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Original Article

Humidity Sensor Based on Rice Husk-Derived Carbon Materials

Sensor de humedad basado en carbono derivado de cascarilla de arroz

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Spanish version

Abstract

Introduction: Carbon-based materials derived from agricultural waste, such as rice husk (RH), have attracted increasing attention due to their potential for sustainable applications, particularly in electronics, environmental sensing, and energy storage.
Objectives: This study aims to investigate the synthesis of carbon materials from RH through thermal decomposition at two different temperatures (900 °C and 1000 °C), and to evaluate their application in humidity sensor fabrication, focusing on the influence of carbonization temperature on their structural and functional properties.
Materials and Methods: Carbon materials were synthesized from RH via pyrolysis at 900 °C and 1000 °C. Their structural properties and degree of graphitization were characterized using X-ray diffraction (XRD) and Raman spectroscopy. The resulting materials were then incorporated into conductive inks using different binders and solvents to fabricate resistive humidity sensors. The electrical response of the sensors was evaluated under controlled relative humidity conditions.
Results: Structural analyses revealed that higher carbonization temperatures led to more crystalline structures and enhanced graphitization. The fabricated sensors exhibited varying electrical responses depending on the pyrolysis temperature, showing different resistance-relative humidity relationships.
Conclusions: Carbonization temperature significantly affects the structural and functional properties of RH-derived carbon materials. These findings highlight the potential of optimizing such materials for environmental sensing applications, particularly humidity monitoring, contributing to the development of sustainable solutions in flexible and printed electronics.

Keywords: carbon, pyrolysis, materials characterization, sensor.

Resumen

Introducción: Los materiales carbonosos derivados de residuos agrícolas, como la cascarilla de arroz (HR), han despertado un creciente interés debido a su potencial en aplicaciones sostenibles, especialmente en los campos de la electrónica, la detección ambiental y el almacenamiento de energía.
Objetivos: Este estudio tiene como objetivo investigar la síntesis de materiales carbonosos a partir de HR mediante descomposición térmica a distintas temperaturas (900 °C y 1000 °C), y evaluar su aplicación en la fabricación de sensores de humedad, analizando cómo la temperatura de carbonización afecta sus propiedades estructurales y funcionales.
Materiales y Métodos: Se sintetizaron materiales carbonosos a partir de HR mediante pirólisis a 900 °C y 1000 °C. Las propiedades estructurales y el grado de grafitización se caracterizaron mediante difracción de rayos X (DRX) y espectroscopia Raman. Posteriormente, los materiales obtenidos se integraron en tintas conductoras, empleando distintos aglutinantes y disolventes, para fabricar sensores resistivos. La respuesta eléctrica de estos sensores se evaluó en condiciones de humedad relativa controlada.
Resultados: Los análisis estructurales indicaron que una mayor temperatura de carbonización favorece la formación de estructuras más cristalinas y un mayor grado de grafitización. Los sensores fabricados mostraron diferentes respuestas eléctricas según la temperatura de pirólisis, evidenciando una relación variable entre la resistencia y la humedad relativa.
Conclusiones: La temperatura de carbonización tiene un impacto significativo en las propiedades estructurales y funcionales de los materiales carbonosos derivados de HR. Estos resultados demuestran el potencial de estos materiales para ser optimizados en aplicaciones de detección ambiental, específicamente en la monitorización de la humedad, contribuyendo al desarrollo de soluciones sostenibles en electrónica flexible e impresa.

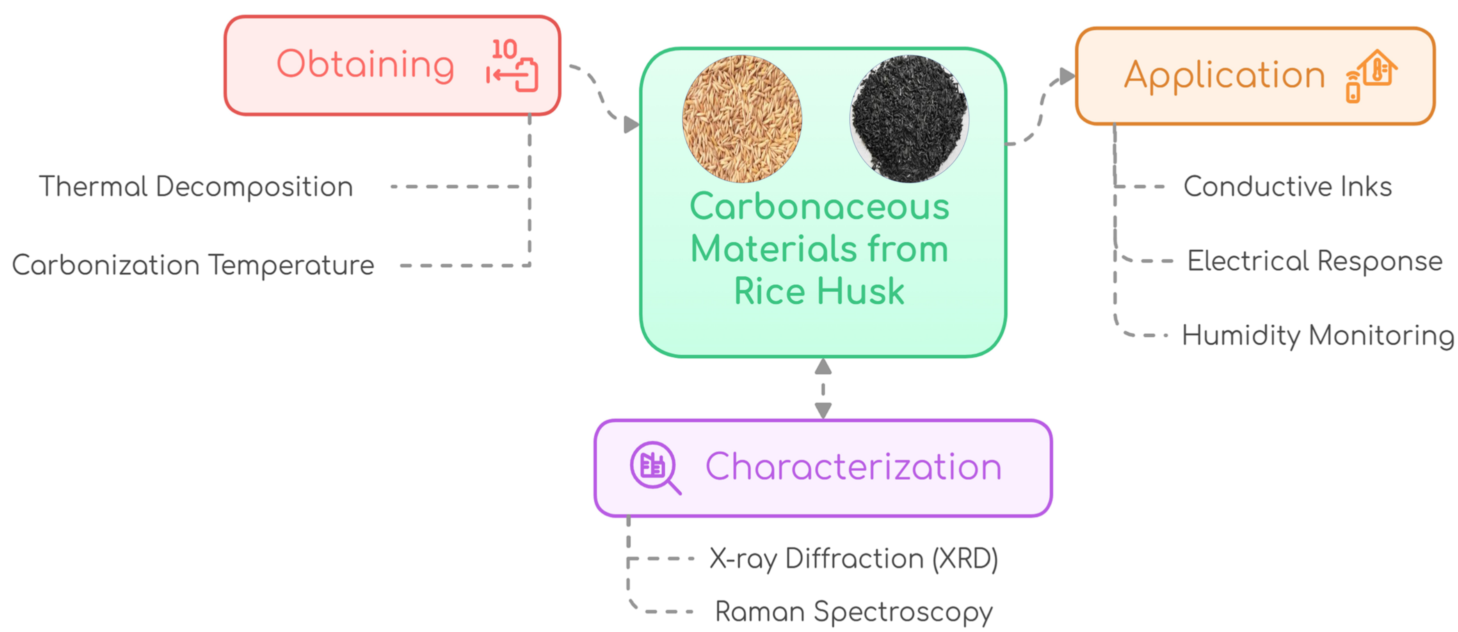
Palabras clave: carbono, pirólisis, caracterización de materiales, sensor

Contribution to the literature

Why was it conducted?
The study aimed to develop and characterize humidity sensors using carbonaceous materials derived from rice husk through pyrolysis. It emerged from a broader research project focused on exploring rice husk as a source of value-added byproducts. This work specifically investigated how carbonization temperature influences the structural, electrical, and sensing properties of the resulting materials. The approach promotes sustainable sensor development by valorizing agricultural waste.

What were the most relevant results? What do these results contribute?

The study demonstrated that carbon materials obtained from pyrolyzed rice husk showed graphitization, conductivity, and sensing performance. These properties were characterized by XRD and Raman Spectroscopy. With the obtained carbons, four inks were made using different binders, to obtain humidity sensor. Among the tested inks, those based on silicone and acetone provided better mechanical stability and consistent humidity responses, which exhibited a highly linear resistance increase with humidity. In contrast, inks with water-soluble binders showed poor adhesion. The research highlights the effectiveness of direct, one-step pyrolysis of rice husk, without chemical pretreatment, as a low-cost, sustainable method for developing high-performance humidity sensors, contributing to versatile solutions in flexible and printed electronics.



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Introduction

Carbonaceous materials derived from agricultural waste have garnered considerable attention in recent years due to their potential for sustainable applications in electronics, environmental sensing, and energy storage (1) (2). Among these, rice husk (RH), an abundant agricultural byproduct, is particularly appealing due to its high carbon content, high availability and the ability to be transformed into value-added products through thermal treatments (3) (4). These treatments, such as pyrolysis, enable the conversion of RH into carbonaceous materials with tailored structural and functional properties, such as electrical conductivity and thermal stability, suitable for various technological applications.

In this regard, the need for innovative, cost-effective, and environmentally friendly materials in the field of flexible and printed electronics has driven research into carbon-based conductive pastes and inks (5) (6). Conductive pastes, which are integral components in sensors, circuits, and other electronic devices, must exhibit high electrical conductivity, structural stability, and compatibility with various substrates (7).

In this respect, humidity sensors are essential in various industries, including agriculture, healthcare, and environmental monitoring. These sensors require materials that are not only sensitive to humidity changes but also robust under varying environmental conditions (8) (9). Carbon-based materials have shown promise in this regard due to their high surface area, functional group variability, and tunable electrical properties. In this regard, the utilization of agricultural waste such as RH for the development of carbon-based sensors represents a sustainable alternative with positive economic and environmental impacts as it decreases pollution by valorizing waste that would otherwise be burned or discarded (10).

However, achieving optimal performance in terms of sensitivity, reliability, and structural integrity, demands precise control over material synthesis, ink formulations and sensor fabrication processes (11). Building on this idea, in this study we report the synthesis of carbonaceous materials from RH using a thermal decomposition process and their subsequent utilization in fabricating humidity sensors. The synthesis process involved thermal treatments at two distinct temperatures (900°C and 1000°C) to investigate the influence of carbonization temperature on the structural and functional properties of the obtained materials. Comprehensive material characterization was conducted using X-ray diffraction (XRD) and Raman spectroscopy.

The fabricated sensors employed carbonaceous inks formulated with different binders and solvents to evaluate their electrical response. By integrating these inks into a copper interdigitated circuit, resistive sensors were developed and tested under controlled humidity conditions. The study explored the electrical behavior of the sensors, focusing on their resistance variations as a function of relative humidity. This research contributes to the growing field of sustainable electronics by demonstrating the potential of RH-derived carbonaceous materials in developing high-performance humidity sensors.

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Methodology

Synthesis of carbonaceous samples

The synthesis of organic material from rice husk (RH) was carried out using the thermal decomposition method, following the workflow illustrated in Figure 1 (12) (13). Subsequently, a cleaning and sieving process was performed before the thermal treatment to remove impurities and dust from the raw material. For the thermal treatment the RH was placed inside a tubular reactor. Then, a vacuum system was activated and then a nitrogen flow was injected. By doing so the oxygen was expelled from the reactor and an inert atmosphere was created inside the chamber. Afterwards, the carbonization process was initiated by controlling the temperature with a PID controller and data collection software. This allowed for graphical monitoring of the temperature behavior within the reactor. The carbonization process was carried out for an average of 2 hours after reaching thermal stability. Two types of samples were obtained, one at 900°C and other one obtained at 1000°C.

Once the process end, the sample was collected and manually grounded using an agate mortar followed by sieving process using an Adamas-Beta TYJS-0074 sieve. This procedure led to sample particles of 105 µm aproximatedly.

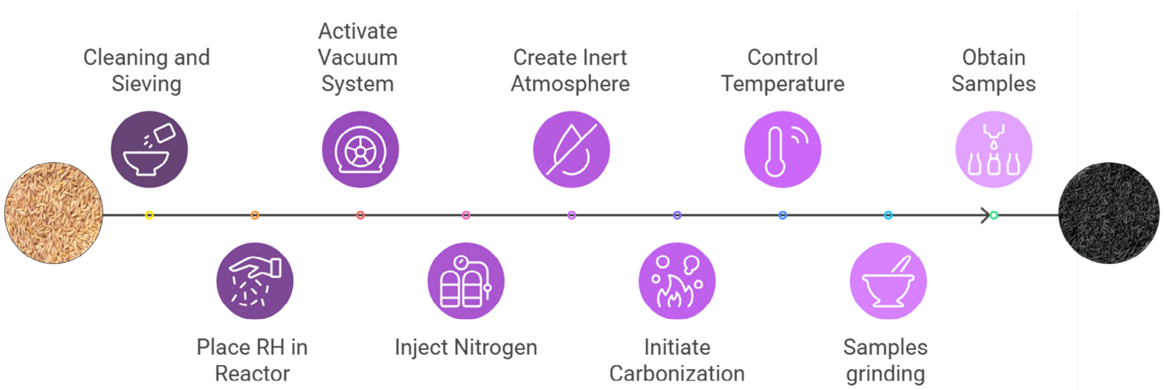

Figure 1. Schematic process of the carbonaceous sample preparation

Characterization of samples and sensor fabrication

X-ray diffraction (XRD) analysis was conducted using a PANalytical Empyrean Series II diffractometer equipped with Ni-filtered Cu Kα radiation. The measurements were taken with a step size of 0.02626° and a scanning speed of 0.11°/s, covering a 2θ range from 5° to 85°**.** Raman spectroscopy was performed at room temperature using a confocal Horiba Jobin Yvon Labram HR spectrometer. A HeNe laser with a 632 nm wavelength and an 800 mm focal length was used for excitation. All spectra were recorded under identical conditions within the 400–3500 cm^{-1} range. To create the sensor, a copper interdigitated circuit was printed on a flexible PCB, as shown in Figure 2a. This circuit was used as the platform to deposit the inks. The carbonaceous inks, used as the dielectric materials between the fingers of the circuit, were prepared by using distilled water, carboximethylcellulose (CMC), acetic silicone, isopropyl alcohol (99%), acetic acid, liquid silicone, and acetone (99%). These reactants were combined in different quantities in order to create four

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inks, as shown in Figure 2b. The electrical properties of the sensors were measured using a FLUKE 289 True RMS Multimeter.

b

Ink	Composition	Quantity (g)
1	Carbonaceous sample Binder (CMC) Distilled water	1 0.08 5
2	Carbonaceous sample Acetic Silicon Isopropyl Alcohol	1 1 2
3	Carbonaceous sample Acetic Silicon Acetic Acid	1 1 2
4	Carbonaceous sample Acetic Silicon Acetone	1 1 2

Figure 2. a) Copper interdigitated circuit and b) Composition of the inks

Sensors testing

For data acquisition, a closed-environment chamber was constructed, enabling the characterization of the sensors. The setup included an airtight container, a conventional vacuum pump, a humidifier (Mini-Nano Vaporizer), and a humidity sensor (Monitoring Traceable Hygrometer, MTH). Two holes were made in the container, one on each side. The nozzle of the humidifier was connected to one of these openings to introduce moisture into the chamber, while the other was connected to the vacuum pump hose to remove humidity. Then the fabricated sensors were placed side by side inside the chamber, allowing measurements to be taken using a multimeter for data collection and comparison under controlled conditions, as illustrated in the Figure 3. The humidity system was initiated within the chamber until the MTH indicated saturation. The vacuum pump was then activated to lower the humidity. Once the maximum allowed humidity was recorded, resistance data for the OGF-based sensors was collected as the humidity decreased, covering a range of 58% to 90%.

Figure 3. Experimental set-up for humidity test of the sensors

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Results and discussion

Characterization of the materials

Figure 4a show a comparison between X-ray diffraction (XRD) patterns of the samples. Both samples exhibited two peaks near 2θ = 25° and 45°, suggesting the existence of disordered carbon with a turbostratic arrangement. (14). In this type of structure, the carbon exhibits order within the graphene layers, but lack order between the layers. It was found that increasing the pyrolysis temperature resulted in more defined XRD peaks. So, the sample obtained at 1000°C exhibited XRD peaks around 26,5°, 35,7° and 57° which correspond to directions (002), (100) and (004) of graphite structure, respectively (15). This result suggests the presence of a graphite structure within the obtained materials, especially for the one obtained at 1000 °C (16). The other peaks were associated with the oxide structural phase (12).

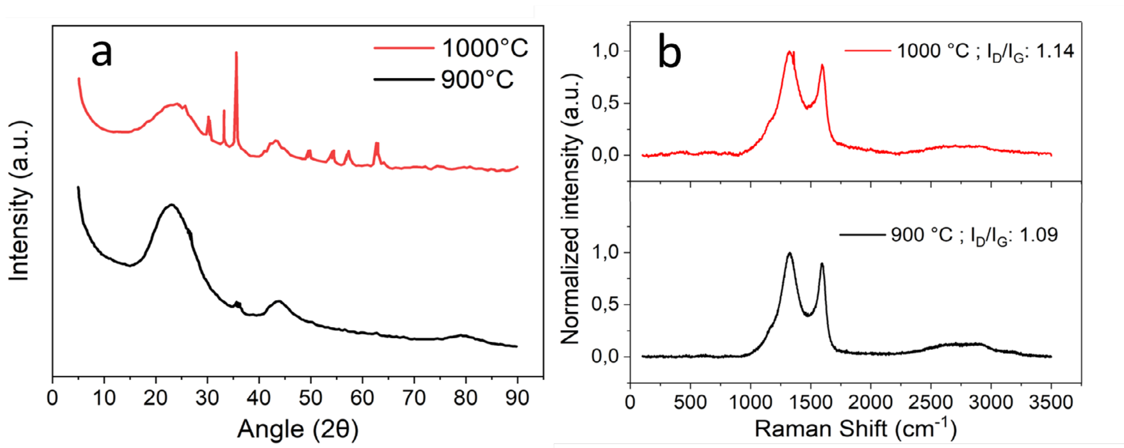

Figure 4. a) XRD patterns and b) Raman spectra, of the obtained samples

A Raman spectroscopy was carried out and the obtained results are shown in Fig. 4b. All materials exhibited similar vibrational characteristics. The spectral peaks corresponding to the D and G at 1328 and 1590, respectively. 2D bands appeared approximately at 2630 cm^{-1}. The D band is associated with defects, and its intensity is directly related to the number of sp3 atoms present on the material's surface (17) (18). On the other hand, the G band is caused by the vibrations of the sp^2 carbon atoms, representing graphitized carbon and is directly related to the crystallinity of the material. In this regard, the ratio of intensities (I_D/I_G) slightly increased with the pyrolysis temperature suggesting the presence of more edges and defects. These results suggest that while the material obtained at 1000°C exhibits better crystalline characteristics than that obtained at 900°C, it also presents a higher number of functional groups (sp^3) (12) (19).

Sensor Fabrication and Implementation Process

Once the materials were characterized, the inks were prepared. The ink 1 was prepared with CMC and distilled water and exhibited excellent consistency, demonstrating the excellent binding properties of CMC (20) (21). Both carbonaceous material, 900 and 1000°C, were well integrated in the ink which led to a homogeneous mixture, highly malleable, with a decent adhesion to the substrate. Figure 5 shows the ink 1 deposited on the substrate.

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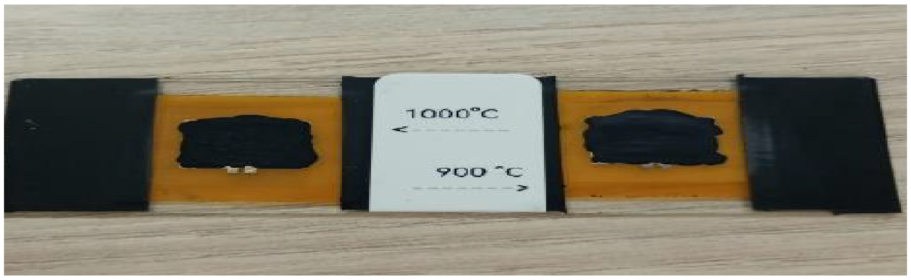

Figure 5. PCB coated with ink 1

Accordingly, the ink 2 made with acetic silicone and isopropyl alcohol, proved to be difficult to mix as they did not integrate properly, resulting in an inconsistent and unmanageable paste. The isopropyl alcohol high volatility caused rapid evaporation upon contact with the silicone, hindering the dispersion of the carbonaceous material.

Likewise, Ink 3 composed of acetic silicone and acetic acid dried almost instantly, forming a paste like the one observed with Ink 2. This behavior can be attributed to the role of acetic acid in facilitating the cross-linking process of acetic silicone (22), which promotes the rapid evaporation of byproducts. Finally, Ink 4 was prepared with liquid silicone and acetone. This ink dissolved quickly and allowed for proper homogeneity. When mixed with the carbonaceous materials, the ink retained its characteristics, enabling smooth application onto the PCBs, as depicted in Figure 6.

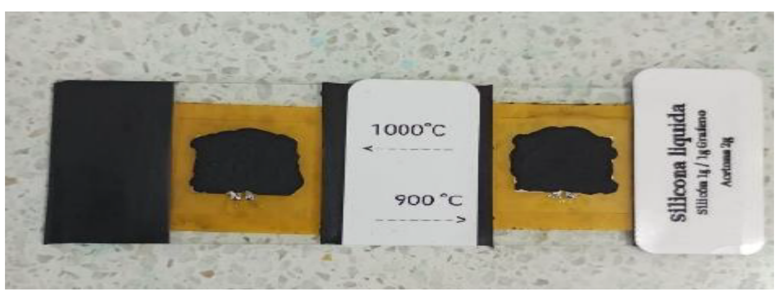

Figure 6. PCB coated with Ink 4

Based on these results, Ink 1 and Ink 4 were selected for sensor fabrication due to their superior homogeneity, ease of mixing, smooth application, and quick drying. So, four sensors were fabricated as follows: Sensors 1 and 2 were composed of distilled water, CMC and 900 and 1000 °C samples, respectively. Sensors 3 and 4 were composed of liquid silicone acetone and 900 and 1000 °C samples, respectively.

Electrical response assembled sensors

Using the forementioned inks, four sensors were fabricated, and their electrical properties were measured. Both capacitance and resistance tests measurements were conducted under identical environmental conditions. It was not possible to measure the capacitance because all four sensors reached a saturation point. Among the reasons for this behavior include: 1) The sensors may have reached their maximum charge storage capacity, rendering the multimeter unable to measure the resulting voltage and capacitance, 2) Environmental factors, such as humidity or temperature, could

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have influenced the electrical properties of the carbonaceous material causing saturation, and 3) Instrument limitations, such as indeterminate impedance in the multimeter, may have affected the sensor's response.

On the other hand, resistance measurements were successfully conducted, and the obtained results are shown in Table 1.

Table 1. Resistance values obtained from the sensors

Ink	Sensor	Composition	Resistance (Ω)
1	1	Water + CMC + 900 °C	87.2
	2	Water + CMC + 1000 °C	15.8
4	3	Silicone + Acetone + 900 °C	22.34
	4	Silicone + Acetone + 1000 °C	4.68

Notably, the lowest resistivity values for each type of sensors were achieved with the carbonaceous materials obtained at 1000°C, confirming the graphite-like structure of the material synthesized at this temperature (23) (24).

Based on the obtained resistance measurements, the decision was made to focus on resistive sensors due to the challenges in measuring the capacitive characteristics. So, the subsequent tests focused on the characterization of the resistive properties of the sensors.

Resistive properties of the sensors under humid environment

The sensors were characterized as humidity sensors based on their resistive properties. This application was chosen due to their reliable performance and structural compatibility with environmental sensing requirements. A closed environment system was implemented to monitor and record resistive variations in response to humidity exposure, as detailed in the Methodology section.

Accordingly, the resistance variation of the Sensor 1 with relative humidity, is shown in Figure 7a. The sensor 2 fabricated with the ink 1, detached immediately upon exposure to humidity, making it impossible to collect data, as depicted in Figure 7b. Since this sensor was fabricated using carbonaceous material obtained at 1000°C, the detachment was likely due to the hydrophobic nature of graphitic-like materials, which restricts the wettability of the carbonaceous component of the ink (25) (26).

For Sensor 1, a clear inverse linear relationship is observed between resistance and relative humidity, as evidenced by the decreasing resistance with increasing RH (Figure 7a). The linear fit, represented as a red line, indicated an excellent correlation and the high accuracy of the sensor in detecting changes in humidity. This linear behavior suggests that the fabricated sensor is highly sensitive to variations in humidity levels within the tested range. The high R^2 value further confirms the reliability of the sensor's response, making it a promising candidate for applications in humidity monitoring systems. As revealed by XRD, the carbonaceous samples exhibited some degree of graphitization, which show good electrical conductivity, promoting the flow of electrons through the sensor material. This conductivity ensures that the sensor can detect small changes in electrical resistance accurately as the humidity varies. Additionally, as explained by Raman spectroscopy the surface structure of the carbon obtained at 900 °C may possess functional groups that enhances its

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interaction with water molecules, causing measurable changes in resistance as the relative humidity shifts.

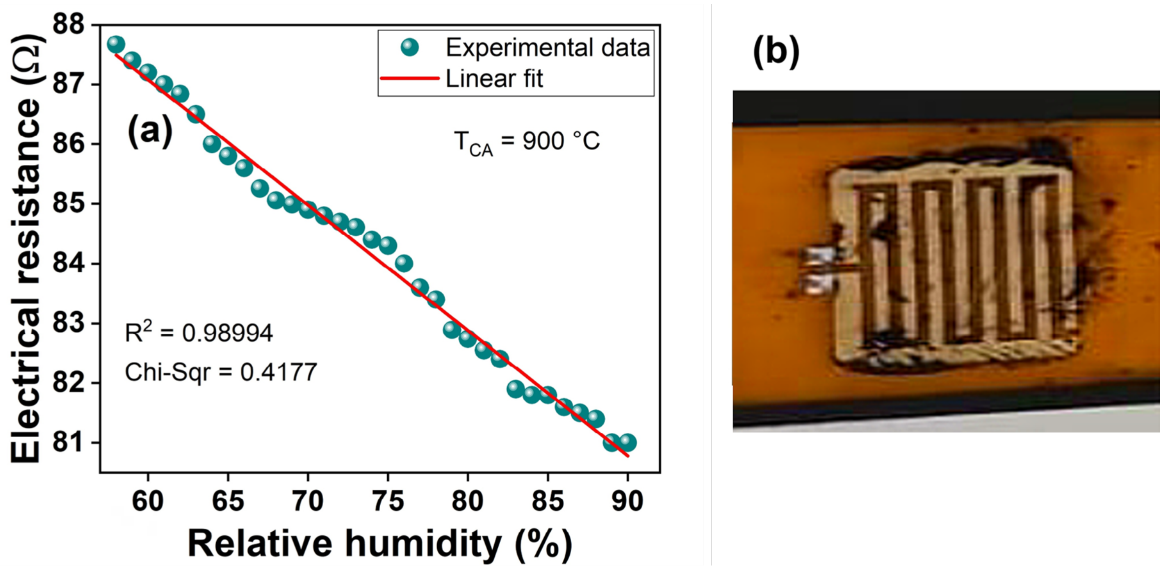

Figure 7. a) Resistance variation with relative humidity of the Sensor 1 and b) Sensor 2 detached from the PCB

Accordingly, the resistance response of Sensor 3 and 4 is shown in Figure 8. The Sensor 3 (Figure 8a) demonstrated good physical properties, as the ink remained intact during the test. This behavior is probably due to the excellent environmental resistance of silicone (27) (28). It was observed a nonlinear behavior, positively correlated relationship between electrical resistance and relative humidity, as resistance increases with higher humidity levels. So, a polynomial fit to the data was achieved, with a coefficient of determination of 0.9975.

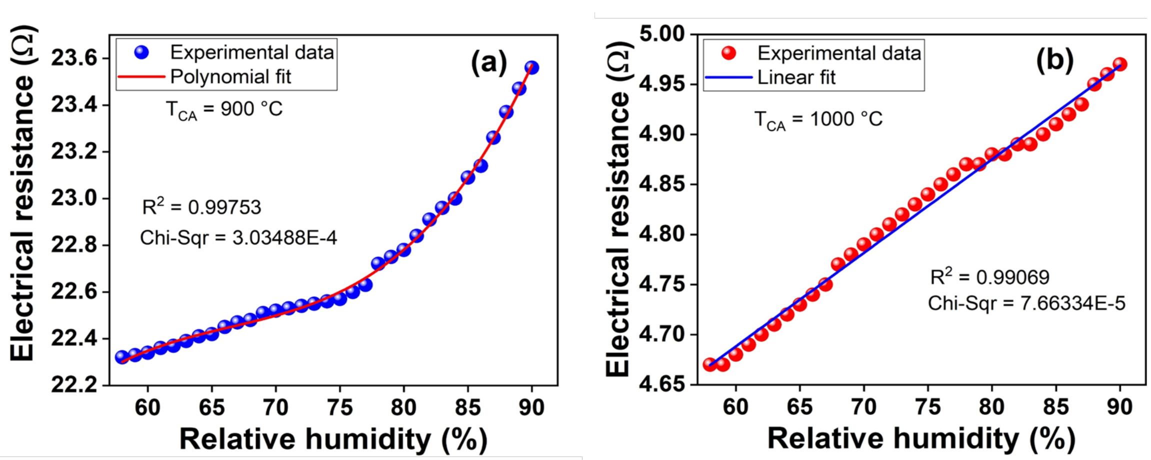

Figure 8. a) Resistance variation with relative humidity of the Sensor 3 and b) Sensor 4

This nonlinear response suggests that the material system's interaction with water molecules changes dynamically at different humidity levels. At lower humidity, the interaction between the sensor material and water molecules is less pronounced, leading to smaller changes in resistance.

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As the humidity increases, water molecules may accumulate on the sensor's surface, resulting in a saturation point that leads to a significant increase in resistance.

On the other hand, the data for Sensor 4 (Figure 8b) demonstrated a clear linear relationship between electrical resistance and relative humidity, with resistance increasing consistently as humidity levels rose. A linear fit with an R^2 value of 0.9906 was obtained, indicating the sensor's precision. Unlike Sensor 1, Sensor 4 exhibited excellent mechanical robustness and resistance to humidity, making it more suitable for practical applications. The observed linearity suggests that the interaction between the sensor material and water molecules occurs uniformly across the tested humidity range, leading to a systematic and predictable change in resistance.
The results obtained suggest that it is possible to develop a humidity sensor from the direct pyrolysis of rice husk, without any prior treatment. In this regard, it is important to note that, in general, sensors developed in other studies use rice husk derivatives that undergo a pretreatment process (29) (30) (31). Therefore, this work highlights the potential of carbon derived from the direct treatment of rice husk.

The carbonization temperature was found to play a pivotal role in the sensor's response characteristics. At 900 °C, the sensor exhibited a polynomial response, likely due to the material's lower conductivity and the reduced number of functional groups available for interaction with water molecules. In contrast, the sensor fabricated with carbon obtained at 1000 °C displayed a linear response, which can be attributed to the higher degree of graphitization (32) and an increased presence of functional groups on the carbon's surface. These features enhance the material's conductivity and its ability to interact consistently with water molecules, resulting in a stable and linear response. The differences in material properties between the two carbonization temperatures were corroborated by X-ray diffraction (XRD) and Raman spectroscopy analyses. These results highlight the critical influence of processing conditions on sensor performance and underscores the potential for tailoring material properties to specific sensing applications.

Conclusions

The carbonization temperature significantly influences the structural and functional properties of the synthesized carbonaceous materials, with samples obtained at 1000 °C exhibiting enhanced graphitization, better crystallinity, and a higher presence of functional groups, as confirmed by XRD and Raman spectroscopy analyses. These characteristics were critical for improving sensor performance, particularly in humidity sensing applications.

Among the tested inks, those formulated with distilled water and CMC (Ink 1) or liquid silicone and acetone (Ink 4) demonstrated superior homogeneity and ease of application, enabling the successful fabrication of four resistive sensors with robust physical and electrical properties. Nevertheless, the sensors fabricated with Ink 1 showed poor adhesion and detached rapidly from the substrate which showed the limitation of CMC binder.

Sensors fabricated with carbonaceous materials obtained at 1000 °C outperformed their counterparts, with Sensor 4 exhibiting a highly linear relationship between resistance and relative humidity, indicating consistent interactions between water molecules and the sensor surface across the tested humidity range. The response of the sensors varied depending on the

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carbonization temperature, as materials obtained at 900 °C showed a nonlinear response due to lower conductivity and fewer functional groups, while those obtained at 1000 °C displayed a linear response attributed to higher conductivity and higher number of functional grous on the surface. This study highlights the critical role of rice husk processing conditions in tailoring material properties to meet specific sensing requirements. Additionally, this study presented the limitations of the inks based on water-soluble binders as electrodes for humidity sensing. Finally, the research highlights the potential for optimizing carbonaceous materials obtained from rice husk as sensors for environmental testing, such as humidity monitoring in controlled environments.

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CRediT authorship contribution statement

Conceptualization - Ideas: J.R. Castro-Ladino. Data Curation: Santiago Mesa. Formal analysis: J.R. Castro-Ladino, Santiago Mesa. Acquisition of funding: Santiago Mesa, Dora A. Hoyos-Ayala. Investigation: J.R. Castro-Ladino. Methodology: J.R. Castro-Ladino. Project Management: Dora A. Hoyos-Ayala. Supervision: Dora A. Hoyos-Ayala. Validation: J.R. Castro-Ladino. Visualization - Preparation: Santiago Mesa. Writing - original draft - Preparation: J.R. Castro-Ladino, Santiago Mesa. Writing - revision and editing - Preparation: J.R. Castro-Ladino, Santiago Mesa, Dora A. Hoyos-Ayala.

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