



ECO FRIENDLY FABRICATION OF CUO/PVP THIN FILM HUMIDITY SENSORS VIA OLIVE LEAF ASSISTED GREEN SYNTHESIS AND ULTRASONIC SPRAY DEPOSITION

Shahed A. Dheyab

Department of Physics, College of Education for Pure Sciences, Tikrit University, Tikrit, IRAQ

Shahed.Ahmed@Tu.Edu.Iq

Abstract.

This work is a low-cost and sustainable new strategy for the green synthesis of CuO/poly (vinylpyrrolidone) (PVP) thin film humidity sensors based on OLE-derived copper oxide, namely CuO nanoparticle. Ultrasonic spray coating of the sensing films on gold interdigitated electrodes (IDEs) This work investigates the effect of calcination temperature (200 and 300°C) and polyvinylpyrrolidone (PVP) concentration (0.10 and 0.50 wt.% PVP-to-oxides mass ratio ATONY) on phase formation, morphological characteristics as well dispersion stability ontario casino lottery winner This study examined effect (up to 50%) on structural, morphological and sensing properties of the material. X-ray diffraction confirmed the formation of phase-pure monoclinic CuO with enhanced crystallinity at higher calcination temperature, while FESEM analysis revealed porous and interconnected nanostructures favorable for water adsorption and charge transport. The sensors exhibited a typical resistive humidity sensing behavior, with a continuous decrease in resistance over the 11-85% relative humidity (RH) range and linear I-V characteristics indicating good ohmic contact. S2 (200 °C, 0.50 wt. % PVP) showed good sensing performance, with a sensitivity of 330%, short response and recovery times (10 s/12 s). The sensors also showed impressive repeatability (RSD < 2.3%, among the two cycles), low hysteresis (< 4%), and stable long-term performance over a period of up to thirty days, showing promise for application as body motion monitors.

Keywords: copper oxide (CuO), Green synthesis, Olive leaf extract, Polyvinylpyrrolidone, Ultrasonic spray deposition, Humidity sensor.

Introduction

It is very important for humidification monitoring throughout a wide range of fields such as environmental control, healthcare, food storage and preservation, industrial processing research in agriculture pattern recognition micro-controllers smart electronic systems. As a result of the large surface area, adjustable electrical properties and compatibility with high-throughput fabrication processes [1,2], nanostructured metal oxide semiconductors are extensively studied as humidity sensors to meet growing demand for economical, ultra-sensitive and environmentally friendly

devices.

Of various oxide semiconductors, copper oxide (CuO) has received great attention as a p-type semiconductor because of its narrow band gap and excellent chemical stability along with cost if it obtained from commoditised resources due to high abundance and strong interaction with water molecules.

Since the sensitivity of CuO is strongly correlated with its particle size, surface morphology, porosity and defect concentration all these parameters can be modified through controllable synthesis routes. Nanostructured copper oxide (CuO) especially has a great potential for excellent adsorption sites and charge transfer pathways, which are required in room-temperature humidity sensors [3,4].

In recent years, green synthesis methods have appeared as a practical substitution for standard chemical approaches to nanoparticle manufacture. Additionally, naturally occurring polyphenols, flavonoids and reducing agents contained in plant extracts that reduce/stabilize/control NP growth without the use of toxic chemicals. Both of them are the most abundant bioactive components in Olive leaf extract (OLE) with eco-friendly synthesis and surface functionality improvement, stability enhancement. [5].

Concomitantly, controlled surface assembly of large nano-graphenes and CMOS-compatible processing technologies for ultrathin Si layers demonstrated the relevance of fabrication conditions to preserve structural order while modulating nanoscale properties. Ultrasonic spray deposition has been steadily gaining attention over the past decade as a feasible large-area, low-cost, scale-up method for channeling and uniformly depositing liquid suspensions into thin films. To prepare porous sensing layers that meet the aforementioned mechanical integrity, zwitterionic hydrophilicity and tunable electrical properties, these thin films containing rod-like nanofillers can be combined with various polymeric binders such as polyvinylpyrrolidone (PVP). The synergistic interaction of CuO nanoparticles and the hydrophilic polymeric PVP matrix prepares for efficient moisture sensing due to strengthened adsorption-desorption processes [6].

While there are many studies available regarding CuO-based humidity sensors, few of them focus on the use of green synthesized CuO nanoparticles obtained from olive leaf extract into ultrasonic spray-deposited cupric oxide (CuO) /polyvinylpyrrolidone (PVP) films onto interdigitated electrode (IDE). Additionally, almost all aspects (structural, morphological and humidity sensing properties) of the individual influence calcination temperature as well polymer concentration is still not explored completely. [7].

Hence, this work presents an eco-friendly and scalable method for the fabrication of CuO/PVP thin film humidity sensors using green synthesized CuO nanoparticles by ultrasonic spray coating process. The crystalline structure, morphology and electrical properties including sensitivity, response time and recovery behavior with respect to different calcination temperatures ranging between 500°-800°C as well as the various PVP concentrations are studied in this work. The proposed methodology presents a new potential avenue toward fabrication of eco-friendly, low-cost and high-performance humidity sensors that could be used in next-generation sensing technologies especially for resource-limited and developing economy settings.

Materials and Methods

1. Preparation of Olive Leaf Extract (OLE)

Six-week-old fresh olive leaves were washed with deionized (DI) water, oven-dried at 60 °C for a period of 36 h and subsequently pulverised into powder. Aqueous extract was generated by mixing 10 g of leaf powder with 100 mL DI water followed by exposure to heat at a temperature of: ~80 °C for 30 min under magnetic stirring. The extract was cooled, filtered through cotton cloth and a 0.45 µm PTFE membrane (Whatman), then stored at 4 °C until use.

2. Green Synthesis of CuO Nanoparticles

A 0.05 M copper nitrate solution was prepared by dissolving $\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$ in DI water. Ten milliliters of OLE were added to 90 mL of the copper solution and stirred at 65 °C. The pH was adjusted to 10.5 using 1.0 M NaOH. After 60 min of reaction, the resulting precipitate was collected by centrifugation (9000 rpm, 12 min), washed with DI water and ethanol, and dried at 60 °C for 12 h. The powder was calcined in air at either 200 °C or 300 °C for 2 h to obtain crystalline CuO nanoparticles.

3. Preparation of CuO/PVP Ink

CuO nanoparticles were dispersed in a DI water/ethanol mixture (1:1 v/v) at a concentration of 1 mg mL⁻¹. Polyvinylpyrrolidone (PVP, K30) was added at concentrations of 0.10 and 0.50 wt%. The suspension was ultrasonicated (40 kHz, 100 W) for 5 min and mechanically stirred for 30 min. Before deposition, the ink was filtered through a 0.45 µm PTFE membrane.

4. IDE Substrate Preparation

Glass substrates equipped with Au interdigitated electrodes (IDEs) having a finger width and spacing of 100 µm and an active area of 10 × 10 mm² were cleaned sequentially with acetone, isopropanol, and DI water, followed by nitrogen drying and oxygen plasma treatment for 2 min.

5. Ultrasonic Spray Deposition

CuO/PVP films were deposited using an ultrasonic spray system operating at 120 kHz. Deposition parameters were fixed at a substrate temperature of 80 °C, ink feed rate of 0.40 mL min⁻¹, N₂ flow rate of 1.5 L min⁻¹, nozzle to substrate distance of 10 cm, scan speed of 50 mm s⁻¹, and 10 spray passes. The deposited films were dried at 80 °C for 15 min and annealed at 140 °C for 45 min, producing porous films with an average thickness of approximately 150 nm.

6. Sample Design

Four samples were prepared according to a 2 × 2 factorial design by varying calcination temperature and PVP concentration, as listed in Table 1.

Sample	Calcination Temperature (°C)	PVP Concentration (%)
S1	200	0.10
S2	200	0.50
S3	300	0.10
S4	300	0.50

7 Characterization and Humidity Sensing Measurements

The X-ray diffraction (XRD) analysis of the prepared films was carried out to understand their crystal structure and scanning electron microscopy (SEM) on a field emission environment for surface morphology and microstructure evaluation. To study humidity sensing performance, the electrical resistance of films was monitored at different relative humidities (RH) using IDE platform. For electrical conduction behavior of the sensing layers, current-voltage (I-V) characteristics measurements were performed. Sensitivity, response time and recovery time were obtained from

R-t curves during cyclic humidity exposure. Humidity Sensitivity was computed by:

$$S (\%) = [(R_{dry} - RRH) / R_{dry}] \times 100 \quad (1)$$

where R_{dry} is the resistance at low humidity and RRH is the resistance measured at a specific relative humidity level.

Results

1. X-ray diffraction (XRD)

Figure 1 presents the X-ray diffraction (XRD) patterns of samples S1 to S4. Distinct diffraction peaks are observed at approximately $2\theta = 32.5^\circ, 35.5^\circ, 38.7^\circ, 48.7^\circ, 53.5^\circ, 58.3^\circ, 61.5^\circ, 66.2^\circ, 68.1^\circ, 72.4^\circ$, and 75.1° , which correspond to the crystallographic planes (110), (-111), (111), (-202), (020), (202), (-113), (022), (220), (311), and (004), respectively. All the peaks observed match closely with monoclinic tenorite phase of CuO (JCPDS card No. 45-0937). Secondary peaks due to the presence of secondary phases such as Cu₂O or Cu(OH)₂ are not detected, thus verifying that phase-pure CuO nanoparticles form under those synthesis conditions.

With the calcination temperature from 200 °C to 300 °C (Samples S1/S3 vs. S2/S4), however, there is a systematic increase in peak intensity and sharper reflections which implied improved crystallinity combined with moderate grain growth processes. In contrast, the concentration variation adjusted by PVP (0.10 wt. % vs. 0.50 wt. %). The variation between 0,5% does not affect significantly on peaks positions neither their relative intensity ratios. This finding affirms PVP polymer only acts as a stabilizing and dispersing agent in films, without penetrating CuO crystal lattices or distorting the structure [8,9].

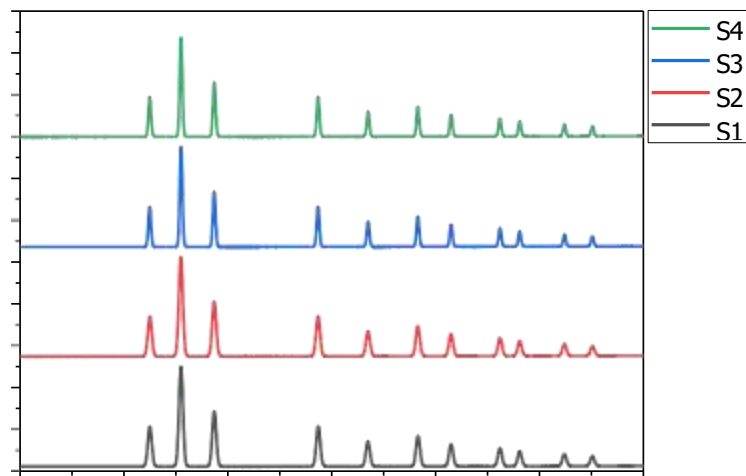


Figure 1. XRD patterns of CuO/PVP thin films (S1-S4).

2. FESEM Analysis

Figure 2 shows FESEM image of CuO/PVP thin films for the samples S1 to S4. All samples possess continuous and porous surface morphology formed by interconnected nanograins, which provides larger than in the case of dense film active area for adsorbed water molecules leading to good humidity sensing properties. Analysis at a lower calcination temperature (200 °C): For samples S1 and S2, the films show smaller grains with many bright agglomerates dispersed randomly within the grain. In contrast, the samples S3 and S4 (calcined at 300 °C) show a denser structure with

better-organized nanostructures suggesting that they have undergone much higher thermal energy which promotes crystallization.

Effect of polymer binder: The concentration of PVP is increased from 0.10 to 0.50 wt. The % provides more uniform films and a homogeneous dissemination of CuO nanoparticles across the substrate. The changes in morphology can be attributed to this PVP action as dispersing and stabilizing agent that prevents the agglomeration of nanoparticles while promoting porous net work formation with good adherence [10,11].

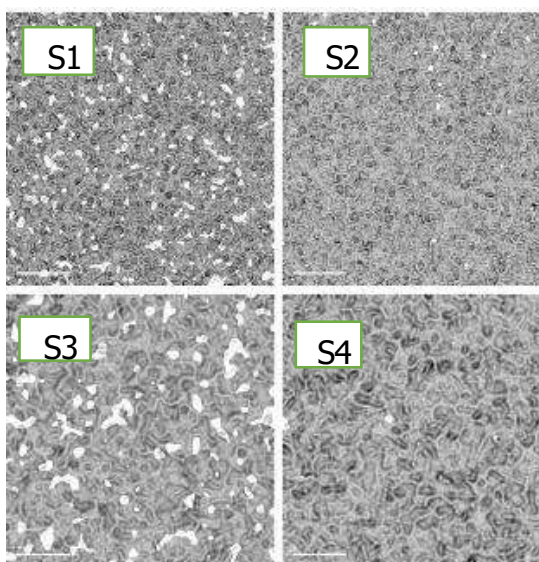


Figure 2. FESEM images of CuO/PVP thin films.

3. Current-Voltage (I-V) Characteristics

The current-voltage (I-V) characteristics of CuO sensing films, S1-S4 were measured in the voltage range from -2 to + 2 V and are displayed in Fig. 3, all obtained I-V curves show near-linear symmetric I-V behavior suggesting ohmic contact between the copper oxide film and gold IDE electrodes. The measured current values of S1, S2, S3 and S4 in the region of +2 V can be observed to be about 20×10^{-3} A, 17×10^{-3} A and 15×10^{-3} A for each value respectively. The microstructure and charge transport of CuO sensing layer can be altered due to differences in calcination temperature as well as PVP concentration, which is reflected by the gradual drop of current from S1 to S4 [12].

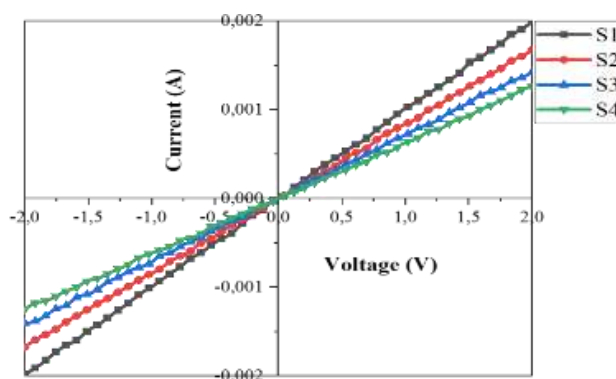


Figure 3. I-V characteristics of CuO/PVP thin films (S1-S4)

4. Resistance and Sensitivity Response

Thus, the resistance response of CuO sensing films (S1-S4) as a function of relative humidity over 11-85% RH is presented in Figure 4(a). As anticipated for resistive humidity sensors, the electrical resistance is seen to fall continuously with increasing RH for all samples. Such decrease is due to the adsorption of water molecules on CuO surface, which promotes protons hopping and therefore increases charge transport in sensing layer. The S2 film shows the lower response as also its low resistance up to 70% whilst, for all tested films and humidity content studied in this work while, S3 presents similar behaviour with higher resistances over the entire range of RH values, indicating a lowest modulus.

The humidity sensitivity (S1-S4) of the CuO sensors measured by a RH range from 11 to 85% is shown in Figure. The sensitivity values obtained are consistent with the variation in resistance and lie within the ranges of acceptable values reported for metal oxide humidity sensors. In particular, the resistance of S2 decreased from $\sim 9 \times 10^5 \Omega$ to $\sim 2.1 \times 10^5 \Omega$ when depicted by RH as shown in Fig. S7(a), where this value is respectively at relative humidity (11%RH and 85%), making it possess a sensitivity of about $\sim 330 \%$, which can be regarded reasonable for CuO based sensors [13, 14]. The increase of resistance with humidity for S2 is shown to be more sensitive and thus provides a higher response, while the change in S3 is weak compared to others.

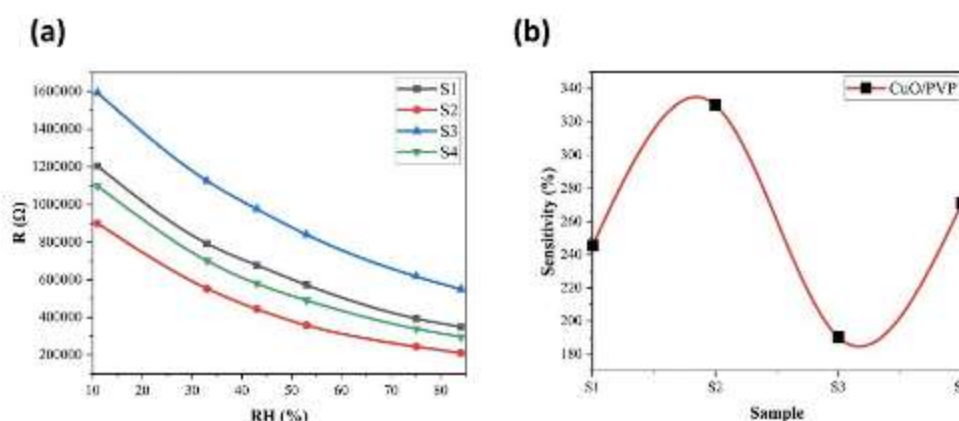


Figure 4. Resistance and sensitivity response of CuO/PVP thin film: (a) Resistance variation as a function of relative humidity, (b) Sensitivity.

5. Repeatability Analysis and Long-Term Stability

Repeatability (Figure 5(a)) was assessed over 30 cycles at 75 % RH. The responses from all samples are stable and reproducible with RSD values $< 2.3\%$ in the presence of 10laM cell lysate, which indicates high operational reproducibility. The response of S2 is the most stable with less variations, corroborating that CuO/PVP sensing layer has more stability. The excellent performance was ascribed to the robust nature of composite and reversible water adsorption-desorption, which avoids particle aggregation, keeping stable active sites by PVP.

For the long-term stability, all only show slight response variation after 30 days tests (Figure5(b)), where S2 is the most stable. Notably, no degradation was evident which means that the sensing films keep their structure. This stability results from the CuO-PVP synergy, as polymer alone improves water adsorption but also plays a stabilising role. The ultrasonic film coating technique

additionally enhances durability through the formation of uniformity and adherence. These results validate the relevance of the proposed sensors for long-terms environmental monitoring.

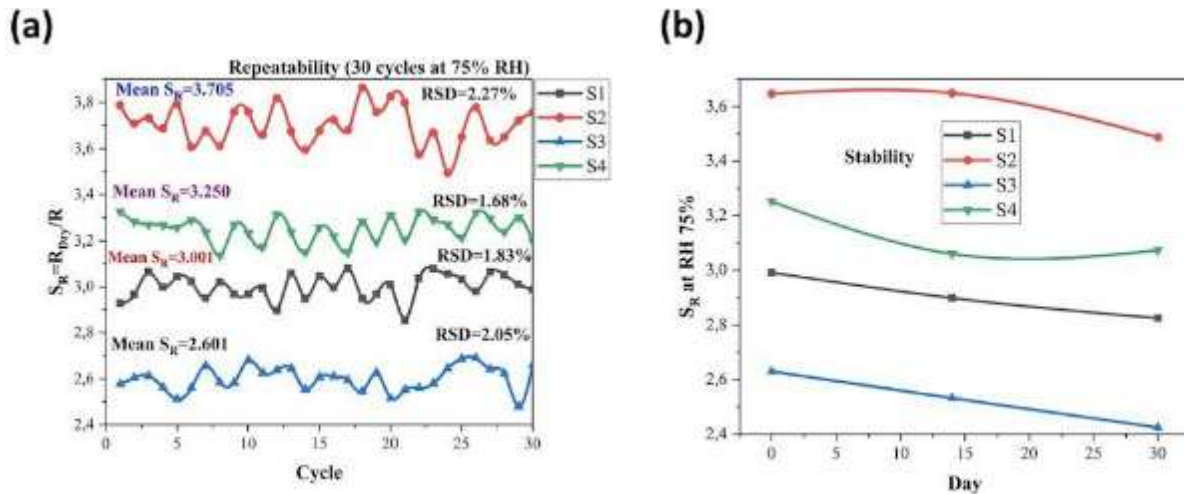


Figure 5. (a) Repeatability of CuO/PVP humidity sensors over 30 cycles at 75 % RH; (b) Long-term stability of CuO/PVP humidity sensors over 30 days.

6. Hysteresis Behavior and Response and Recovery times

Figure 6 (a) shows the hysteresis behavior during adsorption-desorption measurements. Over the span of humidity examined, all sensors demonstrate comparatively low hysteresis ratio ($< 4\%$) values that signify reversible sensing behavior. Hysteresis is caused by the differences in pathways going through adsorption and exit (via desorption) from porous material, which can also be explained with incomplete desorption of water molecules or trapping within. The small hysteresis reported in this investigation indicates that the binding energy between water molecules and the sensing surface is weak enough to allow easy desorption when humidity declines.

S2 prepared specimen exhibits the lowest hysteresis which can be attributed to microstructure and hydrophilicity being optimized. Water molecules can diffuse rapidly through the porous networks and PVP allows uniform distribution of adsorption sites, which reduces localized trapping effects. Lower hysteresis is one crucial parameter for reliable sensing, providing the same response after multiple bulk humidity cycles. These results confirmed that all of these stable and reliable humidity sensing applications can be successfully based on the above-fabricated CuO/PVP films.

Fig. The time response and recovery of CuO humidity sensors (S1-S4) is shown in Fig. 6(b). The recorded response/recovery times for S1 are 14/18 s, and 10/12 s for S2 with the same duration concerning responses from other sensors; here durations were only investigated using two distinct RSUs located in opposite ends of each road segment. S2 shows the dynamic system with fastest response of all while S3 has slowest rise and recover time among them. These are consistent with the sensitivity results (S2 also responded humbly to humidity).

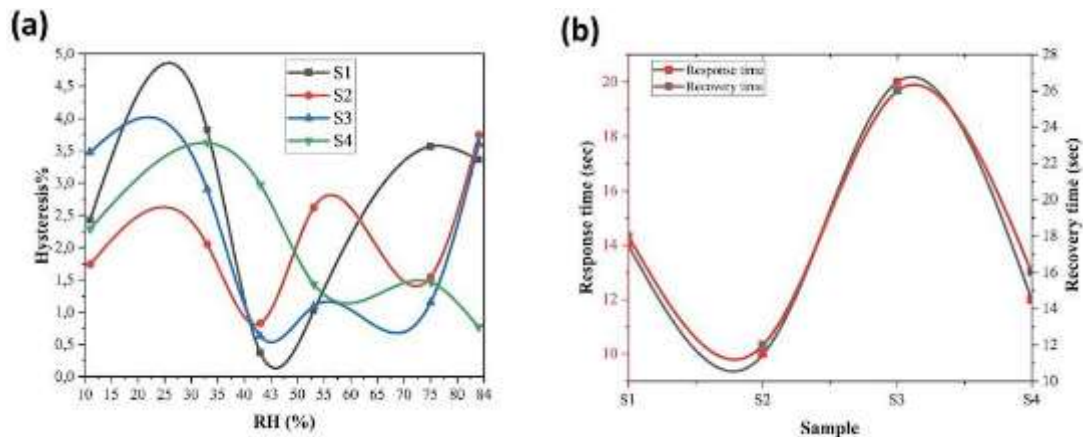


Figure 6. (a) Hysteresis behavior of CuO/PVP humidity sensors during adsorption and desorption cycles; (b) Response and recovery times of CuO/PVP humidity sensors.

7. Humidity sensing mechanism

CuO/PVP films have a humidity sensing mechanism based on surface adsorption processes and the development of charge transport pathways with increased relative humidity (RH) in ambient [87]. In lower RH, water molecules are predominantly chemisorbed onto the surface of CuO through interaction with oxygen species generate hydroxyl groups. In such condition, as CuO is an intrinsic semiconductor (p-type) its charge transport properties govern the conductivity which components to high resistive [15].

The water molecules are physically adsorbed and contacted continuous layers to sensing surface with gradually increasing RH as demonstrated in Fig. As a matter of fact, proton hopping among these layers facilitates charge transport through them resulting in improved conductivity and reduced resistance values [16]. The joint effect of calcination temperature and PVP content on the variance between samples with respect to their sensing performances, in particular that for good response (S2). In S2 (200 (In contrast to the high temperature recycling processes employed in previous studies, (e.g. 1050 °C, 1.00% PVP), which relies on greater mass loss and crystallization of organic species through dissolution thus producing both pore collapse and fragmentation scenarios for dense particulates the comparatively lower processing temperatures result in a microstructural identity preserved as less-densified particulates afforded primarily by thermal energy transfer prior to melt-dependent restructuring with attendant particle coalescence widely experienced at higher processing levels [17]. And this encouragement of early formation of a contiguous water monolayer speeds reproducible proton transport [15].

For samples calcined at a higher temperature (300 °C), the grains grow larger and their structure densifies, which decreases the accessible surface area for adsorption. Moreover, decreased PVP contents also lead to lower density of hydrophilic sites which provides weaker water uptake and conduction pathways that are less efficient [16] Thus, through the synergistic action of surface activity and structural accessibility, an optimal humidity activity is achieved by S2.

8. Comparison with previously reported CuO based humidity sensors

Table 2, Comparative summary of humidity sensing performance of different CuO based nanostructures reported in the literature along with present work. Thermally decomposed duo of the mesoporous CuO structures obtains a very fast response time (1 s), but no continuous reported hysteresis and recovery value [18]. Biosynthesized CuO NP response and recovery times of 22 s and 31 s along with a sensing range were found to be between 7-97% RH [19]. The CuO nanowires were prepared by thermal annealing, and they show symmetric but lower speed of response dynamics with both time spent for the sensing signal rise to 256 s corresponding to RT. [20].

On the other hand, CuO nanosheets deposited by spin-spray show faster response/recovery (2.1/2.8 s) and low hysteresis of 4% [21]. Thin films of CuO were prepared from the SILAR method also, but slower kinetics (130/320 s) and a hysteresis of 5% have been via [22]. One example of this is CuO/PVP composite polymerised previously, which have a response and recovery time being 35 s and 12 s respectively [23] along with extremely low hysteresis value known as well i.e.

In comparison to these reports, the current CuO/PVP-based sensor (S2) fabricated by ultrasonic spray deposition exhibits a good combination of quickly response (10 s), recovery time (12 s), low hysteresis (<4%) and wide working range (11-85% RH). The acquired performance is comparable to or better than that of most previous papers on CuO-based humidity sensors, especially the dynamic response and reversibility.

Table 2. Comparison with previously reported CuO based humidity sensors

Material	Structure / Dopant	Method	RH Range (%RH)	Response (s)	Recovery (s)	Hysteresis	Ref.
CuO mesoporous	—	Thermal decomposition	33-90	~1	11	—	[18]
CuO nanoparticles	—	Biosynthesis	7-97	22	31	—	[19]
CuO nanowires	—	Thermal annealing	20-80	~98	~98	—	[20]
CuO nanosheets	—	Spin-spray	20-90	2.1	2.8	4%	[21]
CuO thin film	—	SILAR	20-80	130	320	5%	[22]
CuO/PVP	Polymer	Polymerization	25-95	35	12	2.1%	[23]
CuO/PVP (This work)	Polymer	Ultrasonic spray	11-85	10	12	<4%	Present work

Conclusion

The present work reports the preparation of CuO/PVP thin film humidity sensors by combining olive leaf extract-assisted green synthesis with ultrasonic spray deposition. XRD analysis showed that the monoclinic structure of CuO (tenorite) was formed, and with increasing calcination temperature crystallinity increased, but it didn't change crystal structure. Through FESEM images, porous hydrogen-bonded interconnected nanostructures were established that offer significant active surface area available for water adsorption. The I-V characteristics in electrical characterization were symmetric and linear, indicating the formation of stable ohmic contact between salt-sensitive films and IDE electrodes. All gazvone electrodes showed a decreasing tendency of the electrical resistance with respect to that relative humidity, indicating their feasibility for resistive type humid sensing. Strikingly, calcination temperature and PVP

concentration had also an essential role for two sensing performances. The Sub sentences of Directional Named Metallic Metal Data Elementative Compositions Of Investigated Samples Some Below-average Example Among The Names : Elements (200 °C, 0.50 wt.) PVP) was the best-performing ISR material overall (showing sensitivity mechanical stimulation by ~330%. S3 has the lowest sensitivity and slowest dynamic response (12 s) among S0, S2, and paired with 85% RH employed in pairing10 s..

It means that, along with the porous microstructure maintenance of S2 at lower transformation temperature, higher PVP mass fraction and more hydrophilic functional groups can be introduced equally coordinately into material. Besides proton hopping conduction that is necessary in hydration period, these surface active species also formulated a favorable state for water adsorption which would reflect itself during impedance stability test on EIS. The results demonstrate that the novel green synthesized CuO/PVP thin films are a promising candidate for cheap, renewable and high-quality humidity sensors. The suggested fabrication route itself demonstrates excellent scalability properties as well for potential usage in future smart environmental monitoring devices.

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