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The Temperature Effect on the Performance of Acetone Sensor Based on $K_2W_7O_{22}$ Nanorods

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Abstract

Acetone that is found in human breath acts as a biomarker that can be used to non-invasively diagnose diabetes and monitor blood sugar levels. However, the detection of breath acetone is very challenging because 1) the concentration of breath acetone is ranging from parts-per-billion (ppb) to parts-per-million (ppm) which is too low to be easily detected; 2) the exhaled gases human breath contain more than 1000 different volatile organic compounds (VOCs). Our study demonstrated that $K_2W_7O_{22}$ (KWO), a newly synthesized functionalized nanomaterial, can detect acetone with excellent sensitivity and selectivity. The lowest detection limit of acetone detection can be down to 0.1 ppm at room temperature with a distinguished response to acetone comparing to the other common chemicals in breath such as ethanol and methanol, and good tolerance of moisture. The study of the sensing mechanism revealed that KWO can have such an excellent response to acetone not only because of its porous and nanostructured morphology but also due to the unique low-temperature ferroelectric property. However, we also observed that this low-temperature ferroelectric property can easily induce a ferroelectric-electrochromic effect which can significantly affect KWO electric stability and then its sensing capability of acetone detection. This paper is to find a solution of reducing the ferroelectric-electrochromic effect and maintain its electric property during the sensing testing. Preliminary results show that heating the sensor in a small margin (30°C) could further improve KWO sensitivity (240.63% at 25 °C whereas 254.96% at 30 °C) to respond to acetone while avoiding the color change caused by the ferroelectric-electrochromic effect. The proposed mechanism is discussed.

Keywords: Chemiresistive Sensor; Charge Transfer; Ferroelectric Property; $K_2W_7O_{22}$ Nanorods; Diabetes; Acetone

Introduction

Human exhaled breath contains more than hundreds of volatile organic compounds (VOCs). Elevated concentrations of some VOCs in exhaled breath show a solid correlation to specific diseases and can be used as biomarkers for certain diseases [1-3]. Because human breath is abundant and can be assessed through non-invasive methods, breath-based sensor devices have attracted much attention and may offer an ultimate solution for noninvasive monitoring and diagnostics of diseases. Diabetes is a chronic disease referring to a group of diseases that affects how one body uses blood sugar (glucose), while glucose is an important energy source for cells in the body for making up muscles and tissues [4, 5]. In general, diabetes is diagnosed on a basis of glucose concentration in the intrusive blood, which is invasive. Also, diabetes treatment is expensive which annually costs about $4.3 billion in the USA [6]. Complications of diabetes are severe which can cause blindness, kidney failure, non-traumatic lower-limb amputation, and even death [7]. Therefore, a non-invasive, convenient, and low-cost method or device for diagnostic, preventive, and monitoring diabetes is very needed [8]. Clinic research reveals that one of the VOCs, breath acetone, has shown a strong relationship of simultaneous blood glucose levels in patients with type 2 and 1 diabetes [3, 9-11]. Therefore, breath acetone can be the biomarker for diabetes diagnosis and monitoring [5, 12, 13]. The acetone concentration in healthy people's breath varies from 0.3 ppm (parts-per-million) to 0.9 ppm [14]. While, for diabetes patients, the concentration of breath acetone is above 1.8 ppm [15].

Considering such a low concentration of breath acetone (around ppm level) and the complexity of human breath (more than 1000 VOCs and high and variable concentration of humidity), the major challenge for breath acetone detection is to find a gas sensor that can detect acetone high sensitively and selectively with good tolerance of humidity. A range of sensing materials, e.g., Pt-InN, WO_3, Pd/TiO_2/Si, have been developed to be used for acetone detection [15,16-18]. However, these materials-based sensors exhibit either low sensitivity or requiring operation at elevated temperatures, ~ 200 °C or even higher. Therefore, these devices have to include a heat source, which consumes high power and deteriorates materials' structural stability, shortened device lifetime, unstable responses, and poor device-to-device consistency. A sensor device operating at low temperature would be preferable to achieve sensitive and selective acetone detection and provide a high-performance, low-cost diabetes diagnosis solution for the need of point-of-care diabetes monitoring and control [20].

In our previous study [12, 15, 19, 21], we found that the new nanomaterial $K_2W_7O_{22}$ exhibits excellent room-temperature sensing response to acetone [12, 19]. The study of material structure and property relationship further reveals that KWO has unique room-temperature ferroelectricity and 1-dimensional porous nanostructured morphology. These cause a strong interaction and efficient charge transfer between acetone molecules and KWO [12, 19] at room temperature. The ferroelectricity in KWO material is also called spontaneous polarity caused by the displacement of tungsten atoms by potassium from the center of each [WO_6] octahedral and then generate the ferroelectricity. On the other hand, Hall measurement confirms that KWO is a p-type semiconductor [12, 13]. It means the resistance of KWO will increase when an electron-rich polar compound, e.g. acetone is adsorbed on its surface and can form a charge depletion region due to the reduction of holes in KWO. Room-temperature ferroelectricity, porous nanostructure, and p-type semiconductor facilitate KWO to become a promising material for breath-based acetone sensor devices. However, our preliminary study noticed that there are a few limitations in KWO based sensors for diabetes application. One limitation is a strong interference of humidity in KWO based sensors [19]; the other limitation is that KWO based sensor quickly shows color change which could be caused by the room-temperature ferroelectric-electrochromic effect [22-24]. In particular, it is observed that the KWO based sensor shows reduced sensing response to acetone and a baseline shift when color change happens. To overcome these limitations, we have found a solution: applying a little external heat (30°C) on the bottom of the sensor can help to avoid the sensor's color change and effectively preventing the ferroelectric-electrochromic effect. This can maintain the sensing performance stably for long-term use. A detailed approach and explanation are described in the following.

Material Synthesis and Characterization

The single crystalline nanostructured KWO was synthesized using a hydrothermal method, and detailed descriptions can be found elsewhere [12, 25, 26]. Briefly, a precursor solution containing Na_2WO_4, oxalic acid, K_2SO_4, and HCl was made. This solution was

then put into a 30 mL autoclave for synthesis at 225°C for 24 hours. The synthesized nanostructured KWO was dispersed in ethanol to form a suspension and drop-casted on glass substrates to form a thin film of about 10 μm in thickness. The morphology of the KWO films was studied with scanning electron microscopy (SEM) (Figure 1a) and indicated a three-dimensional porous mesh made by KWO nanorods. The average length of the nanorods was about several microns, and the diameter of the nanorod was about 10 nm, which shows a large surface-to-volume ratio. The Raman spectroscopy (Figure 1b) clearly shows the □-phase (ferroelectricity) in as-synthesized KWO.

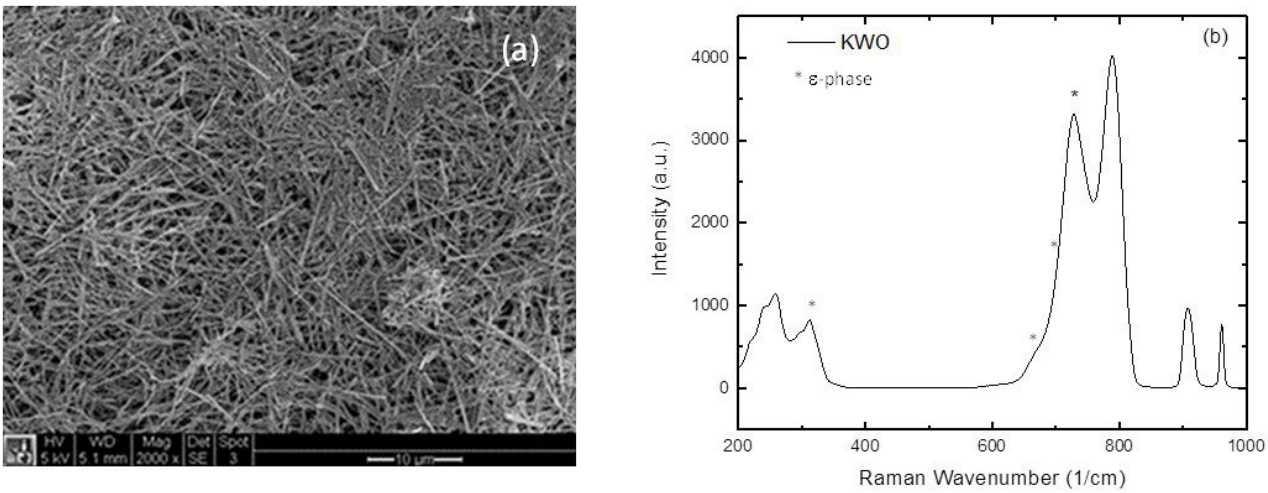

Figure 1: (a) SEM image of the interconnected 3D mesh structure of a KWO thin film; (b) Raman spectrum of KWO

Experimental Setup

Figure 2 shows an overview of the whole testing system. The acetone vapor is precisely generated and controlled through an OVG-4 vapor generator (Owlstone InC., Westport, CT, USA) from 0 ppm to 8 ppm [27]. The 25 ppm acetone can be generated from acetone cylinder. The relative humidity (RH) level can be controlled through OHG-4, humidity generator (Owlstone InC., Westport, CT, USA) from 8% up to 85%. The sensing test has been conducted to detect the variable concentration of acetone at a controlled RH under room temperature. The change in resistance across the film was measured using the Keithly electrometer. RH and temperature were monitored using a commercialized sensor. The concentrations of acetone vapor used in this study are 2.86 ppm， 8 ppm and 25 ppm at RH 30%, respectively, at room temperature. To conduct the experiment, the baseline resistance of the sensors at 30% RH was measured and recorded [19].

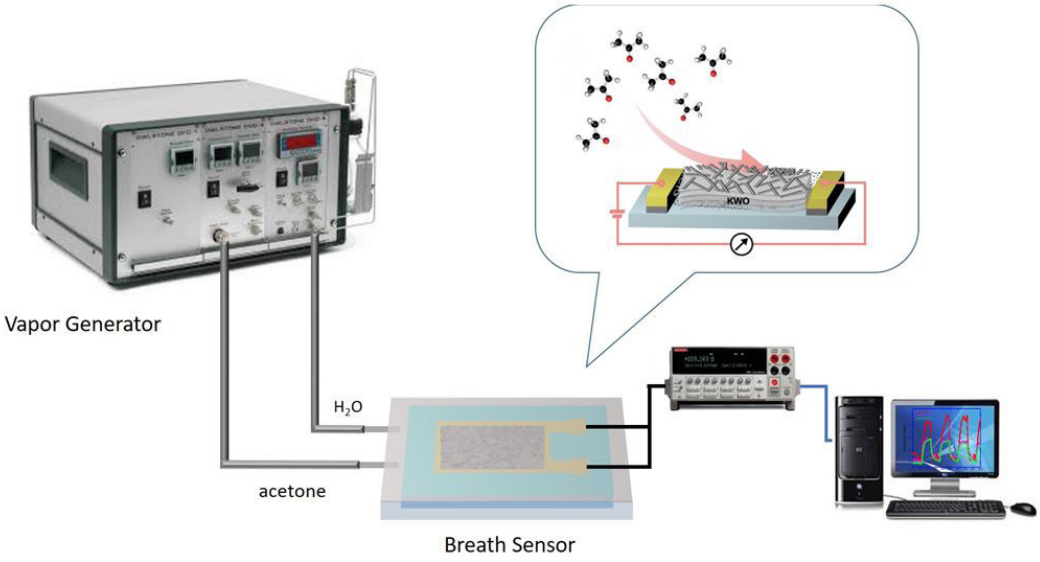

Figure 2: Block diagram of the testing system

Results & Discussion

In our previous study [13, 19, 21, 27, 28], the ferroelectric property of KWO has been confirmed via well-defined phase hysteresis curve and butterfly loop by the piezoresponse force microscopy (PFM) [13] and verified this is the main reason resulting in the efficient interaction between KWO and acetone molecules even at room temperature. That is, the sensing mechanism of KWO to acetone is not following a normal metal-oxide-semiconductors (MOS) based chemiresistive response which needs to operate at elevated temperature. This is because MOS sensors are based on an oxidation-reduction reaction due to the formation and removal of surface oxygen while the temperature of ionosorption of oxygen, O- ions, requires at least 100 °C [9, 10]. Figure 3 shows the sensing performance of KWO based sensor to detect acetone with concentrations from 0 ppm to 6 ppm at room temperature, RH = 30% [28]. The result indicated a significant chemiresistive response in KWO to acetone with excellent sensitivity up to 0.1 ppm of acetone at room temperature.

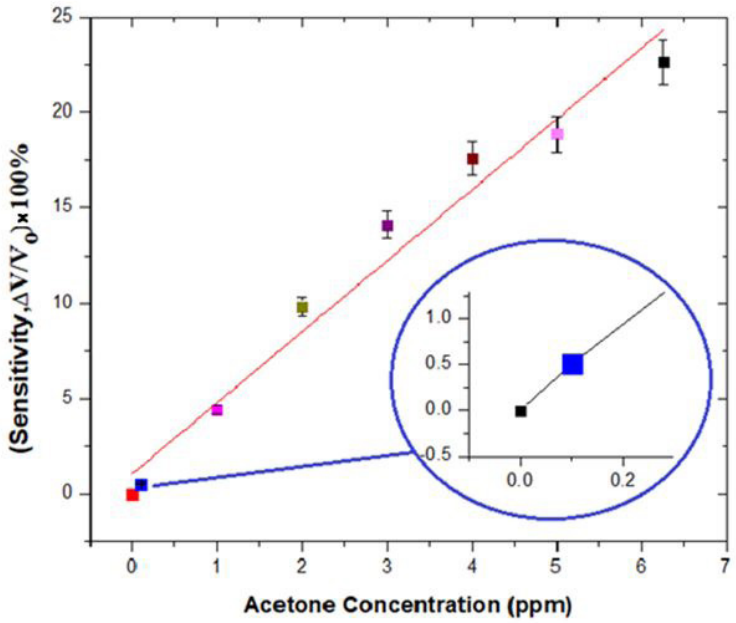

Figure 3: The response curve of the KWO sensor to detect acetone from 0 ppm to 6 ppm at room temperature, RH ~ 30% [28]

To further study the KWO based sensor's sensing mechanism and temperature effect on its sensing performance, we conducted a series of sensing tests to detect acetone (25 ppm) at different operating temperatures from 25°C to 70°C. As shown in Figure 4, the response of the KWO sensor to acetone increases a little at a temperature around 30.68°C but becomes weaker and even disappeared while the operating temperature continuously increases. This result further reveals a different sensing mechanism in KWO based sensor for acetone detection. Generally, the sensitivity is improved in most MOS based chemiresistive sensors when the operating temperature is increased. It is because the higher temperature can reduce the ionized impurity scattering effect and improve MOS electron mobility to promote the charge transfer during the sensing process. The KWO nanomaterial, as a p-type semiconductor, has holes for the majority of carriers. Like other semiconductors, the conductivity of KWO will be increased while temperature increases [29-33] since more holes gained energy to move towards the valence band and cause stronger interaction with acetone molecules. Therefore, the response of KWO to acetone was increased while temperature increased from room temperature 25°C to 30.68°C. However, a weaker or even disappeared response to acetone was observed when the operating temperature was increased above 30.68°C. This dramatic change on increased temperature in KWO could be explained by the temperature-dependent ferroelectric property in KWO [34, 35]. It means the increased temperature can induce a crystalline phase transition in KWO and result in decrease of material ferroelectricity [35]. The lower ferroelectricity of KWO will make it less sensitive to detect acetone. A more detailed mechanism needs to be investigated later on.

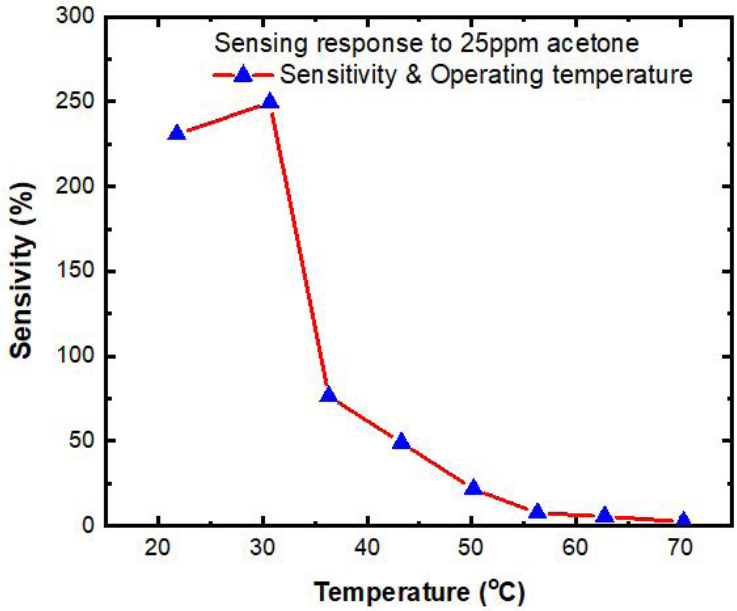

Figure 4: KWO sensor response to 25ppm acetone at different temperatures

Stability is another important parameter to evaluate a sensor performance [36, 37] which can be assessed by the consistent reading over time. We have tested the stability of the sensor for 30 days. Figure 5 shows the KWO based sensor is the maximal concentration of acetone generated by OVG-4 is 8ppm in the room-temperature sensing setup.

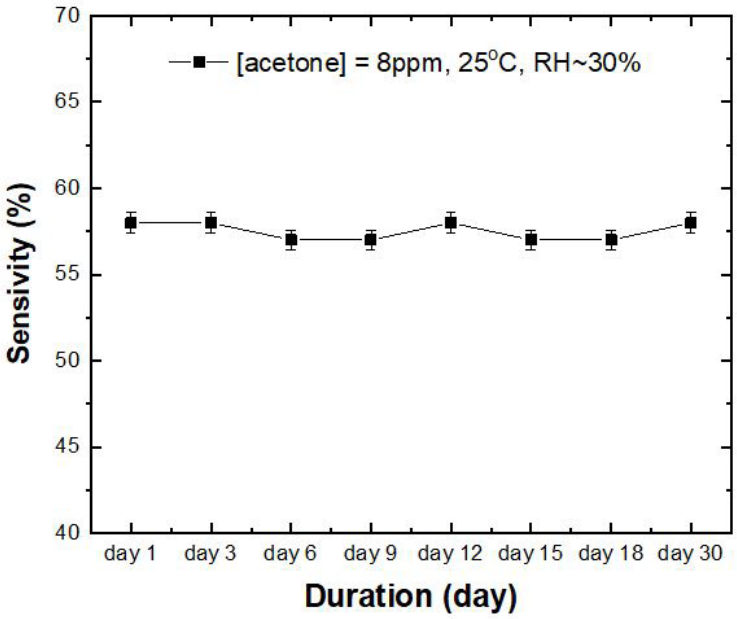

Figure 5: Stability of the KWO based sensor to detect 8 ppm acetone in the air for 30 days

Human exhaled breath contains many different volatile organic compounds (VOCs), and acetone, methanol, and ethanol are major components in the breath [38]. All of these compounds are in trace concentrations in human breath. To verify the selectivity [39, 40] of KWO based sensor, we used KWO based sensor to test 8 ppm of acetone, ethanol, methanol individually at room temperature and the same RH (~ 30%). Figure 6 shows the resistance change of the sensor for acetone, methanol, and ethanol gases. It confirmes that KWO shows the best selectivity to acetone compared to the other two gases.

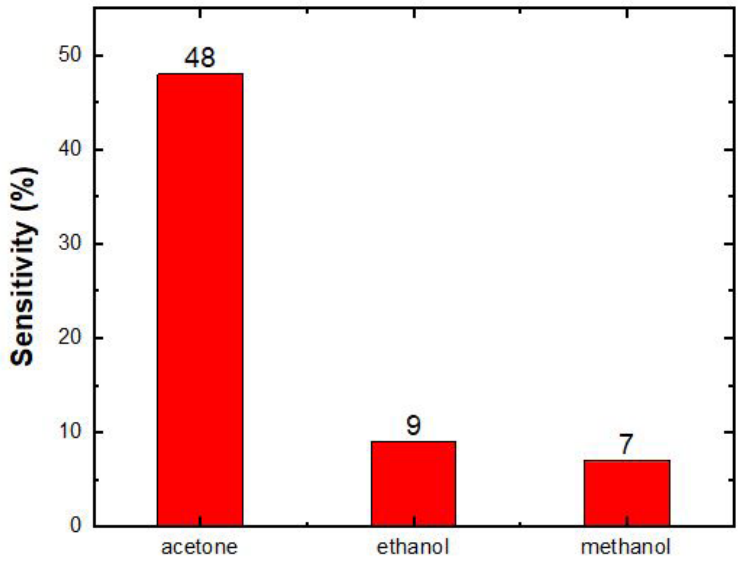

Figure 6: Sensitivity of KWO based sensor to detect 8 ppm acetone, ethanol, and methanol at room temperate, RH ~ 30%

Table 1 lists recently reported materials [5, 12, 13, 41-50] for acetone detection at RH around 30%. As indicated, most sensors are working at elevated temperatures in order to reach a certain level of sensing performance. Only KWO based sensor can work at room temperature with an excellent sensing response to acetone. The results once again confirm that the interaction of charge transfer between acetone and KWO differs from the usual oxidation-reduction-based chemiresistive response, which is mainly dominated by KWO's ferroelectricity [12, 13,19, 28].

Materials Name	Operating Temperature (°C)	References
Si-WO_3	300	[5]
PPy-WO_3	90	[41]
Pt-InN	200	[42]
$K_2W_7O_{22}$	25	[12, 13, 19, 28]
Pd-TiO_2/p-Si	100	[43]
Ni/InGaN/GaN	100	[44]
Fe_2O_3/Pt	300	[45]
Fe_2O_3/RuO_2	300	[45]
TiO_2	500	[46]
SnO_2-ZnO	300	[47]
Cr-WO_3	400	[48]
Ce-SnO_2	210	[49]
ZnO	400	[50]

Table 1: List of materials that are sensitive to acetone at different operating temperatures

In Summary, above experimental results reveal that in a KWO based sensor, the resistance change is mainly due to the unique KWO material property-ferroelectricity, which promotes the charge transfer and induces the resistance change even at low operating temperatures and become a promising sensing material to be used for breath acetone detection with high sensitivity and selectivity. In the meanwhile, the KWO based sensor is sensitive to operating temperature due to the temperature-dependent ferroelectricity in KWO.

Figure 7 exhibits the color change of sensing film caused by the ferroelectric-electrochromic effect [24] while applying a specific voltage during sensor operation. The applied voltage provides an external electric field that can generate a spontaneous polarization in KWO. Meanwhile, the ferroelectricity can facilitate charge accumulation and the oxidation of W in KWO to cause the ferroelectric-electrochromic effect [51, 52]. When the color turns to deep blue, it indicates the ferroelectric-electrochromic effect in KWO which is the reason to cause reduction of sensing response of KWO to acetone.

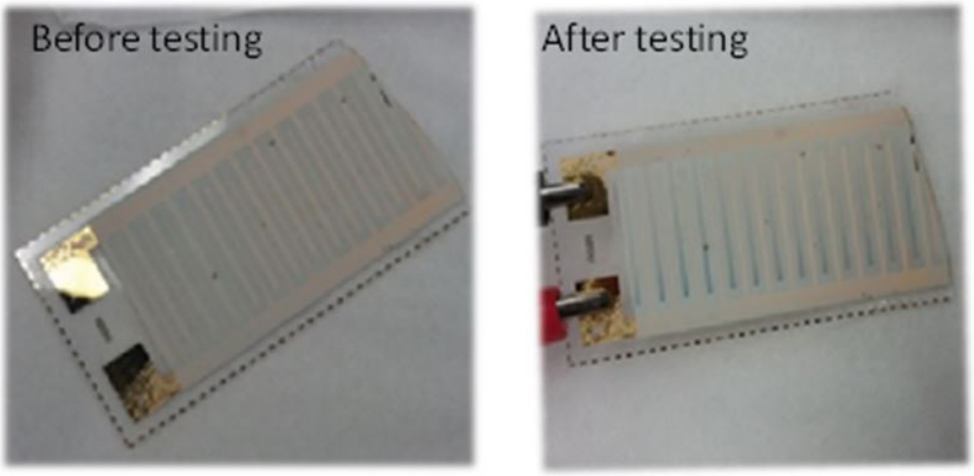

Figure 7: KWO thin-film color change before and after sensing tests

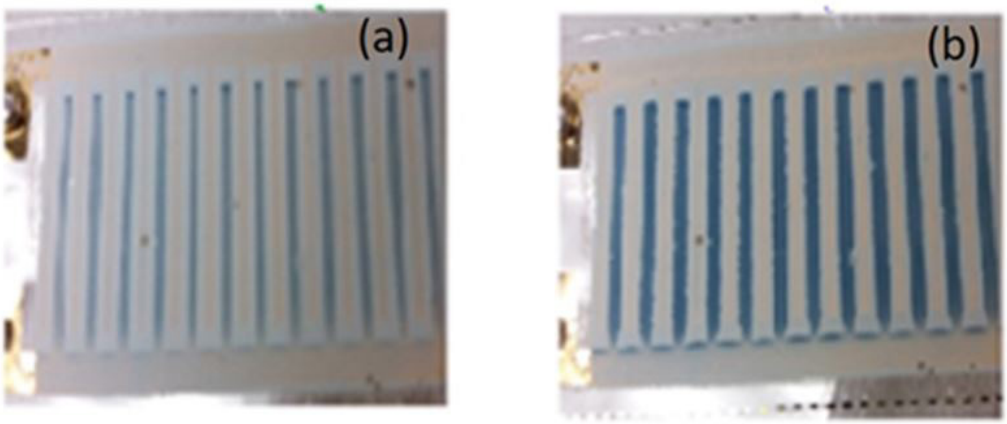

Figure 8: KWO sensor to detect 25 ppm acetone a) with heat 30.32oC; b)without any external heat. The sensitivity is 202.9% in (a) and only 12.5% in (b).

Considering the temperature can influence on material's ferroelectricity [34], it motives us to think that the color change might be reversible while using external heat. Figure 8 shows the KWO film to detect acetone without and with applying extra heating. The results clearly indicate the sensor can maintain high sensing performance operating at 30.32°C stably while the sensor without extra heat shows dramatically reducing sensing response to acetone. This finding provides a potential solution for KWO based sensors to avoid the ferroelectric-electrochromic effect while maintain a good sensing performance. Also, we realize that the external temperature does not need to be too high, a little bit higher than room temperature, e.g., 30.32°C, which is efficiently enough to prevent the color change of the KWO based sensor (Figure 8). It provides information that we can avoid color change of the KWO based sensor while still keeps its high sensing performance and still operating at low temperature. A concern of increasing sensor's power consumption and complexity is not necessary.

To precisely determine the optimal operating temperature, Table 2 lists the KWO based sensor responds to 25 ppm acetone operating at temperatures around 30°C. It is noticed that a faster and higher sensing response was observed when the temperature 30.32°C. The response becomes worse when KWO based sensor working at a temperature lower or higher than 30.32°C. This result indicates that 30.32°C is the optimal working temperature to keep the sensor performance and avoid the ferroelectric-electrochromic effect. Comparing to other sensors' operating temperatures (Table 1), 30.32°C still can be considered as low temperature without causing extra power consumption.

Temperature(°C)	Sensitivity (%)	Rise time (sec)
25	240.63	34.95
28.2	246.03	27.63
30.32	254.96	19.41
32.56	200.10	18.20
36.36	60.82	16.24

Table 2: Comparison of temperature, sensitivity, and rise time [53] at three different temperature of KWO baed sensor to detect 25 ppm acetone (RH ~ 30%)

Conclusion

Due to the unique room-temperature ferroelectric property, nanostructured KWO shows an excellent sensing response to acetone. However, the ferroelectric-electrochromic effect can change the sensing film color during the testing and weaken its sensing response to acetone. This can result in an unstable and decreased sensing response of KWO to acetone. In this paper, results exhibit that the color change can be reversible, and sensor performance will be maintained by slightly increasing oper-ating temperature due to the temperature-dependent ferroelectricity in KWO. This work provides a method to design and fabricate a low-cost, non-invasive, stable, low-power consumption, simple and user-friendly acetone sensor based on the new sensing material, KWO nanorods with great potential as a point-of-care medical device in diabetes diagno-sis, monitoring, and treatment guidance with long-term stability and reliability.

Credit authorship contribution statement

Md. R.H. initially wrote the paper and conducted experiments; A.S. worked on the major revision. M.J. performed all XRD, FT-IR, and Raman spectroscopy experiments, analyzed data, and synthesized the materials; Q.Z. designed material synthesis procedures and analyzed experimental data; D.W. wrote and finalized the paper, guided sensing experiments, analyzed experimental data, and led the research team. All authors have read and agreed to the published version of the manuscript.

Declaration of Competing Interest

The authors declare no conflict of interest.

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