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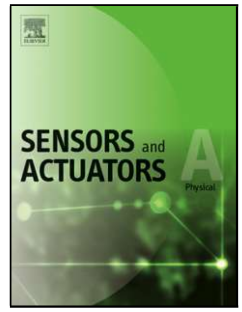

Title: Highly Sensitive Capacitive Cell Based on a Novel $CuTsPc-TiO_2$ Nanocomposite Electrolytic Solution for Low-Temperature Sensing Application

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Highly Sensitive Capacitive Cell Based on a Novel CuTsPc-TiO_2 Nanocomposite Electrolytic Solution for Low-Temperature Sensing Application

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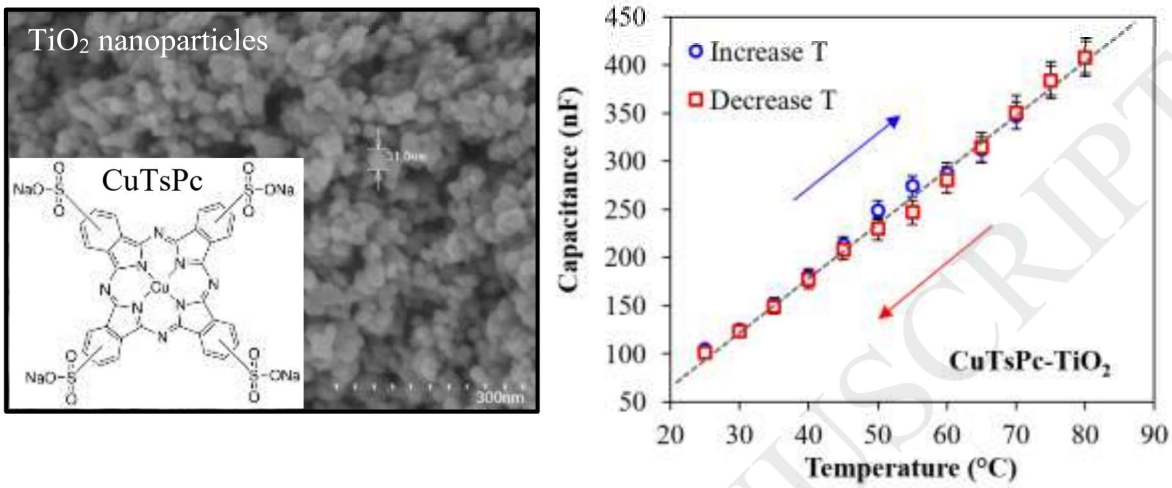
Graphical Abstract



Highlights:

1. A novel temperature sensor was fabricated with CuTsPc based electrolytic fluid.
2. TiO_2 nanoparticles have been added to enhance the sensitivity of the device.
3. Combination of CuTsPc and TiO_2 showed strong temperature dependence on the capacitance.
4. The hybrid electrolytic based device enhanced the performance of the sensor.

Abstract

This work demonstrates a highly efficient electrochemical cell based on the hybrid nanofluid of titanium dioxide (TiO_2) nanoparticles dispersed in a water-based copper (II) phthalocyanine-tetrasulfonic acid tetrasodium salt (CuTsPc) solution. A chemical cell of ITO/nanofluid/ITO has been fabricated to study the effect of temperature variations towards its capacitance and resistance. The resultant device possessed capacitive sensing mechanism, and the synergistic hybridization of the two different sensing elements lead to the superior performance as compared to the single CuTsPc based device. The hybrid device outperformed the pristine CuTsPc based device in terms of sensitivity, stability, linearity, response/recovery and possessed narrow hysteresis loop.

Keywords: Temperature, nanofluid CuTsPc, TiO_2, capacitive sensor.

1. Introduction

Organic-based sensors have been immensely researched due to their realization in next generation flexible electronic modules, compatibility with large-scale production, low cost and light weight [1, 2]. Various kinds of chemical and physical sensors have been developed for a variety of applications such as detection of temperature [3], glucose [4], humidity [5], pressure [6], light intensity [7] and gas sensing [8]. Among the aforementioned sensors, heat is an important parameter that is often measured. Temperature measurement is equally essential for the humans and the other living species as well as the industry, transportation and the agriculture *etc* [9, 10]. Therefore, it has a lot of potential applications such as electronic health monitoring and detecting human body temperatures *etc.* [11]. These organic temperature sensors operate at low temperatures typically upto 100 °C, making them highly suitable for low temperature applications.

In this context, electrochemical sensors based on organic compounds such as porphyrins, porphyrazines and phthalocyanines have been reported for their potential applications in electronics [12-14]. In our recent works, we used phthalocyanine (Pc) that is a well-known material and has many derivatives with diverse properties [15]. Nickel (II) phthalocyanine-tetrasulfonic acid tetrasodium salt (NiTSPc) and copper (II) phthalocyanine-tetrasulfonic acid tetrasodium salt (CuTsPc) were used as an electrolytic solution in the electrochemical temperature sensors [3, 16]. In the later mentioned sensor *i.e.* CuTsPc based electrolyte, it was realized that the sensor was more suitable to be employed as a resistive temperature sensor. However, it lacks in response time for the use in the real temperature sensing applications. This might be attributed to the resistance limitation of the aqueous solution towards the change of temperature. Moreover, the design structure of this sensor device was constructed according to parallel-plate model, which tends to facilitate the change of other electronic variables, such as capacitance, rather than resistance. Consequently, it puts a restriction to the sensor's sensitivity in the resistive mode. Therefore, further improvement was needed to improve the sensing mechanism as well as sensing parameters. The possible solution to circumvent the performance constraints associated with the organic compound as a single material, is through the introduction of nanocomposites based on organic-inorganic hybrids.

However, it is important to understand the characteristics of an ideal sensor and significance of nanocomposite materials in the sensor's technology. An ideal sensor should possess excellent sensitivity and stability, short response/recovery time, low hysteresis, broader sensing range and the low cost [17, 18]. Several nanomaterials such as metal oxides, carbonaceous and organic materials as well as their composites are considered interesting candidates exhibiting remarkable performance in sensing applications [18, 19]. The properties of the nanomaterials can be potentially altered after doping with different materials and performance of the resultant composite based device can be remarkably enhanced. For instance, Chani *et al*. [20] fabricated a temperature sensor based on CNTs-silicon nanocomposite and the resultant sensor showed excellent sensitivity. Similarly, Guo *et al.* [10] demonstrated a low-temperature NO_2 gas-sensor based on polythiophene-WO_3 organic-inorganic composite that possesses very good selectivity and high response. In addition, a lot of work has been done in recent years to develop high performance temperature sensors based on different materials and technologies [21-23]. For instance, Xiaochen *et al*. [24] demonstrated an organic low operating power temperature sensor typically operating below 4 V and the resultant device showed excellent performance. In another study by Myeonghun *et al.* [25], a flexible organic field effect transistors (OFET) temperature sensor typically operating between 25-100 °C, and based on polymeric channel and gate-insulating layers has been demonstrated. The resultant device showed very stable performance that can be attached to the human finger to sense not only the human body temperature but also the surrounding objects. Hence, making it suitable for a variety of applications such as medical instruments, artificial skins for humanoid robots and surveillance systems *etc*. Further in this context, Xiaochen *et al.* [11] presented an organic temperature sensor based on metal-organic (silver NPs- pentacene) thermistor and an OFET. The device showed very strong temperature dependence in the resistance. Owing to the importance of the nanomaterials and nanocomposites in the sensing applications, the current work is expected to yield excellent results due to synergy of the organic-inorganic hybrid materials in a sensor.

Metal oxides owing to their excellent mechanical, chemical and electronic properties as well as their known and facile synthesis routes, when combined with organic compounds are expected to improve the sensing properties of such devices [13, 26]. Particularly, titanium dioxide (TiO_2) has received great attention due to its diverse sensing applications including gas sensors [27], optical sensors [28], environmental sensors [29] and temperature sensors [30, 31]. Besides, it has several inherent advantages such as it is nontoxic, biocompatible, photo-corrosion free and cost effective which makes it an ideal candidate for a range of applications [32, 33]. The synergistic hybridization of TiO_2 and CuTsPc to yield an electrolytic fluid is expected to overcome the disadvantages of any of the individual materials and enhance the overall performance of the device.

In this work, a novel electrochemical cell that detect variation of low temperature, based on the integration of metal oxide (TiO_2) into an organic electrolytic nanofluid is presented. The sensor demonstrated a strong capacitance dependence on temperature in CuTsPc solution with the addition of TiO_2 nanoparticles (NPs). By adding TiO_2 NPs into the CuTsPc organic solution, the sensing mechanism of the sensor changed to capacitive sensing as compared to the pristine cell that originally possessed the resistive sensing. Moreover, the new capacitive based sensor showed a remarkable increase in the sensitivity with a wide range in the capacitance values, significantly small hysteresis, and very good response/recovery time, thus making it very efficient to measure low temperatures with excellent sensitivity. The present work suggests a simple, facile and large scale compatible method to demonstrate the potential of metal oxide/ organic electrolytic materials in the future development of highly sensitive sensors for low temperature applications.

2. Experimental Section

Both CuTsPc (988.28 g/mol molecular weight) and TiO_2 NPs were purchased from Sigma-Aldrich. Optimization of CuTsPc aqueous solution and the cell fabrication were carried out according to our previously reported work [3]. Particularly, the metal oxide-organic electrolytic nanofluid was prepared by mixing 5mg of TiO_2 NPs into the 0.06 wt. % CuTsPc aqueous solution and stirred for a day. The cell that has been used as temperature sensor in this work, was fabricated using silica glass in which two inner walls were facing each other at 7mm distance, and the inner surfaces were coated with indium tin oxide (ITO). The active area of each electrode was 140 mm^2 with a sheet resistance of around 10 Ω/□. The dimensions of the sensors were measured to be about 7 mm x 7 mm x 20 mm (width x length x height) that were filled with 1 mL of CuTsPc-TiO_2 electrolytic nanofluid. A reference sensor (cell) has also been fabricated by using CuTsPc aqueous solution as an active agent. The schematic diagram of the sensor is presented in **Figure 1: (a)** reference cell, and **(b)** CuTsPc-TiO_2 nanofluid based temperature sensor. The micrograph of TiO_2NPs shown in **Figure 1** was taken by field emission scanning electrons microscope (Hitachi, SU8220 SEM).

For the sensing characterization and cell calibration, a type-K thermocouple probe was placed into the CuTsPc-TiO_2 nanofluid as a temperature calibrator during the measurement of both resistance and capacitance against the temperature variations. The values of both resistance and capacitance were recorded simultaneously using an LCR meter (GW Instek 829) at 1 KHz frequency. The experiments were repeated several times to ensure the reproducibility. A very stable sensing-ability was observed with maximum ±1% variation.

3. Results and Discussion

In this work, a simple solution processed temperature sensor has been fabricated by using a composite nanofluid of CuTsPc and TiO_2 as an electrolyte. Further, it is compared with single CuTsPc based sensor (reference sensor) on merits of response/recovery, sensitivity, capacitance and resistivity. The composite sensor largely varies in terms of sensor mechanism where its sensitivity shifted from resistive based sensitivity to the capacitance based sensitivity. Thus, the resultant sensor is suitable as a capacitive temperature sensor as compared to the reference device that is more suitable to be employed as a resistive temperature sensor [3]. Moreover, it is noteworthy that the capacitive sensing mechanism of both the CuTsPc based sensor (reference sensor) and the mixed CuTsPc-TiO_2 is based on synergetic effect of the temperature dependence of dielectric constants of both materials. The capacitance change of the organic electrolyte (CuTsPc) has already been discussed in reference [3]. It was described that the capacitance of the electrolyte is determined by the total ionic strength of the solution and mobility of these ions within the aqueous solution, which induces the dielectric constant change with various temperatures. Based on the measurements, it is envisaged that the dielectric constant of the organic electrolyte increases as the temperature increases, inducing higher capacitance. Adding TiO_2 NPs will enhance this effect, which has outperformed the single CuTsPc based in terms of capacitive sensitivity, as evident in our results too. The schematic of the current work is shown in **Figure 1**.

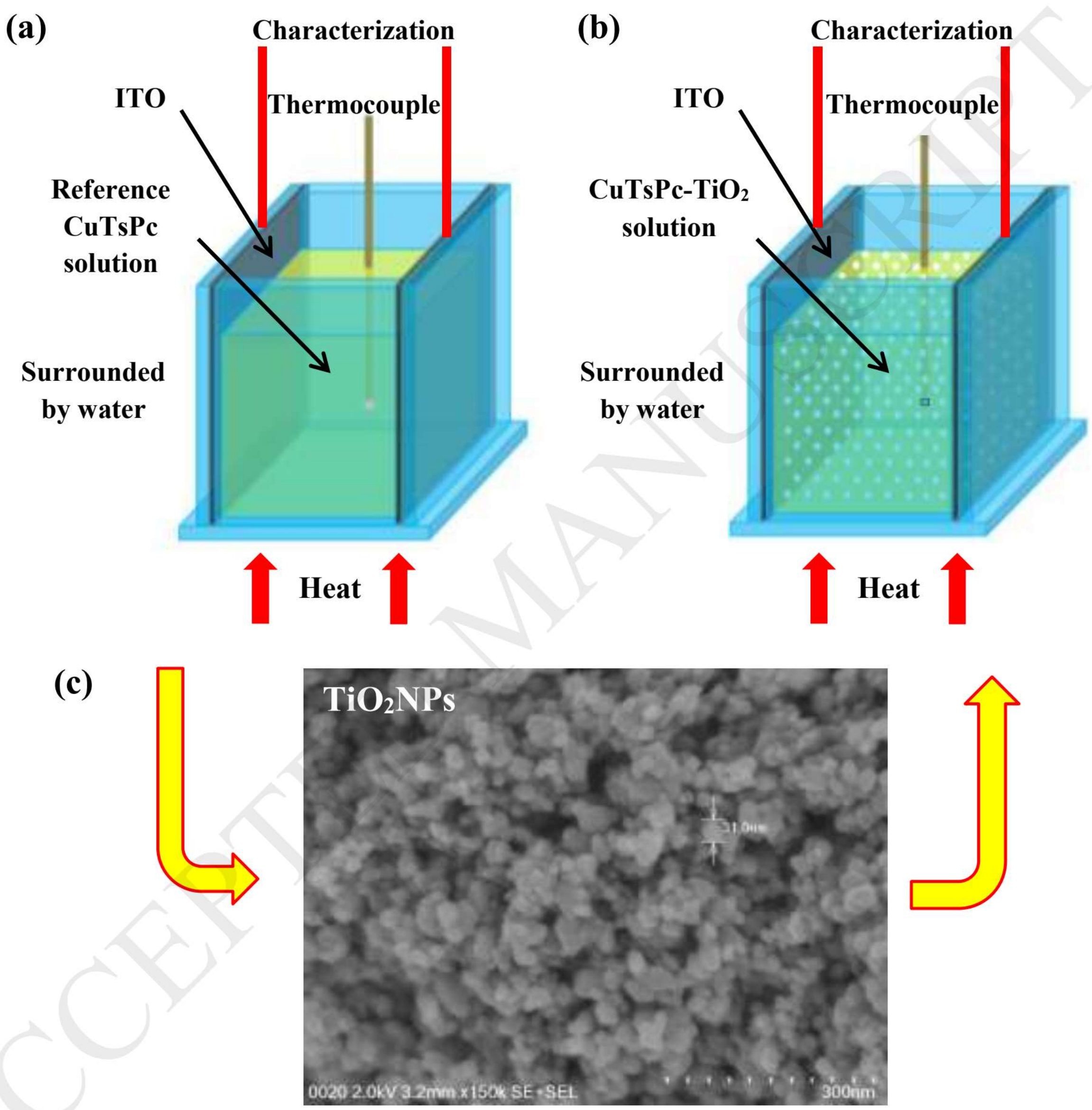


Figure 1: **(a)** Reference cell, and **(b)** CuTsPc-TiO_2 nanofluid temperature sensor.**(c)** FESEM micrograph of TiO_2NPs which were used to form electrolytic solution in the sensor cell.

Figure 2 shows the normalized capacitance and resistance as a function of temperature (°C) for reference and composite sensors. The sensitivity of the sensors in both capacitive and resistive outputswas obtained by the following **equation (1) and (2):**

$$S_C = \Delta C/T \qquad (1)$$

$$S_R = \Delta R/T \qquad (2)$$

Where ΔC, ΔR, and T are capacitance change, resistance change and temperature values, respectively.

The change of capacitance and resistance has been measured in both the directions, *i.e.* during the increase and decrease of the temperature, as shown in **Figure 2**. Almost all of the recorded parameters intersect at particular point of temperature as it increased or decreased. This indicates very excellent hysteresis, as very slight deviation is observed in only few points while most of the recorded parameters perfectly intersected. At the same time, a very good linearity has been obtained especially, in the capacitance-temperature relationship plot containing CuTsPc-TiO_2 nanofluid, as shown in **Figure 2c**. The capacitive linearity of CuTsPc-TiO_2 cell is observed to be better than that of the resistive mode, and better than the linearity of reference cell in both modes. The observed improvement in the linearity with temperature elevation is attributed to the inclusion of TiO_2NPs in the electrolytic fluid. Thus, the cell with the CuTsPc-TiO_2 showed relatively superior performance with the best hysteresis and linearity, simultaneously.

Moreover, **Table 1a** shows the sensivity parameters as the function of change in temperature. Both variants of sensor show small values in terms of resistive sensitivity, however, the capacitive sensitivity has largely improved for CuTsPc-TiO_2 based composite sensor that is approximately five times more sensitive than the reference sensor cell containing only CuTsPc aqueous solution. It is thus evident from the results that the composite sensor has outperformed its pristine CuTsPc based counterpart on account of capacitive based sensitivity.

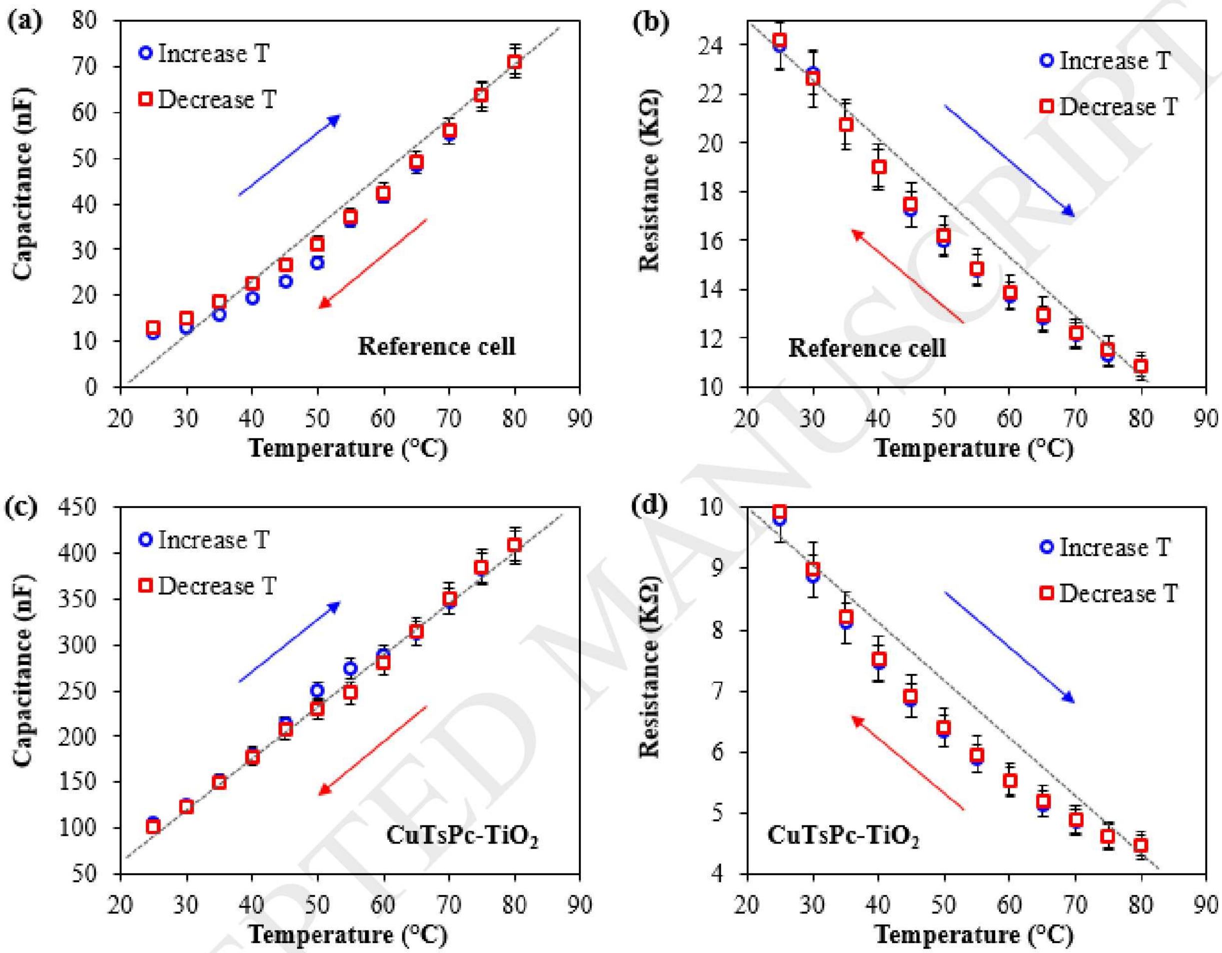


Figure 2: The capacitance-temperature and resistance-temperature plots for reference and $CuTsPc\text{-}TiO_2$ temperature sensors.

Table 1: (a) Capacitive and resistive sensitivities for reference and CuTsPc-TiO_2composite based sensors. **(b)** Response/recovery times for the CuTsPc-TiO_2 temperature sensor.

(a)

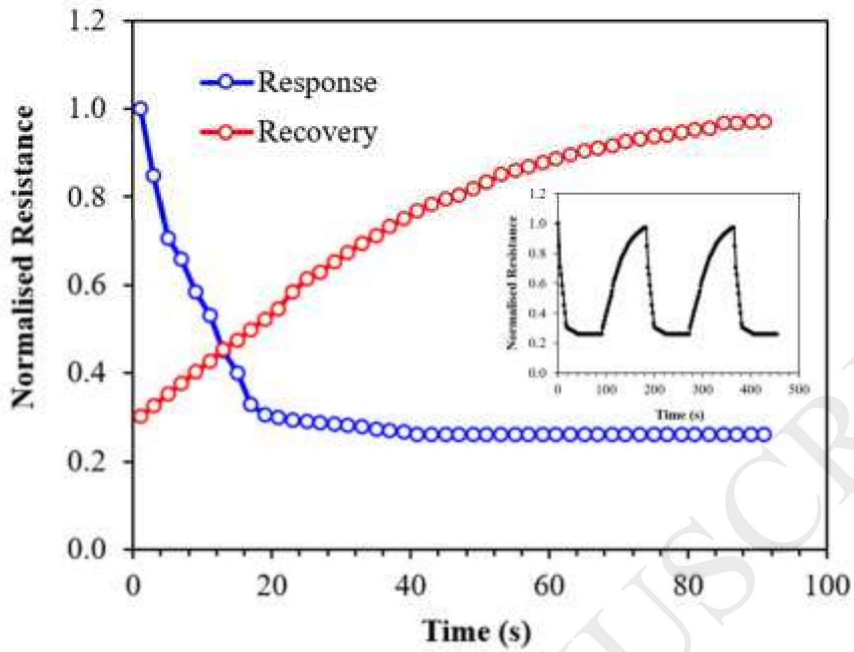
Sensors	Capacitive sensitivity ± 0.002(nF / °C)	Resistive sensitivity ± 0.002(kΩ / °C)
Reference sensor	1.064	0.230
CuTsPc-TiO_2 sensor	5.548	0.098

(b)

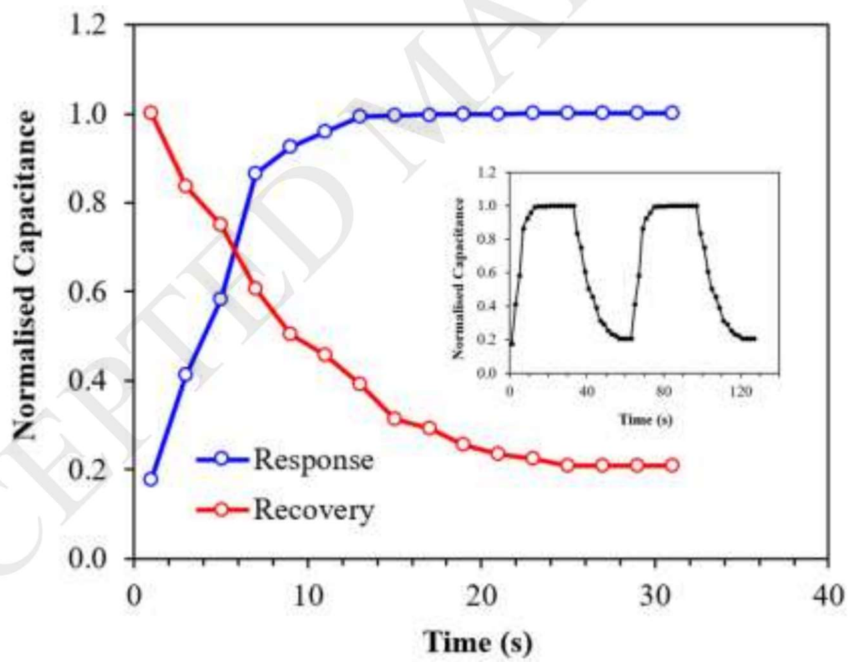
CuTsPc-TiO_2 Sensor	Response time (s) ± 2s	Recovery time (s) ± 2s
Capacitive output	8	15
Resistive output	20	50

The potential of the CuTsPc-TiO_2 composite based sensor was further evaluated in terms of response and recovery. **Figure 3a** and **b** depict the resistive (with normalized resistance at response's initial 10 kΩ) and capacitive (with normalized capacitance at recovery's initial 773 nF) modes, respectively. The cell exhibited very stable response/recovery behaviour and has given a rapid output in capacitive mode compared to its resistance measurements. The values of response and recovery time for both the cases are presented in **Table 1b**.

Here, the capacitive output value is much relevant for the temperature sensing applications typically below 100 °C, as it shows a reliable and consistent response/recovery time including a good sensitivity and a linear change in the capacitance as a function of change in temperature. Thus, the cell containing CuTsPc-TiO_2 nanofluid is very suitable to be applied as a capacitive temperature sensor in the range below 100 °C.



(a)



(b)

Figure 3: Response and recovery for CuTsPc-TiO_2 composite electrolytic based temperature sensor measured in **(a)** resistive and **(b)** capacitive modes.

Table 2: Comparison between the sensor with and without TiO_2

Sensor (type)	Response time (s)	Recovery time (s)	Reference
CuTsPc (Resistive)	20	30	[3]
CuTsPc-TiO_2 (Capacitive)	8	15	Current work

3.1 Mechanistic study on the effect of TiO_2 NPs on the temperature sensing mechanism

With regards to the capacitive sensing mechanism of this device, it is proposed that the permittivity of TiO_2 NPs changes upon temperature variations. According to what has been described in references [34-36], there is a general trend that the relative permittivity increases as temperature rises for TiO_2. Following the definition of the parallel plate capacitor (**Equation 3**)

$$C = \varepsilon\varepsilon_0 \frac{A}{d} \text{-------------- (3)}$$

where ε is the relative permittivity, ε_0 is the permittivity of vacuum, A is overlapping area, and d is the distance between the two plates), it is reasonable to deduce that the capacitance is proportional to the permittivity, which matches with the experimental observations in Figure 2. Moreover, it is noteworthy that owing to the fact that the results of the current work show very small hysteresis, indicating there is no chemical reaction or interaction between materials in the solution. The sensing mechanism is mainly based on the temperature dependence of the dielectric constant of TiO_2, which determines the capacitance based on equation 3. According to references [34-36], rising temperature will increase the dielectric constant, hence increasing the capacitance, which agrees with the experimental results.

In addition to the above mentioned correlation between the capacitance and permittivity as defined by eq. 3, the improved performance of the CuTsPc-TiO_2 composite electrolytic based temperature sensor is attributed to several other factors. Firstly, the TiO_2 NPs, owing to their high thermal stability, possess significant thermal effect on the carrier concentration and efficiency of the luminescence, which is highly desirable for temperature sensors [30]. Secondly, the increased sensitivity of the device is also attributed to the enhanced surface area caused by the inclusion of these NPs into the CuTsPc electrolytic solution [37]. In addition, distribution of TiO_2 NPs acted as an efficient interconnect (bridge) among the neighboring CuTsPc organic matrix which ultimately enhanced flow of electrons via hoping and hence it increased the electrons mobility, as a result the resistance has significantly reduced as evident in our results too. Further, it also leads to relatively faster response/recovery time because of higher carrier mobility of TiO_2 NPs [38]. Moreover, TiO_2 possesses higher relative sensitivities over a wide temperature range and very good temperature resolution [39]. Thus, the increased sensing response can be attributed to the synergistic effect of both TiO_2 and CuTsPc nanocomposite which remarkably improved the performance index.

4. Conclusions

In conclusion, we have successfully demonstrated a highly efficient electrochemical cell, worked as a low temperature sensor, based on CuTsPc organic electrolytic solution mixed with TiO_2 metal oxide, which showed strong temperature dependence on capacitance. By integrating the TiO_2NPs into the organic electrolytic solution, the sensitivity of the cell transformed into the capacitive sensitivity from originally the resistive sensitivity (as shown by reference cell). As a result, the capacitive sensitivity increased from 1.064 nF/°C to 5.548 nF/°C. Similarly, response/recovery time significantly improved from 20/50 sec in the resistive mode to 8/15 sec in capacitive mode. The composite sensor also showed very good linearity and least hysteresis. Such devices are appropriate to use for low-temperature applications with higher sensitivity and accuracy.

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