achieved for 20% ZnO-BFO-based sensors, compared with $5.8 \times 10^3\%$ for a pure BFO-based sensor. At the same time, a low hysteresis effect and excellent long-term stability were observed. In conclusion, the addition of ZnO nanoparticles provides excellent humidity-sensing properties to the BFO material, thereby contributing to its wide range of applications.

Keywords: sensor; humidity; sensitivity; ZnO-BiFeO₃; nanocomposite

1. Introduction

Humidity sensors play a crucial role in various aspects of human life. They facilitate the monitoring and regulation of humidity levels in a wide range of fields, such as cryogenics and food processing, medical instrumentation, antique conservation, electronics, and chemical materials [1,2].

Various sensing materials have been used in the development of humidity sensors, namely polymers [3,4], inorganic/organic composites [5,6], ceramic materials [7,8], cellulose-based materials [9], clay-based materials such as halloysite nanotubes [10], attapulgite [11], and sepiolite nanofibers [12]. Metal oxides, in their pure form or as composites, are more commonly used in the development of sensing layers for humidity sensors due to their low cost and ease of fabrication. These materials have physicochemical properties that are perfectly suited to this type of application, such as a large specific surface area, chemical and thermal stability, and numerous sites for the adsorption of water molecules [13,14]. Humidity sensors can operate according to different transduction mechanisms, such as resistive [15], capacitive [16], impedance [17], optical [18], frequency [19], and electrochemical sensors [20]. Capacitive humidity sensors are particularly attractive



Academic Editors: Meng Lin and Chuanxu Yang

Received: 29 April 2026

Revised: 15 June 2026

Accepted: 16 June 2026

Published: 25 June 2026

Copyright: © 2026 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the [Creative Commons Attribution \(CC BY\)](https://creativecommons.org/licenses/by/4.0/) license.

because of their low power consumption, simple structure, good stability, and suitability for room-temperature operation [2].

Bismuth ferrite BiFeO_3 perovskite is a p-type semiconductor and an archetypal room temperature multiferroic material that has attracted significant research interest in recent years. It possesses various properties, including a narrow band gap (ranging from 2.2 to 2.8 eV at 300 K), excellent chemical stability [21], and the coexistence of simultaneous ferroelectric and magnetic parameters. BiFeO_3 has been widely investigated in sensing applications, particularly in gas sensing applications such as Liquefied Petroleum Gas (LPG) [22], ammonia [23], SO_2 [24], ethanol [25,26], and acetone [27–29]. However, to the best of our knowledge, there are relatively few literature reports on the humidity-sensing properties of BiFeO_3 nanoparticles [30–32]. Indeed, the capacitance of the BFO film-based sensor increases from 25 to 1410 pF when the relative humidity varies from 30% to 90% [32]. Likewise, the humidity sensitivity of the BFO nanoparticle-based sensor rises from 0.7 up to 4652% for an RH ranging from 34% to 92% [31].

In a previous study [31], we highlighted that adding carbon fibers (CF) to BiFeO_3 nanoparticles significantly improves the humidity-sensing properties. Thus, the BFO/CF nanocomposite-based sensor exhibits a humidity sensitivity of up to 12640% for RH varying up to 92%, while the maximum sensitivity of a BFO-based sensor does not exceed 4652% over the same relative humidity.

We believe that incorporating materials other than CF into BFO nanoparticles may also increase their sensitivity to humidity. In terms of material choice, zinc oxide is an environmentally friendly n-type semiconductor that demonstrates enhanced electron mobility and reduced recombination losses when compared to other wide bandgap oxides [33]. Moreover, it possesses a combination of properties necessary for an ideal sensor material, including a bandgap of 3.37 eV, a high exciton binding energy of 60 meV, a high concentration of oxygen vacancies, and excellent chemical and thermal stability. Additionally, ZnO materials offer the advantage of being inexpensive, easy to prepare, and suitable for mass production [34,35]. Various types of ZnO morphologies have been widely utilized as gas and humidity sensors, including nanorods [36,37], nanowires [38,39], nanoplatelets [40], nanotubes [41], and nanoparticles [42–45]. However, some humidity sensors based on pure ZnO may exhibit slow response times, poor linearity [46], poor response in low humidity environments, and a large hysteresis [47,48].

In the present study, ZnO nanoparticles were employed as an alternative additive to further improve the sensing properties of BiFeO_3 perovskite. The humidity-sensing properties of the BFO and x% ZnO-BFO-based sensors (where $x = 10$ and 20) were evaluated at room temperature over a range of 17% to 94% relative humidity. The results showed that the inclusion of ZnO nanoparticles greatly enhanced the humidity-sensing performance of pure BFO particles.

2. Materials and Methods

2.1. Preparation of BFO, ZnO and x% ZnO-BFO Particles

Chemical reagents of high analytical purity were utilized in this study. The chemical reagents used included iron (III) nitrate nanohydrate ($\text{Fe}(\text{NO}_3)_3 \cdot 9 \text{H}_2\text{O}$) (99%), bismuth nitrate pentahydrate ($\text{Bi}(\text{NO}_3)_3 \cdot 5 \text{H}_2\text{O}$) (99%), nitric acid HNO_3 (69%), citric acid monohydrate ($\text{C}_6\text{H}_8\text{O}_7 \cdot \text{H}_2\text{O}$) (99.5%), zinc acetate monohydrate $\text{Zn}(\text{CH}_3\text{COO})_2 \cdot \text{H}_2\text{O}$, sodium hydroxide (NaOH), and deionized water. All chemical reagents were purchased from Sigma-Aldrich (St. Louis, MO, USA).

BiFeO_3 nanoparticles were prepared using a standard sol–gel method. Initially, a 0.105 M aqueous solution of bismuth nitrate was prepared by dissolving 2.5468 g of ($\text{Bi}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}$) in 50 mL of deionized water. Subsequently, 20 mL of nitric acid was

gradually added until a clear solution was obtained. Then, 2.02 g of $(\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O})$ was introduced to this solution. Finally, 5.0708 g of citric acid was incorporated into the mixture and stirred at 80 °C until a gel formed. This gel was then dried at 120 °C. The resulting xerogel was calcined at 350 °C for 2 h, followed by an additional 2 h at 700 °C.

ZnO nanoparticles were prepared using the co-precipitation method. Initially, 1.1 g of zinc acetate ($\text{Zn}(\text{CH}_3\text{COO})_2 \cdot \text{H}_2\text{O}$) was dissolved in 100 mL of methanol (CH_3OH). After stirring for 2 h, a solution of sodium hydroxide (1M) was added dropwise until a pH of 9 was reached. The mixture was stirred for 1 h and then allowed to stand for 12 h. The precipitate was washed several times with distilled water, and the resulting powder was dried at 100 °C for 12 h. Subsequently, the powder was calcined in two stages (at 250 °C/2 h, followed by 550 °C/2 h, with a heating rate of 5 °C/min).

x% ZnO-BFO nanocomposites were synthesized using the hydrothermal process with pre-prepared ZnO and BFO nanoparticles. An appropriate mass of ZnO was dispersed in 25 mL of distilled water, and the resulting suspension was then added to an aqueous suspension of BFO (100 mL, 10 mg/mL) under ultrasonic conditions. The resulting mixture was transferred to a 200 mL autoclave, heated at 150 °C for 6 h, and then naturally cooled to room temperature. Finally, the x% ZnO-BFO composites were washed with deionized water and dried at 100 °C for subsequent use. The various synthesis steps are illustrated in Figure 1.

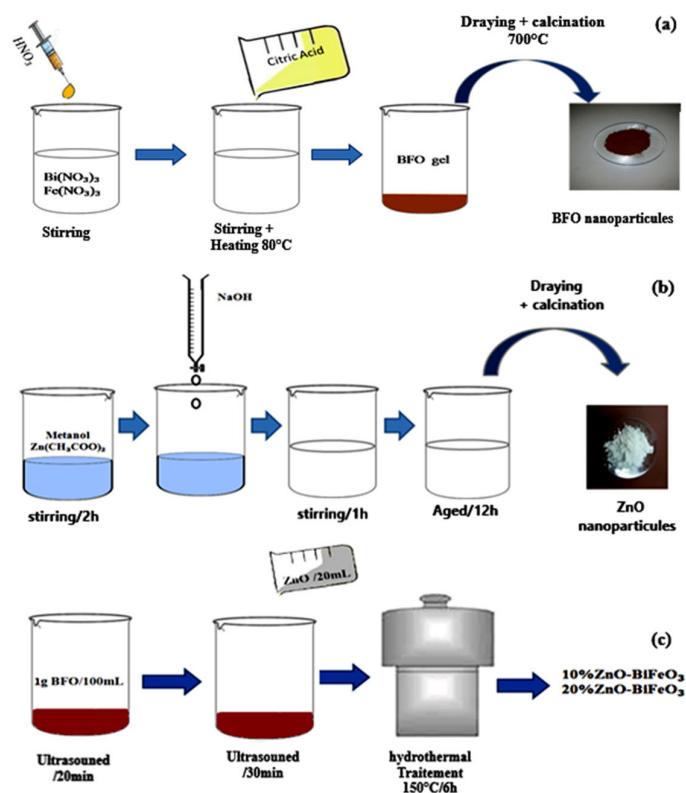


Figure 1. Different steps in the synthesis of: (a) BFO nanoparticles; (b) ZnO nanoparticles; (c) x% ZnO-BFO composites.

2.2. Material Characterizations

The morphology and dispersity of the prepared particles (BFO, ZnO, and x% ZnO-BFO) were investigated using a JEOL SEM7200 microscope (JEOL, Paris, France). The crystallographic structure and the effect of ZnO addition on the crystallinity of BFO nanoparticles were studied using a Panalytical Empyrean X-ray diffractometer (Malvern Panalytical, Paris, France) ($\text{Cu K}\alpha$ (1.5406 Å) at 45 kV). The synthesized particles were also character-

ized by Raman spectroscopy using a Renishaw InVia spectrometer (Renishaw, Bristol, UK) with a visible laser at a wavelength of 632.8 nm.

2.3. Preparation of the Sensors

The sensors under investigation consist of copper spiral electrodes with a surface area of 180 mm², onto which films of sensitive materials are deposited. The preparation process of these sensors is outlined as follows: a homogeneous solution was prepared by dispersing 0.2 g of the synthesized powders (BFO or x% ZnO-BFO) in approximately 3 drops of polyvinyl alcohol (PVA), which was used to ensure the adhesion of the powders to the surface of the structure. The mixture was then applied to the pre-cleaned electrodes and dried at 160 °C for 1 h to remove excess PVA in the sensor matrix.

2.4. Sensing Measurements

The sensors were tested at room temperature (approximately 25 °C) and at atmospheric pressure in a humidity-controlled environment, as depicted in Figure 2.

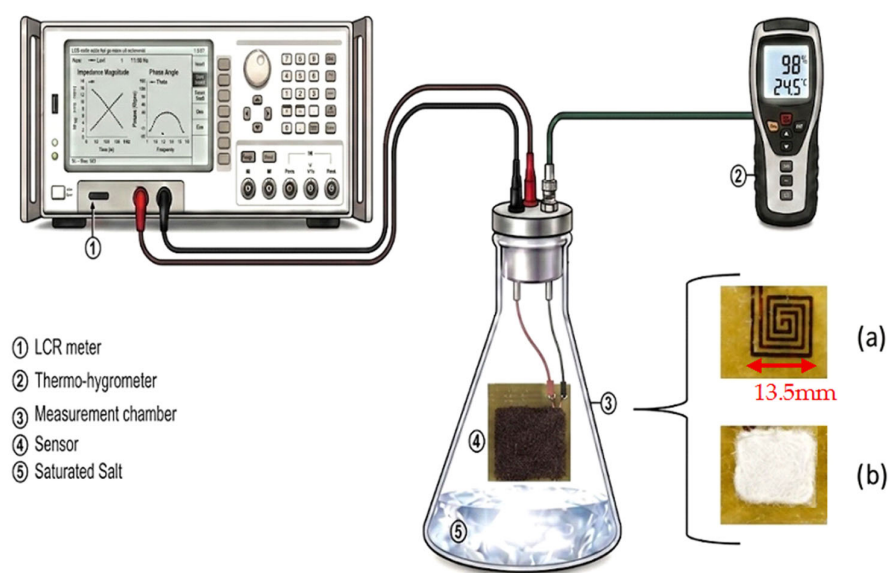


Figure 2. Schematic representation of the electrical measurement system. Example of the sensor (a) before and (b) after the sensitive layer deposition.

The electrical measurements were performed using an LCR HP 2484A impedance-meter, as shown in Figure 2, over a frequency range of 100 Hz to 1 MHz, with an applied voltage of 1 Volt. The humidity sensitivity of the sensors was characterized at different relative humidity levels, obtained by introducing various salts into the measurement systems.

3. Results and Discussion

3.1. Morphological and Structural Characterizations

Figure 3 shows scanning electron microscope images of BFO, ZnO, and x% ZnO-BFO powders. As shown in Figure 3a, the BFO particles appear as aggregates, probably due to the high surface reactivity of the material, which promotes particle agglomeration. By comparison, the SEM micrograph of the ZnO powder shown in Figure 3b reveals spherical nanoparticles with a relatively uniform distribution. Finally, SEM observations of the x% ZnO-BFO composite powder shown in Figure 3c,d reveal a homogeneous distribution of the particles, which are more widely dispersed than those in pure BFO.

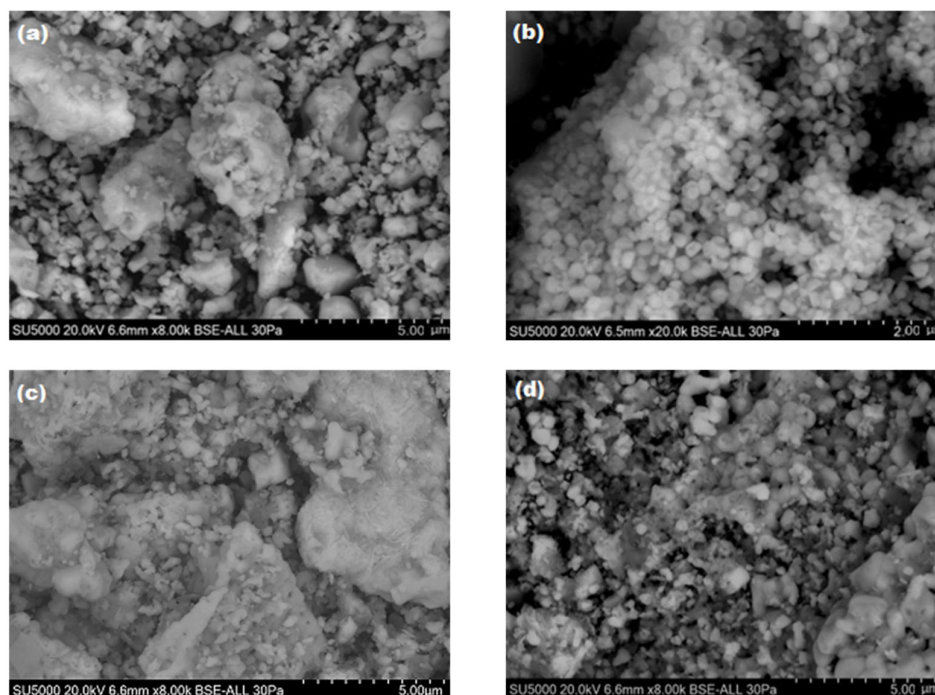


Figure 3. SEM micrographs of (a) pure BFO particles, (b) pure ZnO particles, (c) 10% ZnO-BFO composite, and (d) 20% ZnO-BFO composite.

To investigate the effect of the ZnO incorporation on the crystallinity of BFO during the synthesis of x% ZnO-BFO composite, X-ray diffraction patterns of BFO nanoparticles and x% ZnO-BFO composite powders are shown in Figure 4.

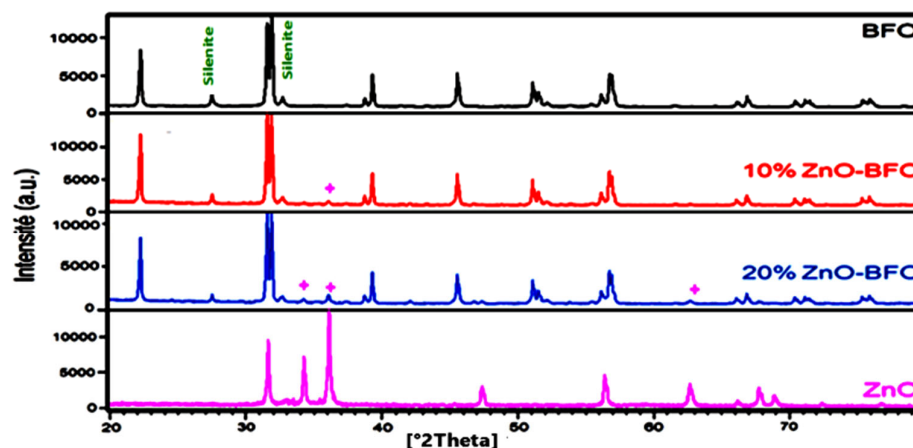


Figure 4. X-ray diffraction patterns of BFO, ZnO, and x% ZnO-BFO powders.

The X-ray diffraction peaks observed in the ZnO powder pattern are in good agreement with the standard space group (P6₃mc) wurtzite diffraction pattern (JCPDS No [01-089-7102]). However, no secondary phase was detected in the patterns. As for the BFO nanoparticles, they crystallize in a hexagonal structure with space group R3c (JCPDS No. 92-210-2910). The peaks appearing at $2\theta = 27.71^\circ$ and 32.904° correspond to sillenite ($\text{Bi}_{25}\text{FeO}_{38.96}$) (JCPDS No. 96-901-1269), which is identified as the secondary phase. Finally, the diffractograms of the x% ZnO-BFO composite powders show the same diffraction peaks as those observed in the diffraction profile of the BFO powder. It can therefore be concluded that the zinc oxide addition to BFO perovskite and the hydrothermal treatment do not alter the crystallographic structure of BFO. The presence of ZnO in the composite is confirmed

by the peaks appearing at $2\theta = 34.33^\circ$ and 36.17° . As expected, these two peaks are more intense for $x = 20\%$ than for $x = 10\%$.

In order to confirm these results, these materials were characterized by Raman spectroscopy. Figure 5 shows the Raman spectra of BFO, 10% ZnO-BFO, 20% ZnO-BFO, and ZnO. For BFO, the determination of the vibrational modes is based on a comparison of the experimental results obtained with those of previous studies on the R3c structure of BFO [49–54]. Ten active Raman modes were observed. The peaks observed at 138, 170, 220 cm^{-1} are attributed to the A_{1-1} , A_{1-2} , and A_{1-3} vibrational modes, respectively, of the Bi-O band. The peaks observed at 265, 275, 336, 469, 521, 613, and 1255 cm^{-1} are associated, respectively, with the E_2 , E_3 , E_5 , E_7 , E_8 , E_9 , and $2E_9$ vibrational modes of the Fe-O band. In addition, the peak observed at 820 cm^{-1} is probably due to the presence of the secondary phase (sillenite) previously identified by X-ray diffraction.

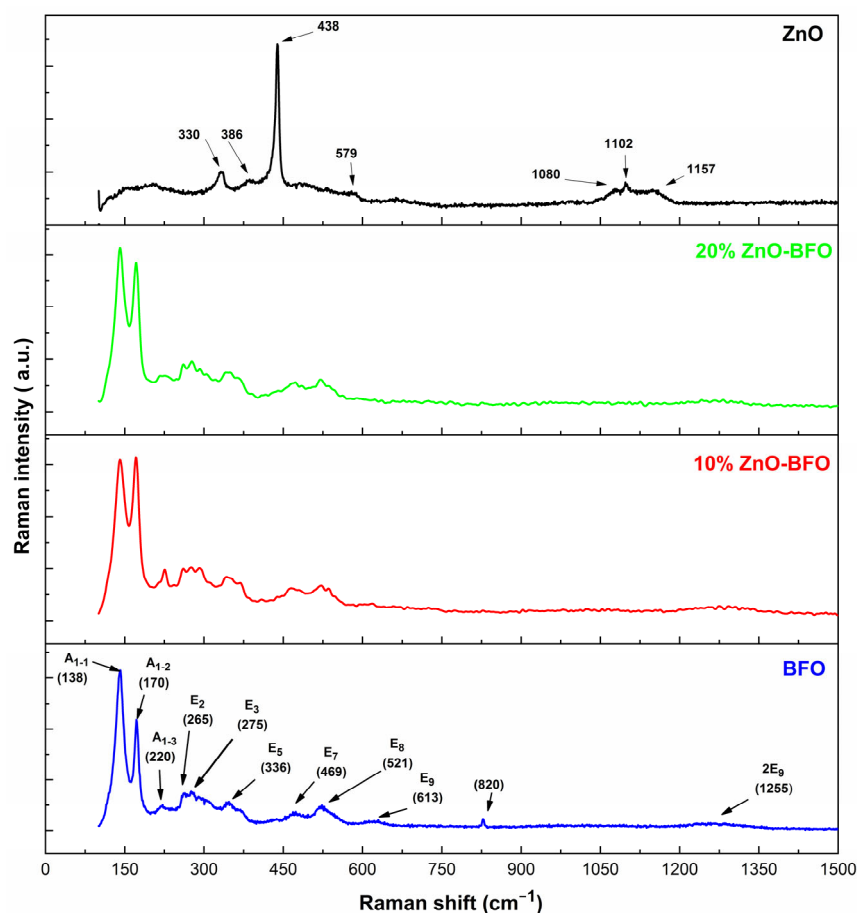


Figure 5. Raman spectra of BFO, ZnO, and BFO- $x\%$ ZnO powders at room temperature.

The Raman spectrum of ZnO exhibits the vibrational modes characteristic of the hexagonal wurtzite structure [55–57]. The peak at around 330 cm^{-1} is attributed to the E_2 (high)- E_2 (low) mode, which corresponds to second-order Raman scattering. The peak at 386 cm^{-1} is attributed to the A_1 (TO) mode, and that at 438 cm^{-1} is attributed to the E_2 (high) mode, both of which are characteristic of ZnO in the wurtzite structure [58]. The peak observed at 579 cm^{-1} is attributed to the A_1 (LO) vibration mode, indicating the presence of electrostatic order in the system. This mode is associated with the formation of structural defects such as oxygen vacancies (VO°), vacant zinc sites (VZn''), or impurities [57]. The peaks appearing at 1080 cm^{-1} and 1102 cm^{-1} are attributed to the combination of A_1 and E_2 modes. Finally, the peak at 1157 cm^{-1} is attributable to vibrational mode $2A_1$ (LO).

For ZnO-BFO composites, the Raman spectra are dominated by the characteristic modes of BFO, and the same Raman bands are observed in all these cases. These results are surprising, as no Raman modes are observed in ZnO.

That is why we have precisely characterized these powders over a range between 400 and 500 cm^{-1} . The Raman peak attributed to ZnO, which has the highest intensity, is located at around 438 cm^{-1} , as shown in Figure 6.

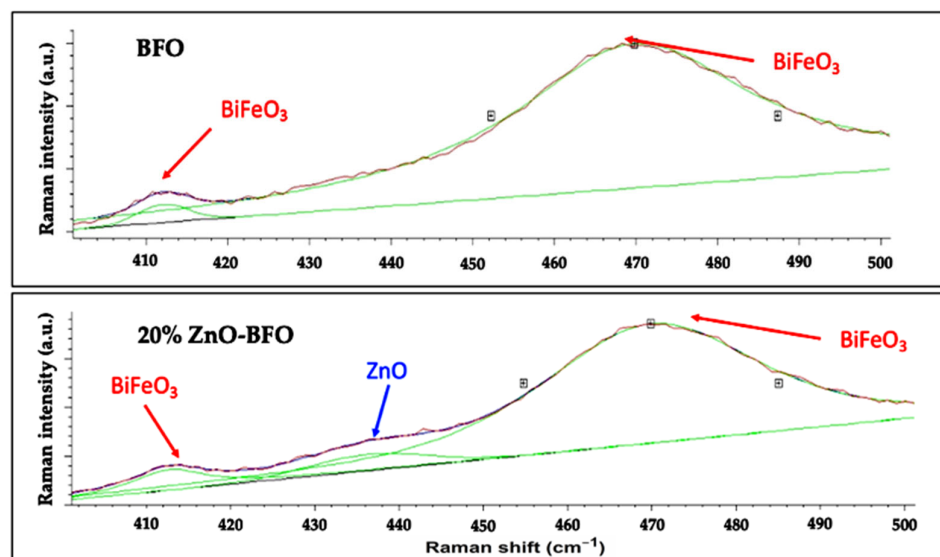


Figure 6. Comparison between the experimental and fitted Raman spectra of BFO and 20% ZnO-BFO powders at room temperature. The green curves represent the baseline and the three peaks used to perform the fit.

We fitted the experimental Raman spectra of 0% ZnO-BFO and 20% ZnO-BFO powders using a Lorentzian and Gaussian model, taking into account two Raman bands at 410 and 469 cm^{-1} attributed to the BFO powder [59–61], as well as a Raman band at 438.7 cm^{-1} associated with the ZnO powder [55–57]. There is a good match between the experimental spectra (red curves) and the fitted spectra (blue curves); it is difficult to distinguish between these two curves as they overlap perfectly.

Although the intensity of the Raman peak attributed to ZnO is low, it nevertheless indicates that ZnO is present in the 20% ZnO-BFO powder, unlike the 0% ZnO-BFO powder. Furthermore, this finding is consistent with the XRD characterizations presented earlier in this article.

3.2. Electrical Characterizations

The variation in capacitance (C_p) of BFO and x% ZnO-BFO-based sensors as a function of frequency (F) has been plotted for a humidity range of 17–94%, as shown in Figure 7. A decrease in C_p is thus observed when F changes from 10^2 to 10^6 Hz, regardless of the relative humidity, for the BFO-based sensor (Figure 7a). However, the effect of frequency appears to be more pronounced when the RH is high. For example, a decrease in C_p from 4 and 230 pF to 2 and 4 pF is observed in this frequency range for RH = 17% and 94%, respectively. Furthermore, it appears that the variation in C_p is more pronounced when F varies between 10^2 and 10^3 Hz than between 10^3 and 10^6 Hz.

These results indicate that the dependence of C_p on relative humidity is very weak above 10 kHz, as is often reported in the literature for the metal oxide sensors [62]. In this case, the electric field alternates too rapidly for the dipoles associated with the water molecules adsorbed on the surface of the sensor to reorient themselves. Consequently, these molecules cannot be effectively polarized during high-frequency electric field variations,

leading to a reduction in the dielectric constant and a negligible response. This means that the capacitance is practically independent of humidity and depends only on the electrical properties of the sensitive layer [63].

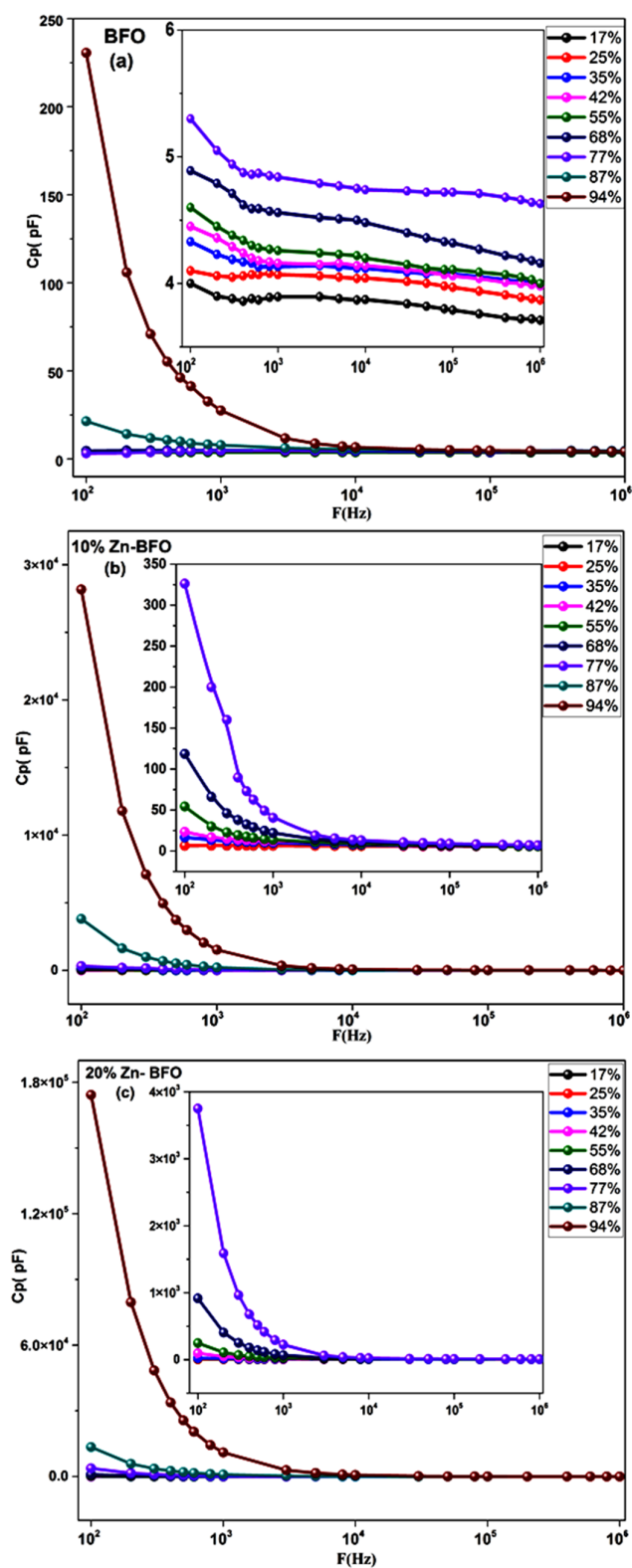


Figure 7. Evolution of capacitance versus frequency at RH values varying between 17% and 94% for BFO (a), 10% ZnO-BFO (b), and 20% ZnO-BFO (c) based sensors.

The same trends are observed for the 10% ZnO-BFO and 20% ZnO-BFO-based sensors (Figure 7b,c). Nevertheless, the C_p values are significantly higher for the sensor based on a 20% ZnO-BFO composite than for those based on a 10% ZnO-BFO composite and for the BFO material, regardless of F and RH values. Similarly, it is interesting to note that the 20% ZnO-BFO-based sensor seems to be more sensitive to humidity than the other two sensors, particularly at low frequencies such as 100 Hz, for example.

To confirm these results, Figure 8 presents the evolution of C_p versus RH for BFO, 10% ZnO-BFO, and 20% ZnO-BFO-based sensors.

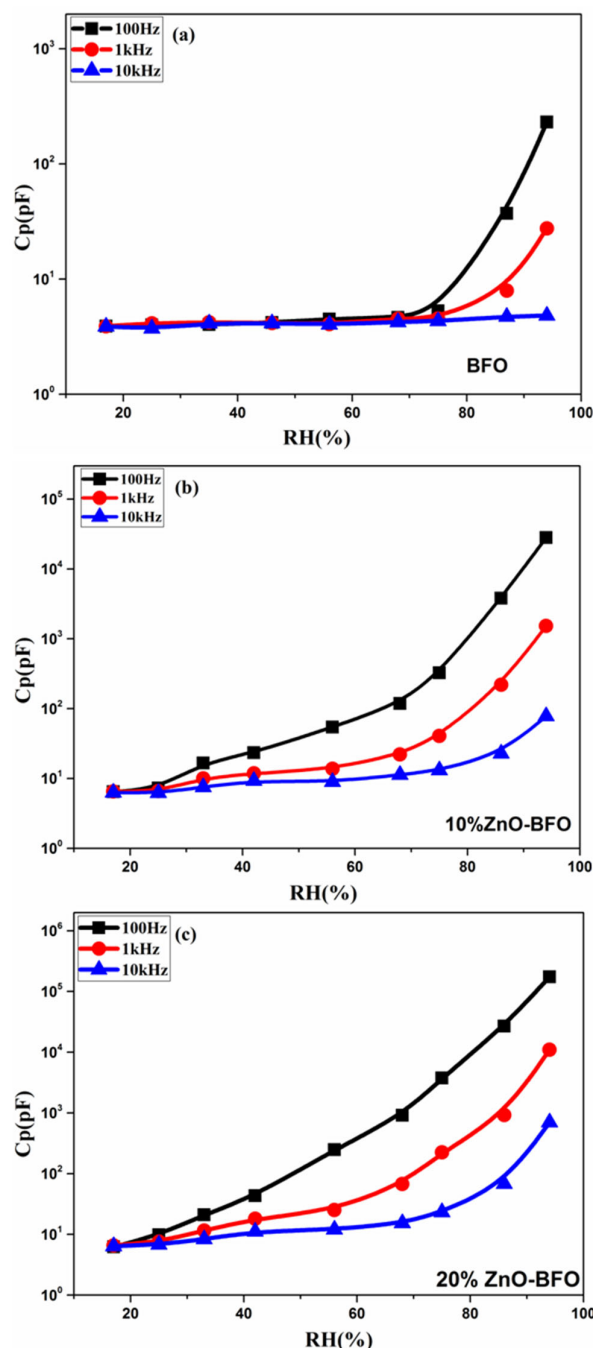


Figure 8. Evolution of capacitance versus RH values for BFO (a), 10% ZnO-BFO (b), and 20% ZnO-BFO (c) based sensors when the measurements are made at a frequency of 100 Hz (squares), 1 kHz (circles), and 10 kHz (triangles).

Thus, the C_p values are higher when measurements are carried out at a frequency of 100 Hz than when they are taken at 1 kHz, and the values obtained at 1 kHz are higher than those measured at 10 kHz, regardless of the RH range. Moreover, C_p varies between 68% and 94%, between 35% and 94% and between 25% and 94%, for BFO, 10% ZnO-BFO and 20% ZnO-BFO-based sensors, respectively. In addition, the variation in C_p with relative humidity is more pronounced at a frequency of 100 Hz for all sensors, as already seen in Figure 7.

It can be concluded that incorporating ZnO particles into the BFO material improves the sensitivity to humidity across all RH levels and increases sensitivity at low RH levels. For example, the 20% ZnO-BFO-based sensors detect moisture at RH = 25%, unlike those made solely with BFO (RH = 68%).

In any case, it can be seen that the increase in the C_p value is slow as RH rises from 17% to 68%, but then accelerates considerably between 68% and 94%. To confirm this phenomenon, Figure 9 shows the linear fit of Log (C_p) against relative humidity for the various sensors.

The results reveal two distinct linear relationships between Log (C_p) and relative humidity. An increase in the slope of the lines is observed for all sensors at humidity levels above their critical points: 75%, 68%, and 42% for BFO, 10% ZnO-BFO, and 20% ZnO-BFO-based sensors, respectively. This change in slope is likely due to the alteration in the mode of water molecule adsorption, transitioning from a chemisorbed monolayer at low RH levels to multilayer physisorption at high RH levels [64,65].

Figure 10 shows the humidity response $R(\%)$ of the various sensors, which is calculated using the following equation:

$$R(\%) = \frac{C_x - C_{17}}{C_{17}} \times 100 \quad (1)$$

where C_{17} and C_x are the capacities measured at RH = 17% and $x\%$, respectively.

We observe that $R(\%)$ of the 20% ZnO-BFO-based sensors is higher than that of the 10% ZnO-BFO-based sensors, which is itself superior to that of the BFO-based sensors, regardless of the RH. Indeed, $R(\%)$ increases from 2.6 to $5.8 \times 10^3\%$, from 12.7 to $4.3 \times 10^5\%$, and from 58.3 to $2.8 \times 10^6\%$ as RH varies between 25% and 94% for BFO, 10% ZnO-BFO, and 20% ZnO-BFO sensors, respectively. These results demonstrate that the highest sensitivity is achieved for 20% ZnO-BFO sensors, with $R(\%)$ being 22.5 and 483 times higher at RH values of 25% and 94%, respectively, compared to the BFO sensors.

The improvement in response and sensitivity of the sensors, resulting from the increase in the amount of ZnO incorporated, can be attributed to the presence of oxygen vacancies generated by the ZnO, as shown in Figure 5. Indeed, the increase in the number of active sites on the material's surface, caused by these oxygen vacancies, improves water adsorption and facilitates proton hopping at the surface, resulting in greater response to humidity.

The hysteresis effect is one of the key parameters used to assess the reliability of a humidity sensor; it is defined as the maximum difference between the sensor's responses during an adsorption–desorption cycle.

Thus, the humidity hysteresis, denoted $E(\%)$, was calculated from the characteristic curves $C_p = f(RH)$ measured for all sensors over a relative humidity range of 17% to 94%, using Equation (2) [48]:

$$E(\%) = \frac{\Delta m}{2Y_{FS}} \times 100 \quad (2)$$

where $\Delta m = C_p(\text{adsorption}) - C_p(\text{desorption})$ and $Y_{FS} = C_p(RH=94\%) - C_p(RH=17\%)$.

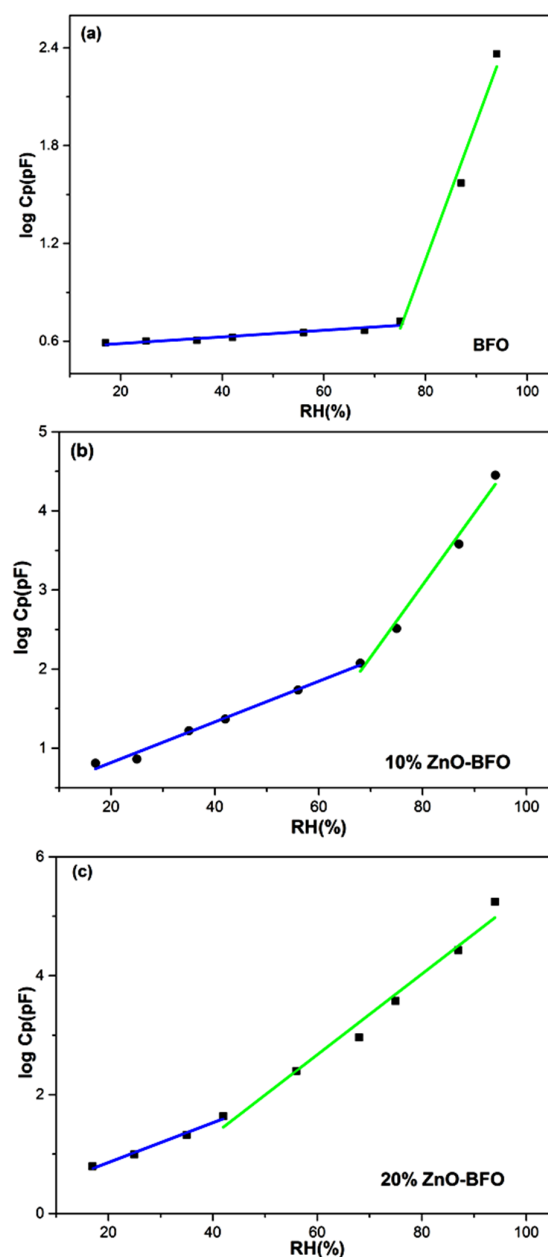


Figure 9. Linear fitted curves Log (C_p) versus RH (%) for BFO (a), 10% ZnO-BFO (b), and 20% ZnO-BFO (c) based sensors when the measurements are taken at a frequency of 100 Hz.

As shown in Figure 11, the sensors exhibit relatively narrow hysteresis loops, regardless of the sensors (<2.2%). This demonstrates that the addition of ZnO particles to BFO-based materials does not result in a significant hysteresis effect on the electrical behavior of the sensors, which is an advantage for humidity detection.

Response and recovery times are important parameters for the implementation of humidity sensors. Figure 12, therefore, shows the capacitance response of the BFO, 10% ZnO-BFO, and 20% ZnO-BFO sensors over a relative humidity range of 17% to 94%. These measurements show that the response time of the sensors decreases from 165 to 98 s as the ZnO powder content in the x% ZnO-BFO composites increases from 0 to 20%. At the same time, the recovery time increases from 40 to 64 s.

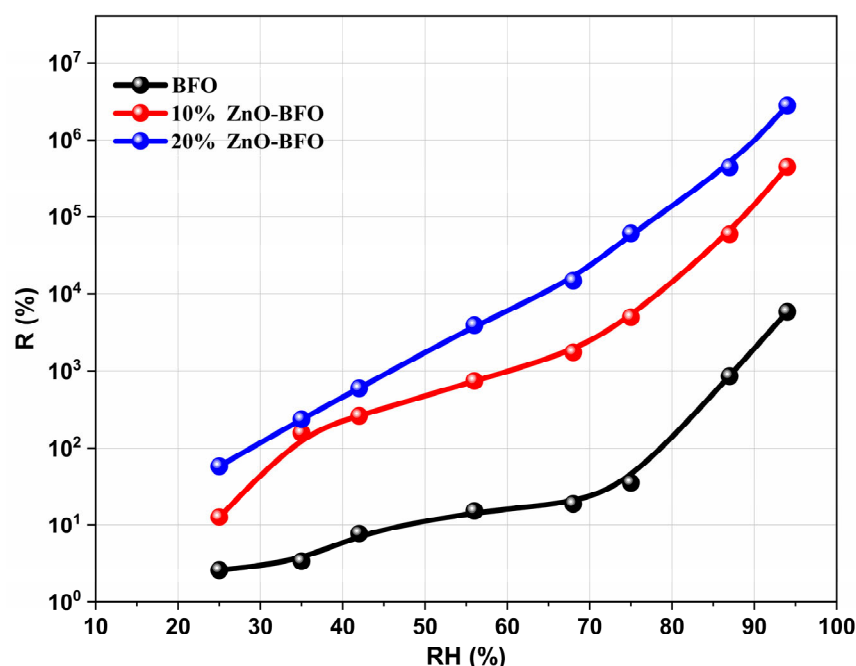


Figure 10. Response as a function of relative humidity for BFO, 10% ZnO-BFO, and 20% ZnO-BFO sensors. All measurements were taken at a frequency of 100 Hz.

These results are compared to those reported in the literature, as shown in Table 1.

Table 1. Comparison of the humidity detection characteristics of BFO and x% ZnO-BFO-based sensors with those of some humidity sensors reported in the literature.

Sensing Material	Range RH (%)	Humidity Response (%)	Hysteresis (%)	Response/Recovery Times (s)	Operating Frequency	Reference
BiFeO ₃	17–94	5.8×10^3	0.52	165/40	100 Hz	Present work
20% ZnO-BiFeO ₃	17–94	2.8×10^6	2.14	98/64	100 Hz	Present work
TiO ₂	11–95	3.5×10^3	4.66	3/50	100 Hz	[13]
BiFeO ₃	16–92	4.6×10^3	3.0	50/150	1 kHz	[31]
BiFeO ₃ /Carbon fibers	16–92	1.2×10^4	3.5	130/75	1 kHz	[31]
ZnO Nanorods	33–95	61.23			200 kHz	[37]
ZnO-SnO ₂	32–92	95	2.9	17/65	100 Hz	[43]
Graphite-oxide	11–97	4.4×10^6		3/5	10 Hz	[66]
ZnO-polyethylenimine	15–95	43.9×10^6	8	5/3	100 Hz	[67]

It is difficult to compare the performance of the sensors shown in Table 1, as their humidity sensitivity, hysteresis effect, response times, and recovery times have not been measured under identical conditions. For example, the operating frequency, sensitive parameters, and humidity range are not always the same. It should nevertheless be noted that the 20% ZnO-BFO sensors are among those in Table 1 that exhibit the highest humidity sensitivities and the lowest hysteresis effects.

The performances of sensors must be repeatable regardless of their field of application. For these reasons, the measurements of the sensor capacitance across the entire relative humidity range were repeated four times, as shown in Figure 13.

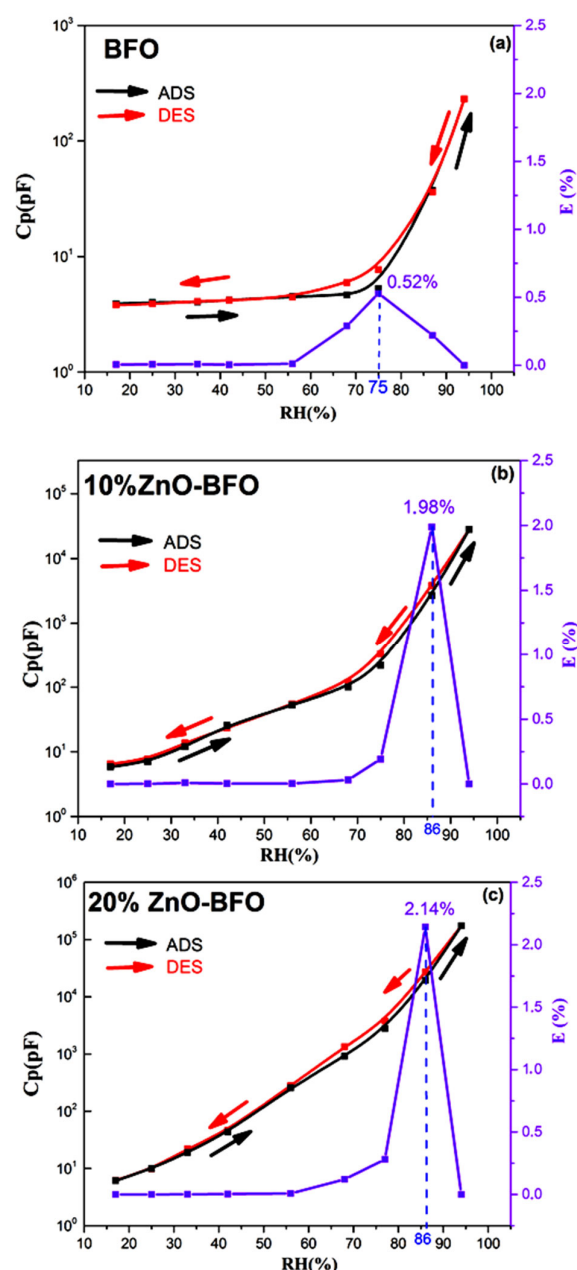


Figure 11. Variation in capacitance and hysteresis as a function of RH (%) during the adsorption–desorption processes of BFO (a), 10% ZnO-BFO (b), and 20% ZnO-BFO (c) sensors.

Thus, we observe that the first, second, third, and fourth C_p (RH) curves are the same for BFO sensors (Figure 13a). The same tendencies are observed for 10% ZnO-BFO (Figure 13b) and 20% ZnO-BFO (Figure 13c) sensors. To confirm these results, the Relative Standard Deviation (RSD) of the x% ZnO-BFO sensors has been calculated by using Equation (3):

$$\text{RSD} = \frac{\text{Standard deviation of capacitance}}{\text{average capacitance}} \times 100 \quad (3)$$

Under these conditions, it can be stated that RSD is always less than 6.5%, regardless of the sensors used, which confirms that the capacitance response as a function of RH is repeatable.

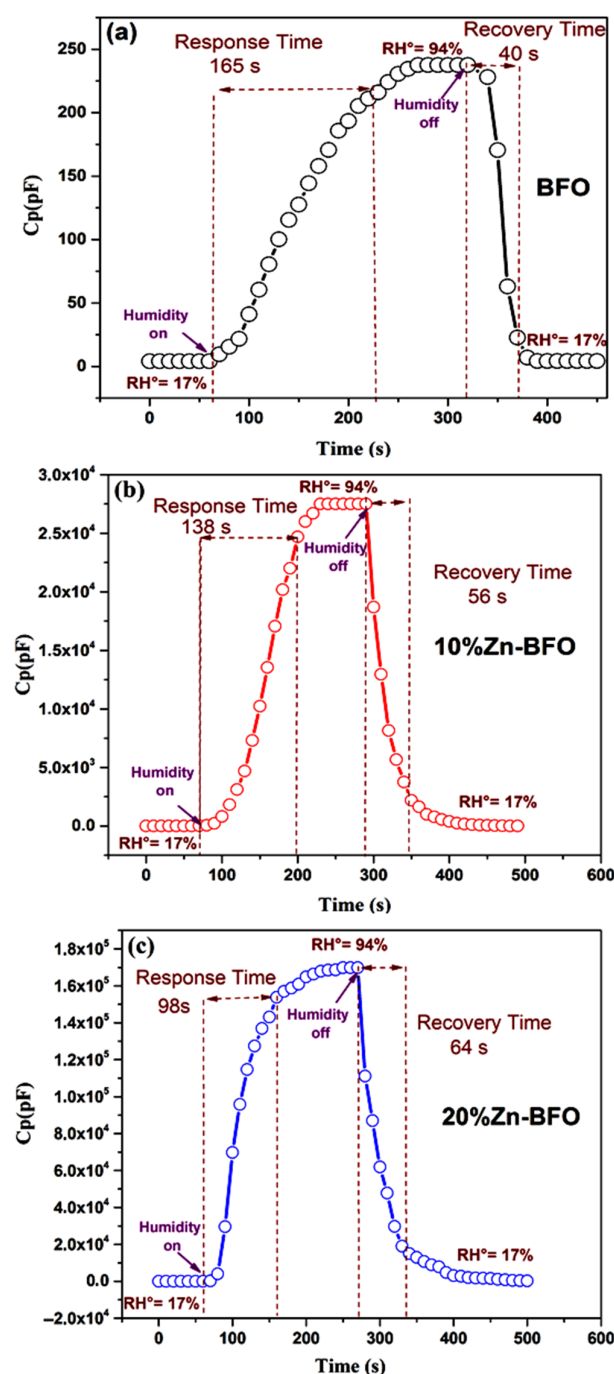


Figure 12. Response–recovery time graphs of BFO (a), 10% ZnO-BFO (b), and 20% ZnO-BFO (c) sensors.

As part of the long-term stability studies, measurements of capacitance as a function of relative humidity were taken every 10 days over a period of 200 days. The RSD results for various RH values ranging from 17% to 94% over time are presented in Tables 2 and 3, for BFO sensors and 20% ZnO-BFO sensors, respectively.

The calculated fluctuations are very small. That is why the change in C_p over time for different sensors is plotted on a logarithmic scale.

The results presented in Figure 14 demonstrate the remarkable stability of all the sensors, as evidenced by the minimal variation in their capacitance with respect to RH throughout the observation period.

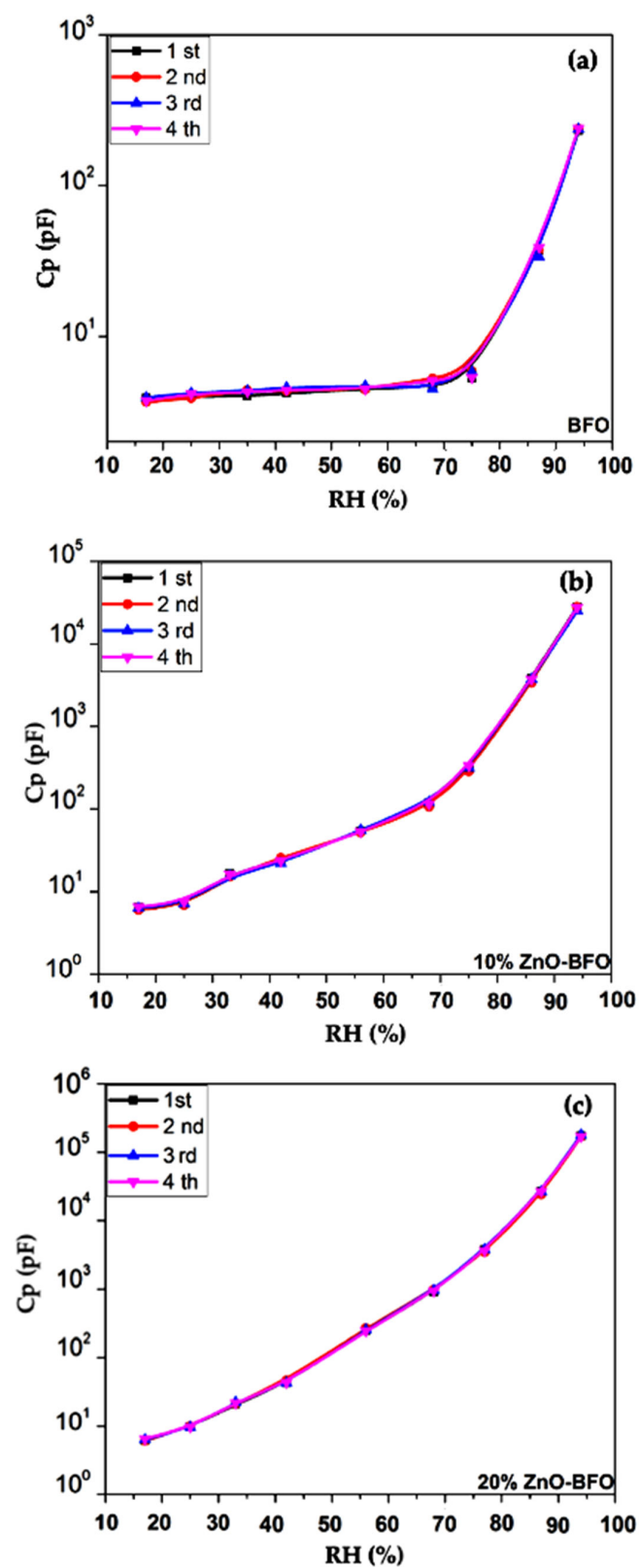


Figure 13. Repeatability of the capacitance response as a function of RH for BFO (a), 10% ZnO-BFO (b), and 20% ZnO-BFO (c) sensors when the measurements are carried out at a frequency of 100 Hz.

Table 2. Time-dependent evolution of RSD for various RH values ranging from 17% to 94% for a BFO-based sensor.

Time (Days)	RH = 17%	RH = 25%	RH = 35%	RH = 42%	RH = 56%	RH = 68%	RH = 75%	RH = 87%	RH = 94%
0	0	0	0	0	0	0	0	0	0
10	0	0	0	1.19	0	−1.68	0.18	0	0.16
20	−5.90	−2.50	1.70	3.09	−0.80	−2.52	−2.39	−2.40	0.35
30	0	2.50	2.97	4.52	0.33	−5.46	−1.65	0.37	0.69
40	−4.10	4.00	3.22	5.95	1.33	−4.41	0.18	0.48	0.99
50	−3.59	4.50	4.21	7.14	2.44	−1.47	0.99	0.61	1.27
60	−2.56	4.25	4.71	6.42	2.22	−2.31	−1.10	0.75	1.50
70	−2.56	4.00	4.96	3.57	2.20	−1.26	2.28	−1.80	1.81
80	−5.90	3.75	5.45	7.14	1.98	0.84	3.13	−1.80	1.93
90	−1.79	3.75	6.45	5.95	1.75	2.94	2.20	−1.66	2.17
100	−1.79	4.50	6.69	4.76	1.31	7.14	4.97	−0.18	2.50
120	−1.28	4.75	6.94	4.52	1.33	8.19	7.73	1.28	2.73
140	−0.51	5.00	7.69	4.04	1.11	10.29	4.97	2.63	2.98
160	0	5.00	8.18	3.73	0.89	9.24	7.55	3.51	3.20
180	1.28	5.00	8.68	3.50	0.44	10.50	9.76	6.79	3.57
200	2.82	5.25	8.93	3.09	0.22	11.34	12.33	7.73	3.20

Table 3. Time-dependent evolution of RSD for various RH values ranging from 17% to 94% for a 20% ZnO-BFO-based sensor.

Time (Days)	RH = 17%	RH = 25%	RH = 35%	RH = 42%	RH = 56%	RH = 68%	RH = 75%	RH = 87%	RH = 94%
0	0	0	0	0	0	0	0	0	0
10	0.48	−0.41	−0.47	−0.43	0.39	−0.26	0.35	3.10	−5.92
20	−0.16	0.30	−0.38	2.32	−3.94	0.11	−0.19	−1.12	−6.92
30	0.80	0	−0.19	5.33	3.69	0.28	−0.53	−1.18	−7.30
40	0.32	−0.81	0.09	10.00	−4.11	0	−2.13	−5.29	−6.40
50	0.64	−0.20	2.24	7.39	−1.16	7.05	−7.46	−8.97	−8.37
60	1.13	0.10	2.29	10.4	6.88	6.90	−5.33	−6.95	−5.70
70	−1.61	0.60	2.62	10.384	2.17	7.46	−7.25	−8.32	−8.49
80	1.51	0.71	2.72	9.77	10.74	7.49	−8.05	−4.98	−8.50
90	3.22	1.12	8.40	−1.14	−1.72	6.86	−7.76	−1.35	−5.90
100	4.99	0.81	6.97	1.81	−1.68	4.77	0.77	−4.71	−3.50
120	7.41	−1.93	5.06	5.43	−5.67	4.61	−4.45	−0.99	−3.55
140	8.86	−1.72	4.58	7.44	−1.97	4.53	0.90	−4.34	−3.54
160	6.92	−2.34	6.97	10.67	2.05	4.75	3.28	3.12	−3.55
180	10.30	−1.52	5.53	13.25	6.97	4.44	0.61	2.37	−5.68
200	12.72	−1.12	4.10	13.80	10.73	2.03	−3.23	7.14	−7.05

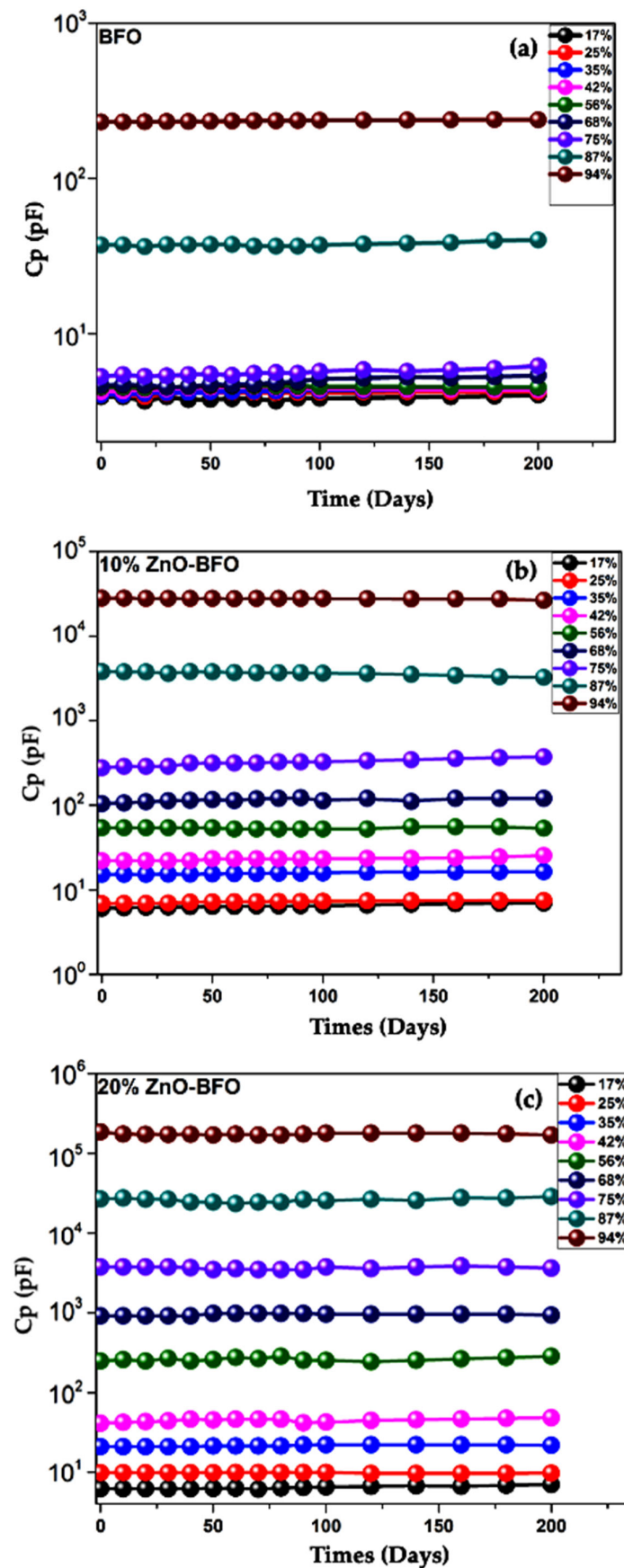


Figure 14. Long-term stability of BFO (a), 10% ZnO-BFO (b), and 20% ZnO-BFO (c) sensors for RH varying between 17% and 94%.

The detection mechanism can be explained by the modification of the electrical response of the sensitive material due to the adsorption and desorption of water molecules. Complex electrical impedance (CEI), as demonstrated by Nyquist diagrams, allows for a thorough and comprehensive analysis of the operating mechanism of humidity sensors. It offers insights into the various physicochemical phenomena taking place at the sensor's surface during water molecule adsorption.

The CEI of the sensors was measured at various relative humidity levels within the frequency range of 100 Hz to 1 MHz. The results are shown in Figures 15–17, where (Z') and (Z'') represent the real and imaginary parts of the impedance in the Nyquist diagram, respectively.

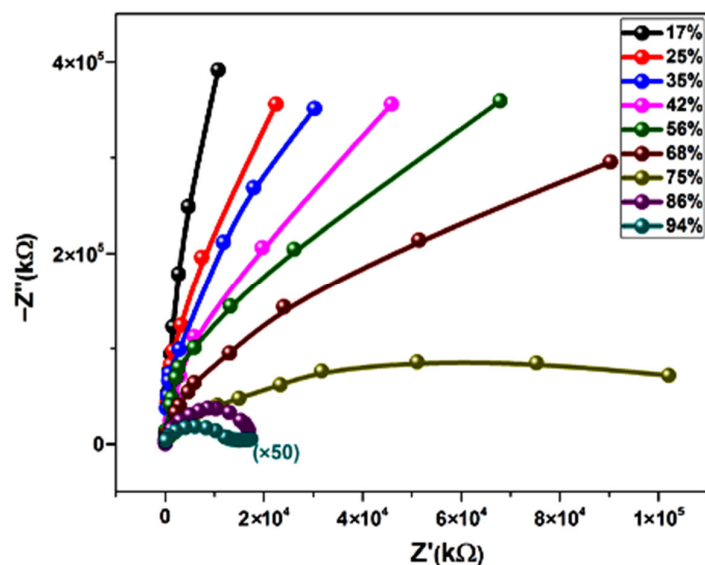


Figure 15. Complex electrical impedance plots of a pure BFO-based sensor for RH varying between 17% and 94%.

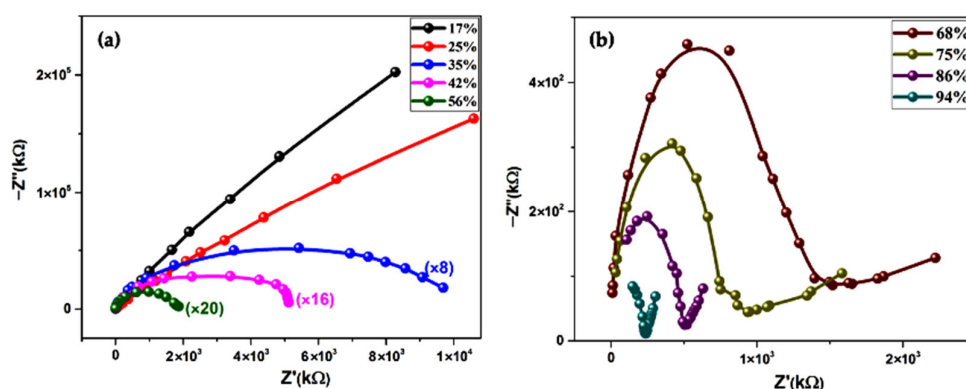


Figure 16. CEI plots of a 10% ZnO-BFO-based sensor at different humidity ranges: (a) 17–56% and (b) 68–94%.

At RH = 17%, only an arc with a very large radius of curvature is observed (almost a straight line). This corresponds to the formation of a discontinuous layer of chemisorbed water molecules on the surface of the sensor [68]. When the sensor is exposed to low humidity, water molecules adsorb onto the active sites (Bi^{3+} and Fe^{3+}) through a dissociative mechanism to form hydroxyl ions (OH^-) and protons (H^+). At this stage, ion conduction is impossible due to the discontinuity in the layer of adsorbed water molecules, so the sensor impedance is attributed to the intrinsic impedance of the sensing material [31].

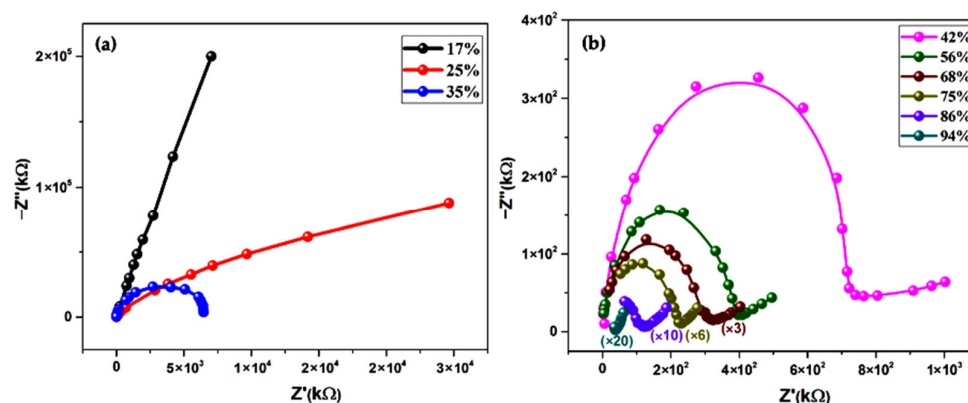
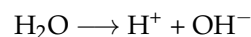


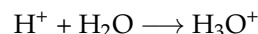
Figure 17. CEI plots of a 20% ZnO-BFO-based sensor at different humidity ranges: (a) 17–35% and (b) 42–94%.

As relative humidity increases, the radius of curvature of the arc decreases until a semi-circle is formed at 75% humidity. Within this range of relative humidity, a greater number of water molecules adsorb onto the surface, thereby forming a continuous film. This occurrence suggests the involvement of ionic conduction (proton hopping) in the charge transfer process, leading to a decrease in sensor impedance.

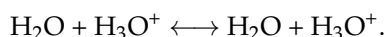
At RH = 94%, the semicircle is extended by a straight line. This phenomenon represents Warburg behavior, which is attributed to ion diffusion at the sensitive layer/electrode interface [69,70]. At high humidity levels, physisorbed multilayers form through the continuous adsorption of water molecules onto the material's surface. The physical bonds between the water molecules in the outer layer gradually undergo a dissociative process, releasing protons (H^+), as described by the Anderson and Parks model [71]:



The adsorbed water molecules then react with H^+ to form H_3O^+ , as shown in the following reaction [72]:



In fact, the outer water layer gradually exhibits liquid behavior, allowing free protons to move between adjacent water molecules through charge transport, following the Grotthuss mechanism:



Consequently, an increasing number of conductive proton transfer channels are formed, leading to enhanced polarization of the electric dipole moment in the sensitive material [63,73,74]. This polarization increase results in higher capacitance and, consequently, reduced impedance.

Compared with BFO-based sensors, those using x% ZnO-BFO composites show a significant reduction in electrical impedance, and a change in behavior is observed as the ZnO content of the composite increases. As shown in Figures 16 and 17, the semicircle and Warburg line appear at lower humidity levels than with pure BFO.

However, at 94% RH, the Nyquist curves show only the line at the rear end of the semicircle, whilst the front end becomes invisible. This phenomenon can be explained by the greater number of physisorbed water molecules, which improve the conductivity of the sensing layer through the penetration of protons into the films (x% ZnO-BFO), leading to a significant decrease in the sensor's impedance [68,75].

4. Conclusions

In this study, nanoparticles of zinc oxide, bismuth ferrite, and x% ZnO-BFO composites were synthesized. The results of XRD characterization showed that the BFO and ZnO nanoparticles crystallized in the hexagonal R3c and wurtzite phases, respectively. Furthermore, XRD measurements also demonstrate that the zinc oxide addition to the BFO perovskite and hydrothermal treatment do not alter the crystallographic structure of BFO. All these findings were confirmed by Raman spectroscopy.

Humidity sensors were fabricated using BFO-based materials, 10% ZnO-BFO and 20% ZnO-BFO, as the humidity-sensitive layers, and were then tested at relative humidity levels ranging from 17 to 94% at room temperature. It was found that the incorporation of ZnO into the BFO perovskite was beneficial for humidity detection. The maximum humidity response of $2.8 \times 10^6\%$ was obtained for the 20% ZnO-BFO-based sensor. At the same time, a low hysteresis effect, reduced response time, good repeatability of the capacitive response as a function of RH, and excellent long-term stability were observed. The increase in humidity sensitivity is explained by the presence of oxygen vacancies generated by the ZnO.

For these reasons, we can conclude that the incorporation of ZnO into BFO-based materials represents a promising alternative for improving the humidity-sensing properties of BFO-based sensors.

Author Contributions: Methodology, M.S.; Validation, N.L., Y.G. and B.B.; Formal analysis, H.H., Y.G. and S.R.; Investigation, M.O., H.H. and M.S.; Data curation, M.O.; Writing—original draft, R.D.; Writing—review and editing, B.B.; Visualization, A.C.; Supervision, N.L., Y.G., A.C. and B.B.; Project administration, N.L. All authors have read and agreed to the published version of the manuscript.

Funding: This research received no external funding.

Data Availability Statement: The data are available on request from the authors.

Conflicts of Interest: The authors declare no conflict of interest.

References

- Farahani, H.; Wagiran, R.; Hamidon, M. Humidity Sensors Principle, Mechanism, and Fabrication Technologies: A Comprehensive Review. *Sensors* **2014**, *14*, 7881–7939. [[CrossRef](#)] [[PubMed](#)]
- Blank, T.A.; Eksperiandova, L.P.; Belikov, K.N. Recent trends of ceramic humidity sensors development: A review. *Sens. Actuators B Chem.* **2016**, *228*, 416–442. [[CrossRef](#)]
- Li, X.; Zhuang, Z.; Qi, D.; Zhao, C. High sensitive and fast response humidity sensor based on polymer composite nanofibers for breath monitoring and non-contact sensing. *Sens. Actuators B Chem.* **2021**, *330*, 129239. [[CrossRef](#)]
- Boudaden, J.; Steinmaßl, M.; Endres, H.E.; Drost, A.; Eisele, I.; Kutter, C.; Müller-Buschbaum, P. Polyimide-Based Capacitive Humidity Sensor. *Sensors* **2018**, *18*, 1516. [[CrossRef](#)] [[PubMed](#)]
- Li, X.Z.; Liu, S.R.; Guo, Y. Polyaniline-intercalated layered double hydroxides: Synthesis and properties for humidity sensing. *RSC Adv.* **2016**, *6*, 63099–63106. [[CrossRef](#)]
- Kumar, R.; Yadav, B.C. Fabrication of Polyaniline (PANI)—Tungsten oxide (WO₃) Composite for Humidity Sensing Application. *J. Inorg. Organomet. Polym. Mater.* **2016**, *26*, 1421–1427. [[CrossRef](#)]
- Tripathy, A.; Pramanik, S.; Manna, A.; Shasmin, H.; Radzi, Z.; Abu Osman, N. Uniformly Porous Nanocrystalline CaMgFe_{1.33}Ti₃O₁₂ Ceramic Derived Electro-Ceramic Nanocomposite for Impedance Type Humidity Sensor. *Sensors* **2016**, *16*, 2029. [[CrossRef](#)] [[PubMed](#)]
- Almar, L.; Taracón, A.; Andreu, T.; Torrell, M.; Hu, Y.; Dezanneau, G.; Morata, A. Mesoporous ceramic oxides as humidity sensors: A case study for gadolinium-doped ceria. *Sens. Actuators B Chem.* **2015**, *216*, 41–48. [[CrossRef](#)]
- Huang, L.; Hu, Q.; Gao, S.; Liu, W.; Wei, X. Recent progress and applications of cellulose and its derivatives-based humidity sensors: A review. *Carbohydr. Polym.* **2023**, *318*, 121139. [[CrossRef](#)] [[PubMed](#)]
- Du, Z.; Chen, Q.; Xia, J.; Ao, J.; Pan, W.; Zhao, C. Halloysite nanotubes/Ti₃C₂T_x MXene composites based acoustic sensor for high performance humidity detection. *J. Mater. Sci. Mater. Electron.* **2026**, *37*, 70. [[CrossRef](#)]

11. Duan, Z.; Zhao, Q.; Wang, S.; Yuan, Z.; Zhang, Y.; Li, X.; Wu, Y.; Jiang, Y.; Tai, H. Novel application of attapulgite on high performance and low-cost humidity sensors. *Sens. Actuator B Chem.* **2020**, *305*, 127534. [\[CrossRef\]](#)
12. Afify, A.S.; Dadkhah, M.; Tulliani, J.-M. Sol-Gel Synthesis of Iron-Doped Sepiolite as a Novel Humidity-Sensing Material. *Ceramics* **2022**, *5*, 575–592. [\[CrossRef\]](#)
13. Gong, M.; Li, Y.; Guo, Y.; Lv, X.; Dou, X. 2D TiO₂ nanosheets for ultrasensitive humidity sensing application benefited by abundant surface oxygen vacancy defects. *Sens. Actuators B Chem.* **2018**, *262*, 350–358. [\[CrossRef\]](#)
14. Kumar, A.; Gupta, G.; Bapna, K.; Shivagan, D.D. Semiconductor-metal-oxide-based nano-composites for humidity sensing applications. *Mater. Res. Bull.* **2023**, *158*, 112053. [\[CrossRef\]](#)
15. Ragazzini, I.; Castagnoli, R.; Gualandi, I.; Cassani, M.C.; Nanni, D.; Gambassi, F.; Scavetta, E.; Bernardi, E.; Ballarin, B. A resistive sensor for humidity detection based on cellulose/polyaniline. *RSC Adv.* **2022**, *12*, 28217–28226. [\[CrossRef\]](#) [\[PubMed\]](#)
16. Duan, Z.; Li, J.; Yuan, Z.; Jiang, Y.; Tai, H. Capacitive Humidity Sensor Based on Zirconium Phosphate Nanoplates Film with Wide Sensing Range and High Response. *Sens. Actuators B Chem.* **2023**, *394*, 134445. [\[CrossRef\]](#)
17. Sekulić, D.L.; Ivetić, T.B. Characterization of an Impedance-Type Humidity Sensor Based on Porous SnO₂/TiO₂ Composite Ceramics Modified with Molybdenum and Zinc. *Sensors* **2023**, *23*, 8261. [\[CrossRef\]](#) [\[PubMed\]](#)
18. Shi, Y.; Wang, F.; Gao, F.; Kang, J.; Wu, J.; Ren, Z.; Zhou, J.; Dong, J.; Zhou, W.; Shen, C. High Recovery Speed Optical Humidity Sensing Enabled with GQD for Non-Contact Finger Distance Detection. *Sens. Actuators B Chem.* **2024**, *421*, 136541. [\[CrossRef\]](#)
19. Jung, S.I.; Jang, I.R.; Ryu, C.; Park, J.; Padhan, A.M.; Kim, H.J. Graphene Oxide Decorated Multi-Frequency Surface Acoustic Wave Humidity Sensor for Hygienic Applications. *Sci. Rep.* **2023**, *13*, 6838. [\[CrossRef\]](#) [\[PubMed\]](#)
20. Hu, J.; Duan, Z.; Tan, C.; Wang, Y.; Yuan, Z.; Jiang, Y.; Tai, H. Self-Powered Electrochemical Humidity Sensor Based on Cu/(Cellulose Nanocrystals/NaCl)/Zn Structure for Respiratory Rate Detection. *Inorg. Chem. Commun.* **2025**, *180*, 114994. [\[CrossRef\]](#)
21. Lam, S.M.; Sin, J.C.; Mohamed, A.R. A newly emerging visible light-responsive BiFeO₃ perovskite for photocatalytic applications: A mini review. *Mater. Res. Bull.* **2017**, *90*, 15–30. [\[CrossRef\]](#)
22. Waghmare, S.D.; Jadhav, V.V.; Gore, S.K.; Yoon, S.J.; Ambade, S.B.; Lokhande, B.J.; Mane, R.S.; Han, S.H. Efficient gas sensitivity in mixed bismuth ferrite micro (cubes) and nano (plates) structures. *Mater. Res. Bull.* **2012**, *47*, 4169–4173. [\[CrossRef\]](#)
23. Dziubaniuk, M.; Bujakiewicz-Korońska, R.; Suchanicz, J.; Wyrwa, J.; Rękas, M. Application of bismuth ferrite protonic conductor for ammonia gas detection. *Sens. Actuators B Chem.* **2013**, *188*, 957–964. [\[CrossRef\]](#)
24. Das, S.; Rana, S.; Mursalin, S.M.; Rana, P.; Sen, A. Sonochemically prepared nanosized BiFeO₃ as novel SO₂ sensor. *Sens. Actuators B Chem.* **2015**, *218*, 122–127. [\[CrossRef\]](#)
25. Dong, G.; Fan, H.; Tian, H.; Fang, J.; Li, Q. Gas-sensing and electrical properties of perovskite structure p-type barium-substituted bismuth ferrite. *RSC Adv.* **2015**, *5*, 29618–29623. [\[CrossRef\]](#)
26. Tong, T.; Chen, J.; Jin, D.; Cheng, J. Preparation and gas sensing characteristics of BiFeO₃ crystallites. *Mater. Lett.* **2017**, *197*, 160–162. [\[CrossRef\]](#)
27. Zhang, Y.; Xu, H.; Dong, S.; Han, R.; Liu, X.; Wang, Y.; Li, S.; Bu, Q.; Li, X.; Xiang, J. A fast response & recovery acetone gas sensor based on BiFeO₃ nanomaterials with high sensitivity and low detection limit. *J. Mater. Sci. Mater. Electron.* **2018**, *29*, 2193–2200. [\[CrossRef\]](#)
28. Chakraborty, S.; Pal, M. Highly selective and stable acetone sensor based on chemically prepared bismuth ferrite nanoparticles. *J. Alloys Compd.* **2019**, *787*, 1204–1211. [\[CrossRef\]](#)
29. Oughanem, M.; Douani, R.; Lamrani, N.; Guhel, Y.; Chaouchi, A.; Boudart, B. Enhanced volatile organic compound sensing properties of BiFeO₃ by carbon fibres addition. *Process. Appl. Ceram.* **2022**, *16*, 310–320. [\[CrossRef\]](#)
30. Ramirez, F.E.N.; Espinosa, E.E.; Pedroza, L.S.; Souza, J.A. Humidity sensing effect in Bi₂₅FeO₃₉ sillenite-like compound. *J. Mater. Sci.* **2016**, *51*, 10982–10989. [\[CrossRef\]](#)
31. Douani, R.; Lamrani, N.; Oughanem, M.; Saidi, M.; Guhel, Y.; Chaouchi, A.; Boudart, B. Improvement of humidity sensing performance of BiFeO₃ nanoparticles-based sensor by the addition of carbon fibers. *Sens. Actuators Phys.* **2020**, *307*, 111981. [\[CrossRef\]](#)
32. Zhang, Y.; Li, B.; Jia, Y. High Humidity Response of Sol-Gel-Synthesized BiFeO₃ Ferroelectric Film. *Materials* **2022**, *15*, 2932. [\[CrossRef\]](#) [\[PubMed\]](#)
33. Ko, S.H.; Lee, D.; Kang, H.W.; Nam, K.H.; Yeo, J.Y.; Hong, S.J.; Grigoropoulos, C.P.; Sung, H.J. Nanoforest of Hydrothermally Grown Hierarchical ZnO Nanowires for a High Efficiency Dye-Sensitized Solar Cell. *Nano Lett.* **2011**, *11*, 666–671. [\[CrossRef\]](#) [\[PubMed\]](#)
34. Zhu, L.; Zeng, W. Room-temperature gas sensing of ZnO-based gas sensor: A review. *Sens. Actuators Phys.* **2017**, *267*, 242–261. [\[CrossRef\]](#)
35. Yanto, W.F.; Salim, A.Q.; Lathifah, K.; Rohman, A.D.; Suryadi; Azzahidah, R.; Ansari, A.S.; Rochman, N.T. Humidity sensors based on doped ZnO: An overview. *Mater. Today Proc.* **2024**. [\[CrossRef\]](#)

36. Sudha, M.; Radha, S.; Kirubaveni, S.; Kiruthika, R.; Govindaraj, R.; Santhosh, N. Experimental study on structural, optoelectronic and room temperature sensing performance of Nickel doped ZnO based ethanol sensors. *Solid State Sci.* **2018**, *78*, 30–39. [\[CrossRef\]](#)
37. Narimani, K.; Nayeri, F.D.; Kolahdouz, M.; Ebrahimi, P. Fabrication, modeling and simulation of high sensitivity capacitive humidity sensors based on ZnO nanorods. *Sens. Actuators B Chem.* **2016**, *224*, 338–343. [\[CrossRef\]](#)
38. Sinha, M.; Mahapatra, R.; Mondal, B.; Maruyama, T.; Ghosh, R. Ultrafast and Reversible Gas-Sensing Properties of ZnO Nanowire Arrays Grown by Hydrothermal Technique. *J. Phys. Chem. C* **2016**, *120*, 3019–3025. [\[CrossRef\]](#)
39. Wei, S.; Zhou, M.; Du, W. Improved acetone sensing properties of ZnO hollow nanofibers by single capillary electrospinning. *Sens. Actuators B Chem.* **2011**, *160*, 753–759. [\[CrossRef\]](#)
40. Mani, G.K.; Rayappan, J.B.B. Novel and facile synthesis of randomly interconnected ZnO nanoplatelets using spray pyrolysis and their room temperature sensing characteristics. *Sens. Actuators B Chem.* **2014**, *198*, 125–133. [\[CrossRef\]](#)
41. Yu, X.; Song, F.; Zhai, B.; Zheng, C.; Wang, Y. Electrospun ZnO nanotubes and its gas sensing applications. *Phys. E* **2013**, *52*, 92–96. [\[CrossRef\]](#)
42. Jaballah, S.; Benamara, M.; Dahman, H.; Ly, A.; Lahem, D.; Debliquy, M.; Mir, L.E. Effect of Mg-doping ZnO nanoparticles on detection of low ethanol concentrations. *Mater. Chem. Phys.* **2020**, *255*, 123643. [\[CrossRef\]](#)
43. Velumani, M.; Meher, S.R.; Alex, Z.C. Composite metal oxide thin film based impedometric humidity sensors. *Sens. Actuators B Chem.* **2019**, *301*, 127084. [\[CrossRef\]](#)
44. Toranjizadeh, H.; Shabani, P.; Ramezani, A. Ethanol gas sensing improvement at high humidity levels and optical features using Ba-doped ZnO NPs. *Mater. Res. Express* **2019**, *6*, 095904. [\[CrossRef\]](#)
45. Misra, S.K.; Pandey, N.K. Analysis on activation energy and humidity sensing application of nanostructured SnO₂-doped ZnO material. *Sens. Actuators Phys.* **2016**, *249*, 8–14. [\[CrossRef\]](#)
46. Arularasu, M.V.; Harb, M.; Vignesh, R.; Rajendran, T.V.; Sundaram, R. PVDF/ZnO hybrid nanocomposite applied as a resistive humidity sensor. *Surf. Interfaces* **2020**, *21*, 100780. [\[CrossRef\]](#)
47. Lin, W.D.; Lin, Y.C.; Wu, R.J.; Chavali, M. Gr/3D-ZnO Nanocomposites as Humidity Sensors with Enhanced Sensing Response. *Polymers* **2021**, *13*, 1623. [\[CrossRef\]](#) [\[PubMed\]](#)
48. Feng, M.H.; Wang, W.C.; Li, X.J. Capacitive humidity sensing properties of CdS/ZnO sesame-seed-candy structure grown on silicon nanoporous pillar array. *J. Alloys Compd.* **2017**, *698*, 94–98. [\[CrossRef\]](#)
49. Yang, Y.; Sun, J.Y.; Zhu, K.; Liu, Y.L.; Wan, L. Structure properties of BiFeO₃ films studied by micro-Raman scattering. *J. Appl. Phys.* **2008**, *103*, 093532. [\[CrossRef\]](#)
50. Amouri, A.; Aydi, S.; Abdelmoula, N.; Dammak, H.; Khemakhem, H. Evidence of magnetoelectric coupling in 0.9BiFeO₃-0.1Ba [Ti_{0.95}(Yb_{0.5}Nb_{0.5})_{0.05}]O₃ ceramic. *J. Alloys Compd.* **2018**, *739*, 1065–1079. [\[CrossRef\]](#)
51. Si, Y.; Xia, Y.; Shang, S.; Xiong, X.; Zeng, X.; Zhou, J.; Li, Y. Enhanced Visible Light Driven Photocatalytic Behavior of BiFeO₃/Reduced Graphene Oxide Composites. *Nanomaterials* **2018**, *8*, 526. [\[CrossRef\]](#) [\[PubMed\]](#)
52. Ortiz-Quinonez, J.L.; Pal, U.; Villanueva, M.S. Effects of Oxidizing/Reducing Agent Ratio on Phase Purity, Crystallinity, and Magnetic Behavior of Solution-Combustion-Grown BiFeO₃ Submicroparticles. *Inorg. Chem.* **2018**, *57*, 6152–6160. [\[CrossRef\]](#) [\[PubMed\]](#)
53. Wang, T.; Song, S.H.; Wang, X.L.; Chen, J.J.; Tan, M.L. Chemical substitution-induced structure transition and enhanced magnetic and optical properties of sol-gel synthesized multiferroic BiFeO₃ nanoparticles. *J. Sol.-Gel Sci. Technol.* **2018**, *85*, 356–368. [\[CrossRef\]](#)
54. Kumar, A.; Sharma, P.; Yang, W.; Shen, J.; Varshney, D.; Li, Q. Effect of La and Ni substitution on structure, dielectric and ferroelectric properties of BiFeO₃ ceramics. *Ceram. Int.* **2016**, *42*, 14805–14812. [\[CrossRef\]](#)
55. Ristic, M.; Music, S.; Ivenda, M.; Popovic, S. Sol-gel synthesis and characterization of nanocrystalline ZnO powders. *J. Alloys Compd.* **2005**, *397*, L1–L4. [\[CrossRef\]](#)
56. Kumar, P.; Singh, V.; Sharma, V.; Rana, G.; Malik, H.K.; Asokan, K. Investigation of phase segregation in yttrium doped zinc oxide. *Ceram. Int.* **2015**, *41*, 6734–6739. [\[CrossRef\]](#)
57. Kane, A.O.; Ngom, B.D.; Sakho, O. Influence of *Adansonia digitata* leaves dye extraction solvent nature on the structural and physical properties of biosynthesized ZnO nanoparticles. *Mater. Today Proc.* **2021**, *36*, 290–297. [\[CrossRef\]](#)
58. Dutta, R.K.; Sharma, P.K.; Bhargava, R.; Kumar, N.; Pandey, A.C. Differential Susceptibility of *Escherichia coli* Cells toward Transition Metal-Doped and Matrix-Embedded ZnO Nanoparticles. *J. Phys. Chem. B* **2010**, *114*, 5594–5599. [\[CrossRef\]](#) [\[PubMed\]](#)
59. Szafraniak, I.; Polomska, M.; Hilczer, B.; Pietraszko, A.; Kepinski, L. Characterization of BiFeO₃ nanopowder obtained by mechanochemical synthesis. *J. Eur. Ceram. Soc.* **2007**, *27*, 4399–4402. [\[CrossRef\]](#)
60. Popa, M.; Crespo, D.; Calderon-Moreno, J.M.; Preda, S.; Fruth, V. Synthesis and Structural Characterization of Single-Phase BiFeO₃ Powder from Polymeric Precursor. *J. Am. Ceram. Soc.* **2007**, *90*, 2723–2727. [\[CrossRef\]](#)
61. Porporati, A.A.; Tsuji, K.; Valant, M.; Axelsson, A.K.; Pezzotti, G. Raman tensor elements for multiferroic BiFeO₃ with rhombohedral R3c symmetry. *J. Raman Spec.* **2010**, *41*, 84–87. [\[CrossRef\]](#)

62. Zhang, D.; Chang, H.; Li, P.; Liu, R.; Xue, Q. Fabrication and characterization of an ultrasensitive humidity sensor based on metal oxide/graphene hybrid nanocomposite. *Sens. Actuators B Chem.* **2016**, *225*, 233–240. [[CrossRef](#)]
63. Wang, J.; Xu, B.K.; Ruan, S.P.; Wang, S.P. Preparation and electrical properties of humidity sensing films of BaTiO₃/polystyrene sulfonic sodium. *Mater. Chem. Phys.* **2003**, *78*, 746–750. [[CrossRef](#)]
64. Wang, W.C.; Tian, Y.T.; Li, K.; Lu, E.Y.; Gong, D.S.; Li, X.J. Capacitive humidity-sensing properties of Zn₂SiO₄ film grown on silicon nanoporous pillar array. *Appl. Surf. Sci.* **2013**, *273*, 372–376. [[CrossRef](#)]
65. Tripathy, A.; Pramanik, S.; Manna, A.; Bhuyan, S.; Azrin Shah, N.F.; Radzi, Z.; Abu Osman, N. Design and Development for Capacitive Humidity Sensor Applications of Lead-Free Ca, Mg, Fe, Ti-Oxides-Based Electro-Ceramics with Improved Sensing Properties via Physisorption. *Sensors* **2016**, *16*, 1135. [[CrossRef](#)] [[PubMed](#)]
66. He, P.; Brent, J.R.; Ding, H.; Yang, J.; Lewis, D.J.; O'Brien, P.; Derby, B. Fully printed high performance humidity sensors based on two-dimensional materials. *Nanoscale* **2018**, *10*, 5599–5606. [[CrossRef](#)] [[PubMed](#)]
67. Samaeifar, F.; Azadinia, M.; Ghorbani, A.; Chen, J.; Gratuze, M.; Ketabi, M.; Izquierdo, R.; Aziz, H. Highly sensitive and fully printable humidity sensor on a flexible substrate based on a zinc oxide and polyethylenimine composite. *Flex. Print. Electron.* **2022**, *7*, 034003. [[CrossRef](#)]
68. Zhang, D.; Sun, Y.; Li, P.; Zhang, Y. Facile Fabrication of MoS₂-Modified SnO₂ Hybrid Nanocomposite for Ultrasensitive Humidity Sensing. *ACS Appl. Mater. Interfaces* **2016**, *8*, 14142–14149. [[CrossRef](#)] [[PubMed](#)]
69. Wang, J.; Zhao, B.; Wang, C. TiO₂/KNbO₃ nanocomposite for enhanced humidity sensing performance. *Sens. Actuators Phys.* **2023**, *349*, 114057. [[CrossRef](#)]
70. Cui, Y.; Zhu, S.G.; Zhang, L.X.; Shao, H.; Zheng, J.; Yin, Y.Y.; Bie, L.J. MOF-derived high-entropy oxides (YTbDyErYb)₂O₃ for boosted non-contact physiological humidity monitoring induced by oxygen vacancy modulation. *J. Alloys Compd.* **2024**, *1005*, 176005. [[CrossRef](#)]
71. Wu, Z.; Yang, J.; Sun, X.; Wu, Y.; Wang, L.; Meng, G.; Kuang, D.; Guo, X.; Qu, W.; Du, B.; et al. An excellent impedance-type humidity sensor based on halide perovskite CsPbBr₃ nanoparticles for human respiration monitoring. *Sens. Actuators B Chem.* **2021**, *337*, 129772. [[CrossRef](#)]
72. Wu, Z.; Sun, X.; Guo, X.; Ding, Y.; Ou, Y.; Yang, H.; Chen, Y.; Hu, Y.; Kuang, D.; Zhao, C.; et al. Development of a rGO-BiVO₄ Heterojunction Humidity Sensor with Boosted Performance. *ACS Appl. Mater. Interfaces* **2021**, *13*, 27188–27199. [[CrossRef](#)] [[PubMed](#)]
73. Dwiputra, M.A.; Fadhila, F.; Imawan, C.; Fauzia, V. The enhanced performance of capacitive-type humidity sensors based on ZnO nanorods/WS₂ nanosheets heterostructure. *Sens. Actuators B Chem.* **2020**, *310*, 127810. [[CrossRef](#)]
74. Alemayehu, B.; Shin, E.; Vasilyev, V.; Subramanyam, G. Synthesis and Characterization of Indium-Doped SnO₂-Based Impedance Spectroscopy Sensor for Real-Time Humidity Sensing Applications. *Crystals* **2024**, *14*, 82. [[CrossRef](#)]
75. Yu, X.; Chen, X.; Ding, X.; Chen, X.; Yu, X.; Zhao, X. High-sensitivity and low-hysteresis humidity sensor based on hydrothermally reduced graphene oxide/nanodiamond. *Sens. Actuators B Chem.* **2019**, *283*, 761–768. [[CrossRef](#)]

Disclaimer/Publisher's Note: The statements, opinions and data contained in all publications are solely those of the individual author(s) and contributor(s) and not of MDPI and/or the editor(s). MDPI and/or the editor(s) disclaim responsibility for any injury to people or property resulting from any ideas, methods, instructions or products referred to in the content.