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Design and Implementation of Leaf Wetness Sensor with different Graphene Oxide Concentration

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Prediction of early plant diseases is essential to reduce crop loss. Early disease prediction models have been investigated for this purpose, where data on leaf wetness duration (LWD) is one of the important elements. Leaf wetness sensors (LWS) are used to measure the leaf wetness duration (LWD). Here, graphene oxide (GO) is used as the sensing film in the sensor to detect the water molecules on the leaf canopy. LWS is fabricated on a flexible polyamide substrate using interdigitated electrodes. The fabricated GO LWS was tested in a lab condition. We exposed the entire sensing film to water molecules and observed that it responded by around 120000% for GO-3 sensor, 90000% for GO-2 sensor, and 37000% for GO-1 sensor at 1KHz compared to the air. The observed response time of the fabricated sensor is around 180 sec with a recovery time of about 10 sec. However, when exposed to temperatures ranging from 20 0C to 70 0C, the fabricated sensor shows only a 2.3% change in response.

Keywords: Leaf wetness sensor, Graphene oxide, Field evaluation, Plant disease.

1. Introduction

An enormous number of crops are lost due to plant disease, pests, and weeds, which reduces crop yield. So, the identification of early plant diseases becomes more important to avoid such graving situations for the agriculture industry. There are various methods available to do this job. Direct conventional lab-based methods, such as pathogenicity, physiological, and biochemical testing, can be used to identify plant disease (Fang et al., 2015; Martin et al., 2000) . This test has a good selectivity for disease diagnosis, but it takes a while and requires knowledge of plant pathology. Another method is an indirect method, where self-collected photos, hyperspectral images, and satellite data are used in indirect ways to identify plant disease (Martinelli et al., 2015; Wongsai et al., 2017; Nagasubramanian et al., 2021; Hornero et al., 2017; Abdulridha et al., 2018). Although these indirect methods provide accurate measurement, they necessitate a large amount of data and technical expertise to evaluate the images.

In today’s technology-driven era, Sensor-based technology can play a crucial role in early plant disease prediction models by forecasting plant diseases which improves crop growth by reducing the impact of diseases. There are various atmospheric factors that affect plant growth like temperature, humidity, soil temperature, soil moisture, wetness or moisture of the leaves, and many more. All these parameters are used in early disease detection models, water management models, and smart agriculture systems. The leaf wetness duration (LWD) is one of the important components in these models.

The germination of many fungal infections is caused by the presence of water molecules on the leaf canopy for an extended period of time and in environments with suitable climatic conditions such as temperature and humidity (Kumar et al., 2020; Huber et al., 1992) . The LWD measurement can be done by the Leaf wetness sensor. The leaf wetness sensor can be either artificial leaves or a sensor placed on top of actual leaves. The source of water molecules can be mist, rain, or sprinkler irrigation. The effectiveness of sensor-based methods to identify plant diseases has also been investigated by researchers (Hornero et al., 2017; Patle et al., 2021). There are the resistive and capacitive types of electronic leaf wetness sensors reported earlier. As the sensor is exposed to water molecules, the resistance of the sensor in resistive-based leaf wetness sensor (LWS) varies. Capacitive-based LWS, on the other hand, delivers a change in the dielectric medium due to the presence of water molecules leading to a change in the sensor capacitance. The resistive type sensor has a disadvantage over the capacitive type of sensor in performance with respect to diurnal temperature variation for in-situ applications. The capacitive type of sensor offers more stable performance than resistive-based LWS, capacitive-based LWS is frequently chosen for in-situ measurements (Patle et al., 2021; Patle et al., 2022).

The capacitive type of LWS is fabricated on flexible substrates like polyamide (Patle et al., 2021; Lu et al., 2020) and printed circuit board PCB (Hornero et al., 2017). Printed circuit boards (PCBs) are commonly utilized in the fabrication of LWS, including commercial LWS on the market (PHYTOS 31). GO based soil moisture sensor [Neema et al.,2024] has reported. However, these sensors' (PCB-made) weight and contact resistance are their biggest limitations when compared to their operating exposure and accuracy during in-situ measurements. Therefore, flexible capacitive sensors are one of the potential candidates for in-situ applications.

In addition to this, to achieve higher sensitivity, selectivity, and minimum diurnal temperature variation researchers have reported a 2D-nanomaterial-based flexible capacitive type leaf wetness sensor [Priyanka MoS2 Paper and Kamlesh GO Paper]. Researchers have implemented flexible capacitive-type leaf wetness sensors using GO (Graphene oxide) and MoS_2 (molybdenum disulfide) as a sensing film to achieve higher sensitivity. It was reported that GO is highly sensitive and has selectivity to water molecules, as well as has a negligible effect on diurnal temperature variations (Palaparthy et al., 2018). [Kamlesh GO paper] has reported that 10mg/100µL concentrated GO has been drop-casted on LWS for both lab and in-situ experimentation. So, there is a need to explore LWS further with respect to different GO concentrations.With this motivation in this work, we used three flexible LWS where IDEs fabricated on a flexible polyimide substrate with three different concentrations of GO as the sensing film to produce a highly sensitive, selective, and flexible LWS with minimal variance in diurnal temperature variations. Further, the sensitivity, response time, hysteresis, and temperature response of the sensor are also investigated.

2. Materials and methods

2.1. Synthesis of sensing material

In the LWS, Graphene oxide (GO) is used as a sensing material. Here, GO is synthesized from graphite powder using the modified Hummer's process (Al-Gaashani et al., 2019). In the first step, 1 gm of powdered graphite is added to a mixture of H2SO4 and NaNO3, which is then continuously stirred at 540 rpm. The above mixer is then gently filled with potassium permanganate (KMnO4) over the course of an hour. Exothermic in nature, the reaction is conducted in an ice bath at a temperature of 0 to 4 ° c The reaction is maintained under the same conditions for the following three hours after the final addition of KMnO4. Then, with a 340 rpm stirring speed, the reaction is allowed to continue for the following five days. After five days, a 5 wt% aqueous solution of H2SO4 is gently added for one hour. Following the first two hours, the reaction is stopped by adding 3 gm of H2O2 to the solution, which is then kept for an additional two hours while being constantly stirred. The above-prepared solution is then mixed with an aqueous solution of H2SO4 (5 wt%) and H2O2 (0.5 wt%). The finished solution is then centrifuged and repeatedly rinsed with water to produce the GO solution, which is brown in color.

2.2. Characterization techniques

A scanning electron microscope equipped with a field emission gun (JEOL JSM7600F) was used to study the morphology of the GO. GO's Raman spectrum was taken using a Jobin Yvon Ramanor HG 2S. Atomic force microscope (AFM) measurements were performed using Digital Instruments Nanoscope IV Multimode scanning probe microscopy to determine the thickness of the GO sheets. X-ray photoelectron spectroscopy (XPS) examination was carried out utilizing a Thermo VG Scientific Multilab 2000 photoelectron spectrometer.

2.3. Device fabrication

In this study, we have fabricated three sensors on a flexible polyamide substrate with three concentrations of GO. For this purpose, we have prepared three solutions of GO with concentrations of 10mg/10µL, 10mg/100µL, and 10mg/1000µL named GO-1, GO-2, and GO-3 respectively. Then we perform sonication for 20 minutes at room temperature and after that, we dropcast these solutions on the sensor IDE structure as illustrated in Fig. 1(a), (b), and (c) and left to air dry for 24 hours.

Fig. 1 Fabricated sensor on a flexible substrate with (a) GO-1, (b) GO-2, (c), GO-3, and (d) Experimental set-up

2.4. Measurement set-up

To determine the LWD, one of the crucial criteria for predicting plant fungal disease, fabricated LWS is used. To accomplish this, we first examined the transfer characteristics of the fabricated LWS under laboratory conditions. The experimental setup used to investigate the transfer characteristics of the LWS, such as sensitivity, response time, hysteresis, and effect of temperature, is shown in Fig. 1(d). An LCR meter (HIOKI IM3536) is utilized in the experimental setup (Fig. 1(d)) to investigate the sensor transfer characteristics of the LWS when exposed to water molecules. To carryout lab measurement testing protocol has been previously reported. the sensor's area is devided into 0% (dry), 25%, 50%, 75%, and 100% (completely wet) referred as the sensing area wetness. and water is then sprayed on the sensor (Fig. 1(d)). The excitation voltage of the LCR meter is kept at 1 Vpp for lab measurements to avoid losses caused by the water electrolysis effect (Surya et al., 2020). To carry out measurement sensors exposed to wetness, we have connected the fabricated flexible sensor to an LCR meter, as shown in Fig. 1. (d). All of the experiments were conducted at 25 °C room temperature with 50% relative humidity.

3. Results and discussion

3.1. Sensor transfer characteristics

In this work, the effectiveness of the fabricated flexible LWS is investigated, and the sensor's transfer characteristics, including sensitivity, hysteresis, response time, and temperature effect were studied. For this purpose, GO (Graphene Oxide) with three different concentrations were used as the sensing film in the flexible LWS (leaf wetness sensor), and in order to investigate its effectiveness, we looked at the sensor transfer function at three different GO concentrations, i.e. (GO-1, GO-2, GO-3). We adhered to the LWS testing technique used in this study, as was previously published in ref [9]. The sensing area is separated into five different regions in percentage i.e., 0% (totally dry), 25%, 50%, 75%, and 100% (completely wet) in order to understand the sensor's change in capacitance with respect to the wetness. Similarly the characteristics of moisture sensor are experimented [Neema et. Al., 2024]

Further, we measured the change in capacitance of the different concentrations of GO sensors i.e., GO-1, GO-2, GO-3 for different wetness levels with a frequency range from 100 Hz to 8MHz as shown in Fig. 2 (a), 4 (b), and 4 (c), respectively. The sensor capacitance decreases with increasing frequency as seen in Fig. 2 (a), (b), (c). it is also found that the sensor capacitance increases with an increase in the wetness area, as per ref [7, 9].

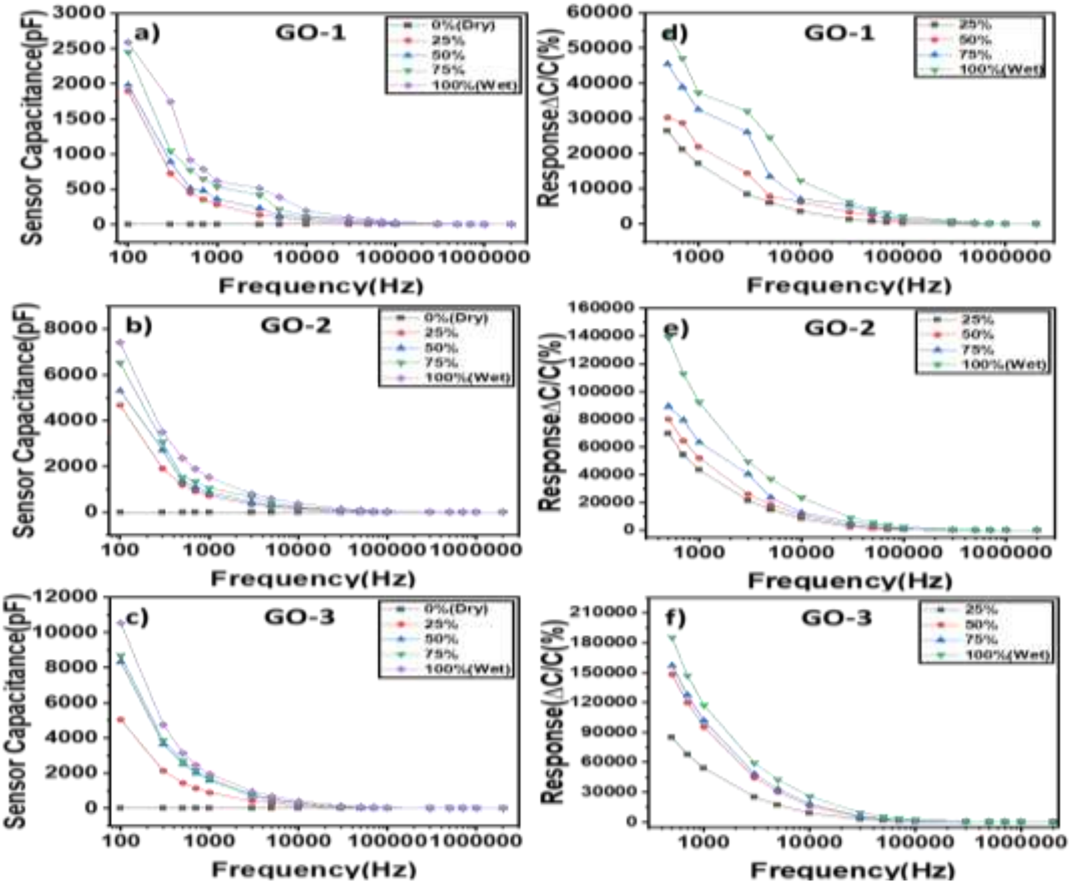

Fig. 2 Fabricated flexible LWS sensor change in capacitance w.r.t the wetness at different frequencies with different concentrations of GO for (a) GO-1 (b) GO-2 and (c) GO-3 and sensor response for (d) GO-1 (e) GO-2 and (f) GO-3

Further, we have examined the fabricated flexible LWS response for different wetness at different frequencies, we have calculated the sensor response using (3) ref [7] for the GO based sensor as shown in Fig. 2 (d), 2 (e) and, 2 (f) for GO-1, GO-2, GO-3, respectively. Interestingly, the sensor with three different concentrations of GO, we have found that, the highest response approximately of 120000%, 90000%, and 37000% for GO-3, GO-2, and GO-1, respectively at 1KHz frequency.

Additionally, as shown in Figs. 3(a),5(b), and 5(c), we have investigated the response times of the fabricated flexible LWS of GO based sensors at 100% wetness. It is clear from Figs. 3(a) ,3(b) and 3(c) that all the GO sensor has a response time about 180 seconds and recovery time around 10 seconds when the sensing area of the fabricated flexible LWS is exposed to wetness.

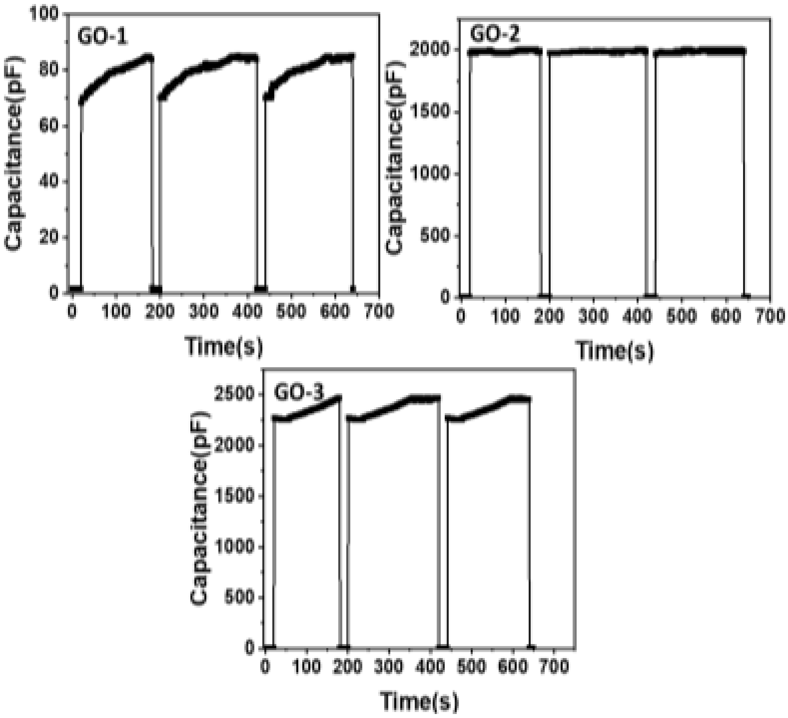

Fig. 3 Fabricated flexible sensor response time of GO flexible leaf wetness sensors i.e., GO-1, GO-2, GO-3 for 100% wetness respectively.

We further followed the techniques recommended in ref [7] in order to study the hysteresis of the fabricated flexible LWS. The sensor's hysteresis is depicted in Figs. 4(a) ,4(b), and 4(c), respectively, for three different concentrations of GO flexible LWS sensor i.e., GO-1, GO-2 and GO-3. Firstly, sensor reading taken at o% wetness and then the sensor area wetness increases by 25%, 50%, 75%, 100% i.e. in increasing order of wetness and respected measurement has been noted down this termed as adsorption. Same measurement has been done in reverse order, it termed as desorption. From figure 4 (a), (b), (c) it is observed that GO sensor has hysteresis of about 10%, 3%, and 5% of wetness for GO-1, GO-2, and GO-3 respectively.

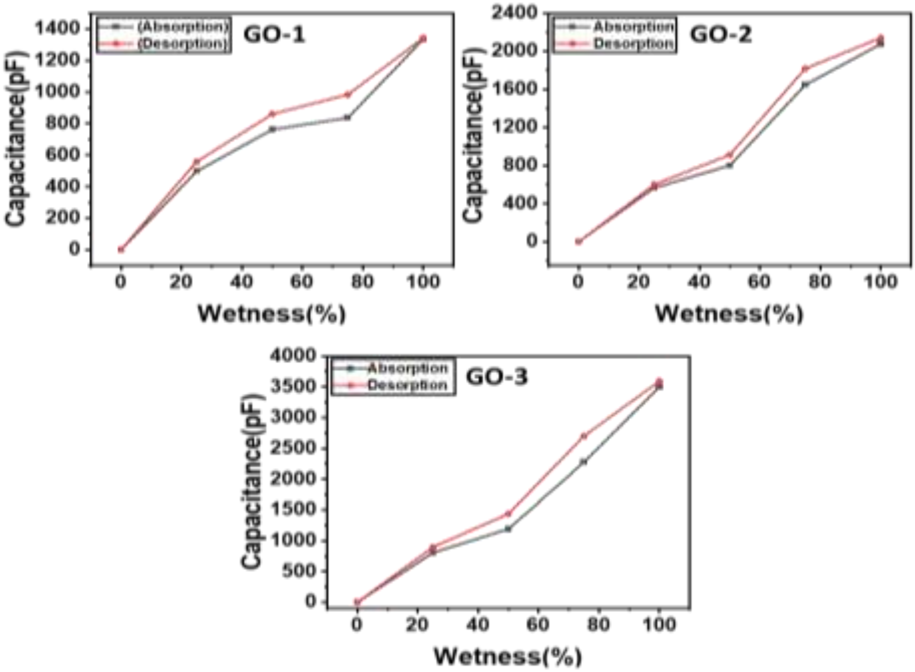

Fig. 4 Fabricated flexible three different concentrations of GO sensor hysteresis i.e., GO1 (a), GO2 (b) and GO3 (c) sensors respectively.

We also explored the temperature response of the sensor form the temperature range of 20 ^{0}C to 70 ^{0}C. for this purpose we have kept sensor in the oven and start temperature increasing from 20 ^{0}C to 70^{0}C and the corresponding reading has been calculated. Form figure 5, it can be observed that the temperature response of the sensor is about 2.3% when temperature varies from 20 ^{0}C to 70^{0}C.

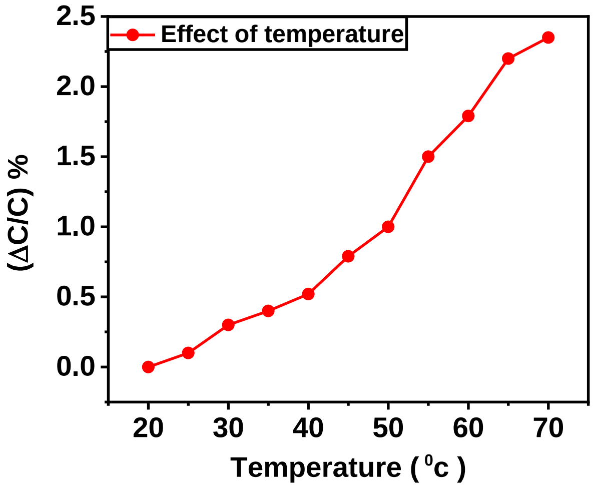

Fig. 5 Temperature response of the Fabricated GO LWS

4. Conclusion

Monitoring and early detection of plant diseases are crucial to reduce agricultural yield loss. Information on LWD is crucial for calculating the probability of plant diseases, and LWS is frequently used to measure it. In this work, we have developed the LWS on flexible substrates with three different concentrations of GO and evaluated the performance of the fabricated flexible LWS in the lab. According to the lab measurements, the fabricated flexible LWS provides a response of around 120000%, 90000%, and 37000% with concentration of 10mg/1000µL, 10mg/100µL, and 10mg/10µL respectively, when the sensing region is totally wet with respect to air. Furthermore, it is found that the response time for the fabricated flexible LWS is approximately 180 seconds, with a hysteresis of 10%, 3%, and 5% of wetness for GO-1, GO-2 and GO-3, respectively. It's interesting to observe that when temperature changes, the LWS sensor capacitance only changes by 2.3%.

Data availability

Data will be made available on request.

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