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Eu_2O_3 Coated Thick Film for Acid Citric Sensing

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Abstract

This study presents the development of a citric acid sensor based on a Europium Oxide-doped thick film, evaluated across varying concentrations. The device was fabricated via the screen-printing method, with Europium Oxide blended into an organic binder to optimize rheological properties for printing. Given that excessive intake of citric acid, a common dietary acid, can pose health risks, reliable detection methods are essential. The sensor's performance was assessed by measuring the output voltage in relation to citric acid concentrations ranging from 20% to 100%. Data acquisition was facilitated using an Arduino Nano for precise voltage readings. Results indicate a positive linear correlation between citric acid concentration and output voltage, achieving a linearity of 99.28% and a sensitivity of 0.0228 V per percent concentration. Comparative experiments with malic acid demonstrated the superior sensing characteristics of the citric acid sensor in terms of stability, repeatability, and hysteresis. Additionally, sensing tests were performed with malic acid for comparative analysis. The findings underscore the potential of Eu_2O_3 as an effective and economical material for acid sensing applications, offering a viable alternative for industrial and medical monitoring systems.

Keywords: Eu2O3; Thick Film Sensor; Citric Acid Detection; Screen Printing Technique; Acid Sensing Applications

1. Introduction

Over the past decade, thick film technology has seen substantial advancements within nanoscience and nanotechnology. Thick films consist of material layers with thicknesses spanning from a few nanometers to micrometers [1]. These films are typically produced using conductive polymers, which are preferred over glass substrates owing to their economic viability, flexibility, and capacity for tailored functionalities. Screen-printing methods are widely utilized in the fabrication of thick film sensors.

The coating of Europium oxide is well-known for its high sensitivity, selectivity, and stability, making it a promising nanomaterial for acid sensing applications. These nanoparticles are ferromagnetic semiconductors with a band gap of 1.12 eV, featuring a unique 4f electronic structure, remarkable optical properties, and enhanced catalytic activity, which makes them ideal for sensing purposes [2]. Without proper precautions, excessive consumption of citric acid can lead to serious health issues such as skin irritation, dental problems, allergic reactions, and fatigue. The use of Europium oxide coated thick films provides an accurate and reliable method for measuring acid levels. This approach offers a practical solution applicable in various fields, including healthcare [3].

Monitoring acid concentrations within the human body is essential, as these levels can profoundly influence biochemical reactions and biological systems. Blood acid concentration provides a rapid and practical means to assess whether blood pH deviates from optimal levels [4]. Overall, the Europium oxide-doped thick film sensor constitutes a notable advancement in acid detection technology. This innovation offers considerable potential to enhance public safety and health by addressing risks associated with excessive acid exposure in everyday products and food.

Citric acid is a prevalent acid encountered in everyday items like food and beverages, serving to improve flavor, regulate pH, and act as a preservative. Additionally, it finds applications in cosmetics and pharmaceuticals [5]. Excessive intake of citric acid can result in health concerns including dental erosion, gastrointestinal distress, and the potential for kidney stones. Furthermore, exposure to high concentrations may cause skin irritation, chemical burns, and heightened sensitization risks [6]. This project aims to benefit individuals by enabling them to determine the citric acid content in their products. In comparison to malic acid, citric acid offers advantages in electrical properties and chemical bonding. Both substances show promise for acid sensing applications using Europium oxide thick film sensors.

Existing acid sensing techniques remain limited in accessibility owing to their elevated costs, intricate fabrication processes, and inadequate sensitivity. Conventional materials such as titanium oxide and glass-based substrates present notable limitations, including high manufacturing expenses, prolonged production timelines, and suboptimal performance when detecting acid concentrations [7]. Europium oxide (Eu_2O_3) has shown considerable promise for electronic sensor applications due to its distinctive electronic characteristics.

This investigation represents the first reported instance of an Eu_2O_3-coated thick film sensor specifically engineered for acid detection. The work fulfills the necessity for an economical and straightforward system possessing superior sensitivity suitable for health monitoring purposes. Standard electronic sensors commonly depend upon glass substrates to support the sensing layer. Manufacturing such sensors necessitates several stages, such as cleaning, infrared roasting, surface oxidation, and vacuum coating intended to bolster adhesion and surface energy. These stages prove especially laborious and afford minimal adaptability. In comparison, the current endeavor leverages a thick film platform for the coating material, demanding markedly shorter preparation intervals than those required for glass substrates.

2. Literature Review

2.1. Eu_2O_3 Coated Thick Film Preparation

For the fabrication process, 0.5 g of ethyl cellulose and 9.5 g of terpinol were weighed with a digital mini scale to prepare an organic binder.The mixture was placed on a hotplate stirrer for 24 hours at 40 °C and 200 rpm to achieve a homogeneous blend [8]. In addition, 1 g of Europium oxide was combined with 1.43 g of the organic binder and stirred on a hotplate for 3 hours at 30 °C and 80 rpm to produce the Eu_2O_3 paste [9]. This preparation enhances the sensitivity, resistivity, and optimal operating temperature of the Europium oxide.

Furthermore, the preparation of the paste also incorporated a silver conducting paste. This paste was initially deposited onto the thick film substrate as the first layer of the sensor using the screen-printing method, followed by firing at 150 °C for 15 minutes [9]. Subsequently, the paste was applied onto the interdigitated electrode as the second layer via the screen-printing technique. The fabricated sensor was then annealed at 200 °C for 1 hour in an ambient atmosphere using a furnace [10]. Finally, copper wires were attached to the sensor with silver paste.

Table 1. Ratio of citric acid and distilled water.

Concentration	Ratio of citric acid and distilled water
20%	5g citric acid + 5 ml distilled water
40%	10g citric acid + 5 ml distilled water
60%	15g citric acid + 5 ml distilled water
80%	20g citric acid + 5 ml distilled water
100%	25g citric acid + 5 ml distilled water

Table 2. Ratio of malic acid and distilled water.

Concentration	Ratio of malic acid and distilled water
20%	5g malic acid + 5 ml distilled water
40%	10g malic acid + 5 ml distilled water
60%	15g malic acid + 5 ml distilled water
80%	20g malic acid + 5 ml distilled water
100%	25g malic acid + 5 ml distilled water

Eu_2O_3 Coated Thick Film Preparation

To evaluate acid concentrations, a spectrometer was utilized to analyze solutions prepared with varying amounts of citric and malic acid. Both acids were dissolved in distilled water to create the test solutions [11]. For both citric and malic acid, solutions were prepared using a fixed volume of 5 mL of distilled water across five mass increments: 5 g, 10 g, 15 g, 20 g, and 25 g. To ensure consistent comparisons, identical solution volumes were maintained throughout the experiment. The spectrometer was set to a wavelength of 550 nm to record absorption, transmission, and concentration data, which served to standardize the experimental protocol for future sensor validation. The experimental setup was designed to acquire data generated by the sensor during the detection of acids at varying concentrations. The copper wires from the sensor were connected to the input and ground pins of an Arduino Nano, as illustrated in Figure 1. This microcontroller was interfaced with a computer via a USB connection to monitor the voltage readings produced by the sensor. Firmware was uploaded through the Arduino Integrated Development Environment to configure the Nano as the sensor controller. Measurements were recorded multiple times to verify the reproducibility of the proposed sensor. Finally, sensing performance was evaluated based on several key metrics, including sensitivity, linearity, resolution, repeatability, hysteresis, stability, and standard deviation.These performance characteristics were systematically quantified by exposing the sensor to varying acid concentrations under controlled laboratory conditions, similar to the protocols established for evaluating conductimetric and resistive sensing architectures [12], [13].

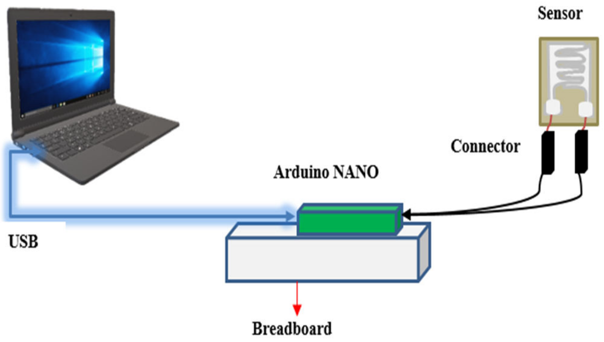

Figure 1. The experimental setup of acids sensing.

3. Sensing Mechanism

Citric acid with molecular formula $C_6H_8O_7$ will undergoes partial ionization when mixed with distilled water. The citric acid molecules will release one hydrogen and split into hydrogen ions (H+) and citrate ions $C_6H_8O_7$. The citric acid will undergo three stages of ionization. As the reaction enters a dynamic equilibrium, the degree of ionization is determined by the citric acid concentration and the pH of the solution [14]. Besides that, the release of hydrogen ions (H+) will cause the solution increase in acidity and low in pH.

Furthermore, malic acid with molecular formula $C_4H_6O_5$ is a weak dicarboxylic acid that will undergo partial ionization when mix with distilled water. There are two stages to this ionization, and each one represents the separation of a hydrogen ions from one of its two carboxylic acid groups[15]. The first stage malic acid will release hydrogen with molecular formula of malate ion $C_4H_6O_5$ and the second stage with malate ion $C_4H_6O_5$. The water's ion concentration rises as a result of this action, making the solution acidic.

The Europium oxide-coated thick film sensor detects acidic solutions via interactions with hydrogen ions that modulate its electrical properties [16]. These shifts correlate with acid concentration which higher concentrations induce increased hydrogen ion release, resulting in more pronounced signal changes. Captured by electrodes, these signals are transmitted to a measurement apparatus for real-time monitoring. Device response is ultimately governed by material composition, film thickness, and analyte characteristics.

4. Result and Discussion

Figure 2 (a) & (b) show the absorption of both acids by using spectrometer. Based on Figure 2 (a), the lower absorption value at the lower concentration was -0.19 and increase when concentration goes up to 100% reaching at -0.05. Besides that, based on Figure 2 (b), the absorption value is -0.30 when the concentration value at 20%. It increases significantly when the concentration goes up to 100% reaching at -0.21. A light beam is used to illuminate the sample, and each substance in the acid absorbs light with a 550nm wavelength. The likelihood of a photon being absorbed increases with the path-length of light that must pass through an acid solution before it reaches the detector[17].

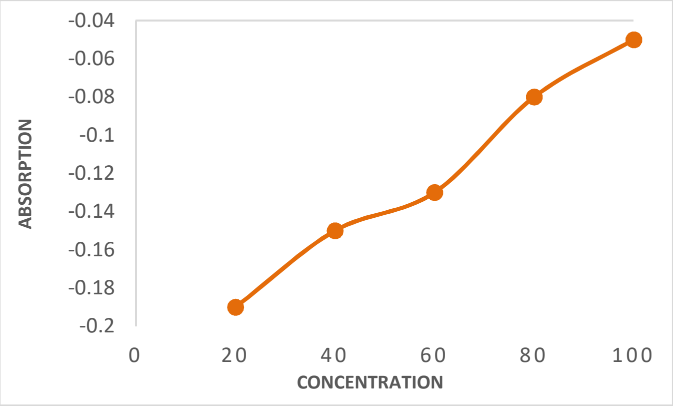

Figure 2 (a). Absorption; Citric Acid.

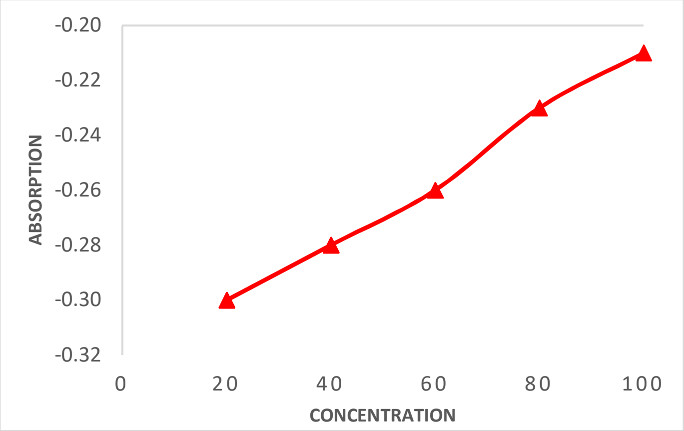

Figure 2 (b). Absorption; Malic Acid.

Figure 3 (a) shows the transmission of citric acid decrease steadily from 20% to 100%. The transmission is 99.8 at 20% concentration. It goes up until 97.7 at concentration 100%. Besides that, based on Figure 3 (b), when malic acid at lower concentration which is 20%, the transmission value is 107.1. It decreases linearly until the concentration of malic acid at 100% which is the transmission at 105. The transmission is the uabsorbed light that detect by the detector after passing through the sample [18].

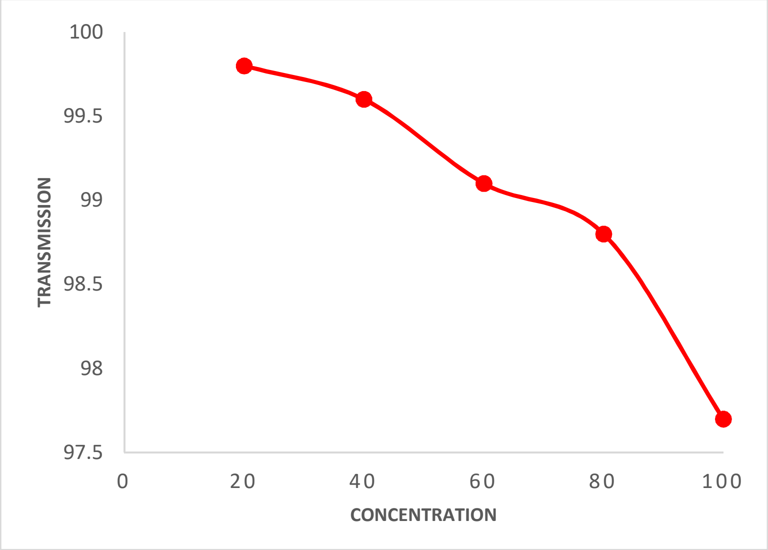

Figure 3 (a). Transmission; Citric Acid.

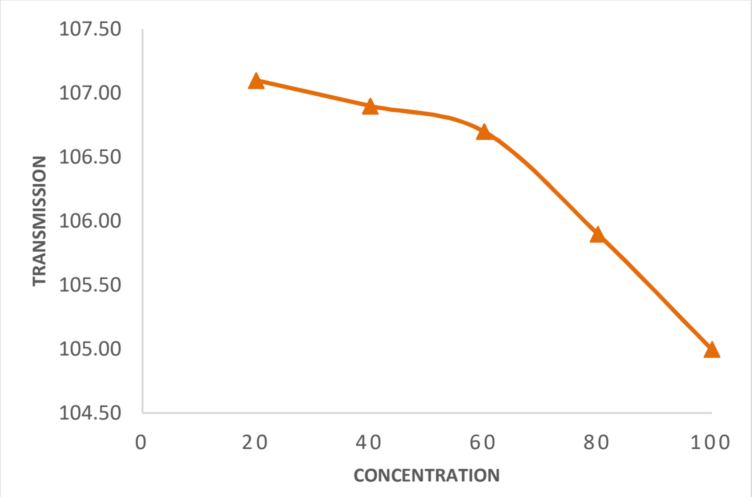

Figure 3 (b). Transmission; Malic Acid.

Figure 4 (a) illustrates that the measured value is directly proportional to the percentage of citric acid, exhibiting a linear increase from 20% to 100%. At an initial concentration of 20%, the recorded value is -20, which rises to -3 as the percentage reaches 100%. This trend indicates that higher acid percentages facilitate increased concentrations, attributed to the higher volume of citric acid within the distilled water. Furthermore,

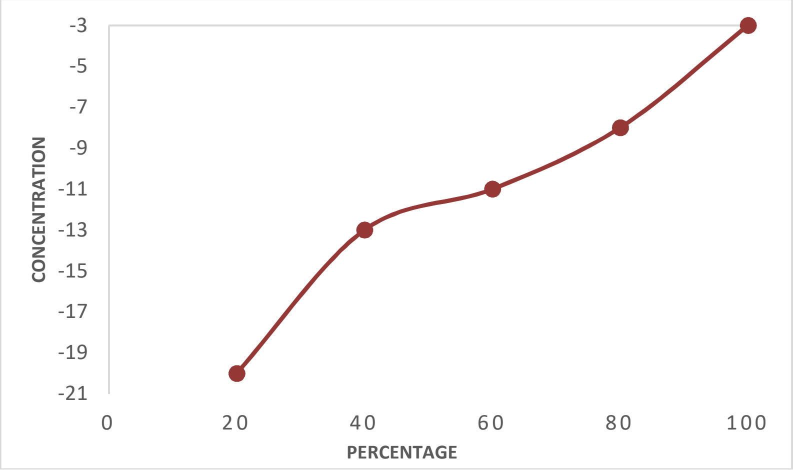

Figure 4 (a). Concentration; Citric Acid.

Figure 4 (b) reveals a similar direct proportionality for malic acid, with values rising from -32 at 20% to -20 at 100%. Establishing precise concentrations across varying ratios is critical for ensuring experimental accuracy. By utilizing a 550 nm wavelength to measure intensity, the system determines solution concentration in accordance with the Beer-Lambert Law, which establishes a linear relationship between absorbance and concentration which consequently, higher absorption values correspond to elevated acid concentrations[19]

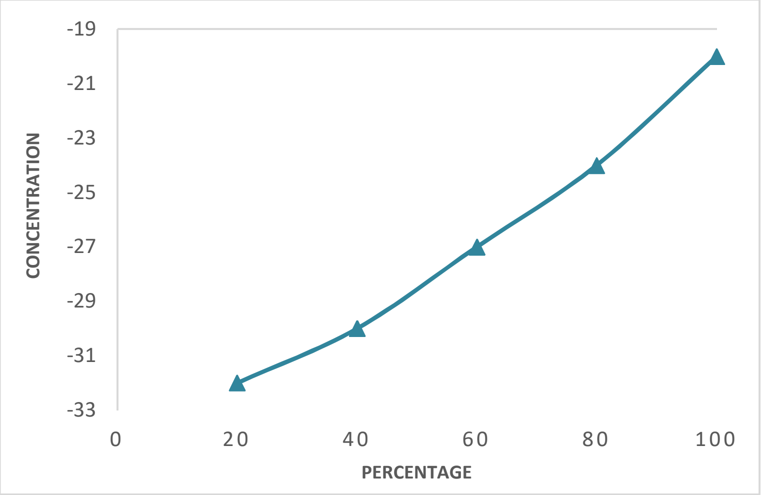

Figure 4 (b). Concentration; Malic Acid.

Figure 5 shows the experiment tested citric acid and malic acid with a concentration ranging from 20% to 100%, increasing in 20% increments. Figure 5 (a) shows a positive linear correlation between citric acid concentration and average output voltage. The voltage increased by 0.0228V per 1% concentration increase, starting from 20% at 10.28V until reaching 100% at 12.14V. This indicates that higher concentrations improve output voltage due to enhanced material properties or system efficiency. Europium oxide was a sensing material that works especially well for detecting citric acid because of its special qualities, which include its catalytic performance and ferromagnetic semiconductor features [20]

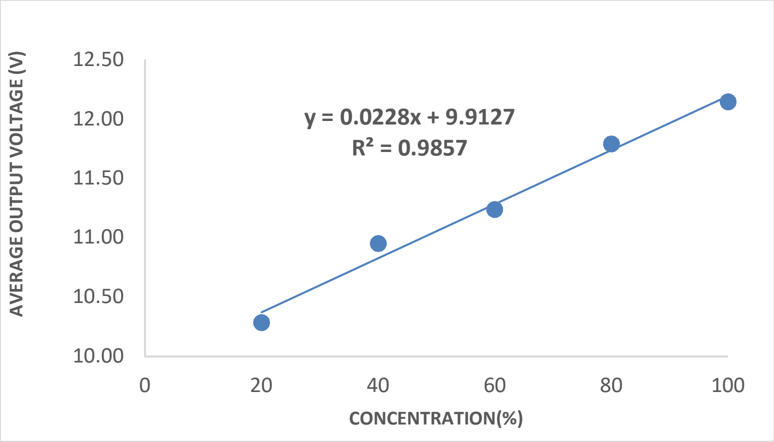

Figure 5 (a). Average output voltage for acids; Citric Acid.

Figure 5 (b) shows the average output voltage is directly proportional to the concentration of malic acid, increasing by 0.0214V per 1% concentration. Atrting at 20% concentration at 11.95V, the voltage rises to 13.74V at 100%. The exceptional water solubility of malic acid enhances its versatility.

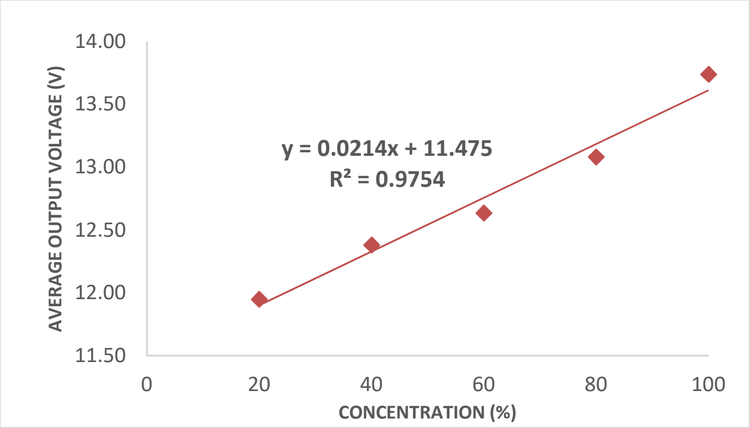

Figure 5 (b). Average output voltage for acids; Malic Acid.

Table 3 summarizes the sensing performances of both samples. citric acid outperforms malic acid in terms of total sensing performance. Acid citric is 1.1 times more sensitive than malic acid. The resolution of citric acid was found to be 2.9 times improved, and standard deviation 2.7 times improved than malic acid with the percentage of linearity higher than malic acid.

Table 3. Sensing performance of the sensors.

Parameters	Citric Acid	Malic Acid
Linearity (%)	99.28%	98.76%
Sensitivity (dBm/%RH	0.0228	0.0214
Standard deviation (dBm)	0.0596	0.1618
Resolution (%RH)	2.6158	7.5591

Figure 6 (a) shows the steady output voltage value show that the Europium oxide coated thick film has outstanding repeatability for sensing citric acid. The molecular structure and behavior of citric acid made it easy for ionizing and releasing hydrogen ions [14]. The sensor's small variance indicates that it performs consistently and demonstrating its dependability for citric acid detection.

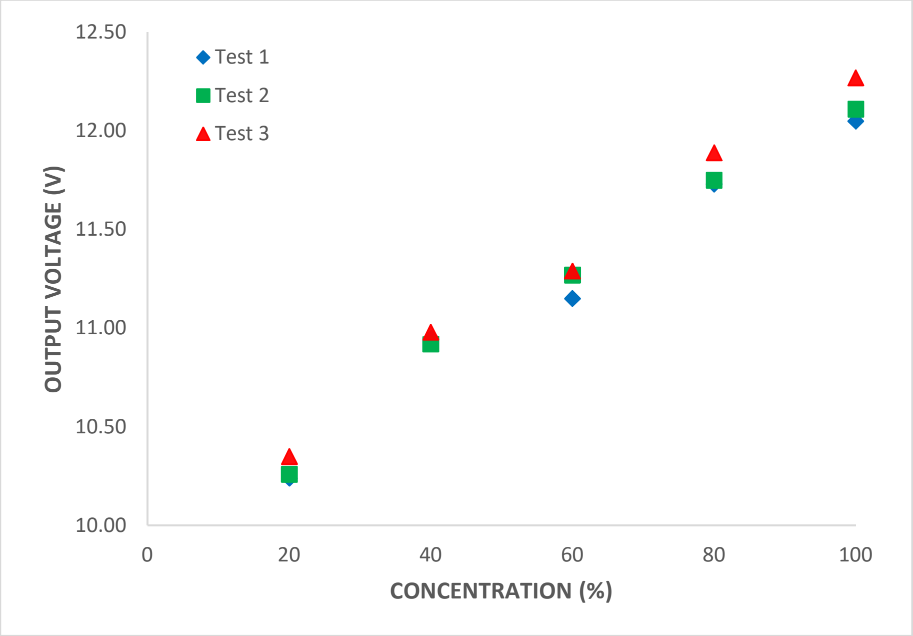

Figure 6 (a). Repeatability for acids; Citric Acid.

Based on Figure 6 (b), Malic acid's higher output voltage fluctuation indicates that sensor's performance is less stable when detecting this acid. Less accurate sensing occurs from variations in the chemical interactions between malic acid and the Europium oxide layer [21]. The gap of output voltage of malic acid for repeatability was enormous compare to the output voltage of citric acid.

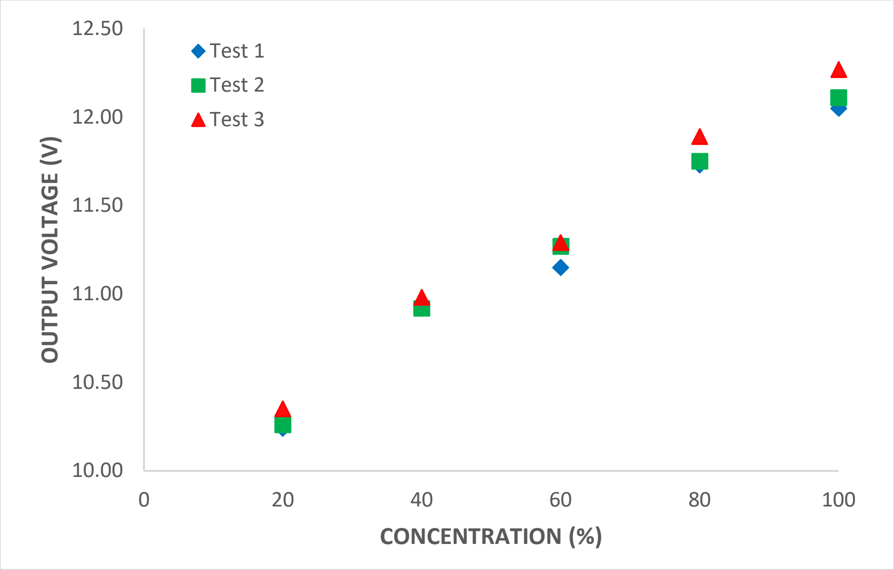

Figure 6 (b). Repeatability for acids; Malic Acid.

Figure 7 (a) and (b) shows the hysteresis of citric acid and malic acid applied to Europium oxide coated thick film sensor. The sensory response was tested by increasing the concentration both acid and reducing the concentration from 100% to 20%. According to Figure 7 (a), citric acid produces a different trend line pattern and show a slightly different on the output voltage measurement.The significant change of output voltage change exists between forward and reverse measurement on a malic acid as in Figure 7 (b). This indicates that the recommended acid shows more consistent hysteresis behavior in both forward and reverse measurements due to nearly similar output voltage.

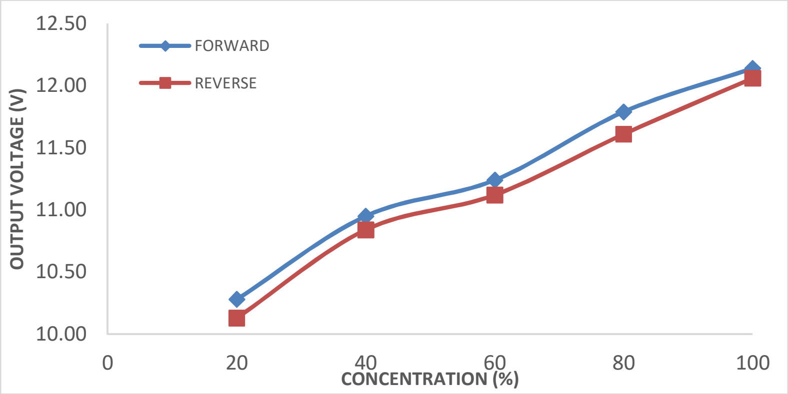

Figure 7 (a). Hysteresis for acids; Citric Acid.

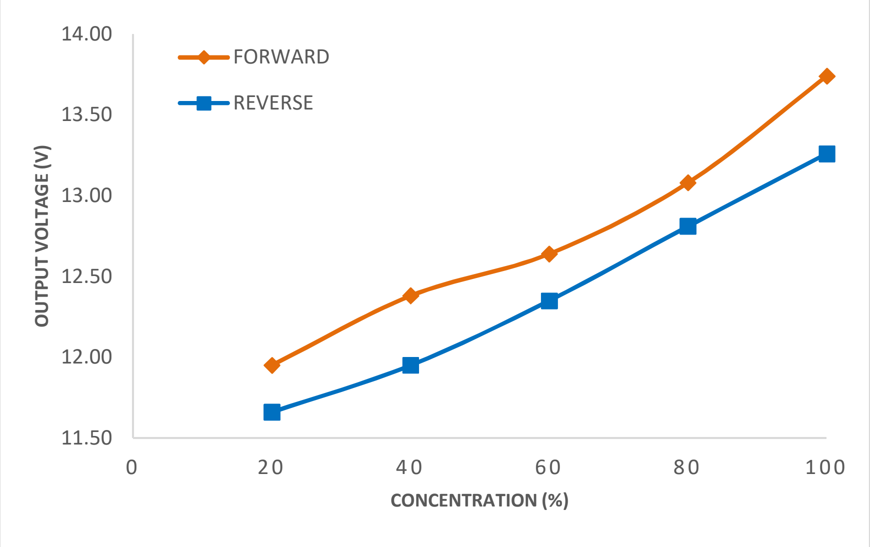

Figure 7 (b). Hysteresis for acids; Malic Acid.

Figure 8 (a) and (b) illustrates the output voltage versus time on the Europium oxide coated thick film for citric acid and Malic acid from 20% to 100%. The citric acid and malic acid stability was evaluated by storing output voltage data for every 30s for a total of 15 minutes which is 900s [22]. According to Figure 8 (a), the stability of citric acid on Europium oxide coated thick film significantly increased throughout the 900s period. The higher of concentration citric acid affect the improvement of electrical conductivity that cause citric acid more stable [14]. The result also shows that the output voltage recorded on thick film coated Europium oxide for malic acid in Figure 8 (b) was not stable especially at 40% and 60% for the whole 900s. Based on both figures, citric acid was more stable compare to the malic acid on Europium oxide coated thick film.

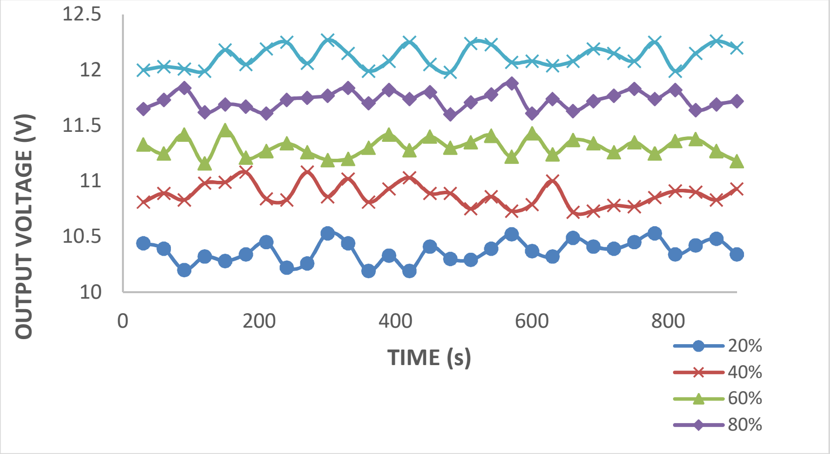

Figure 8 (a). Stability for acids; Citric Acid.

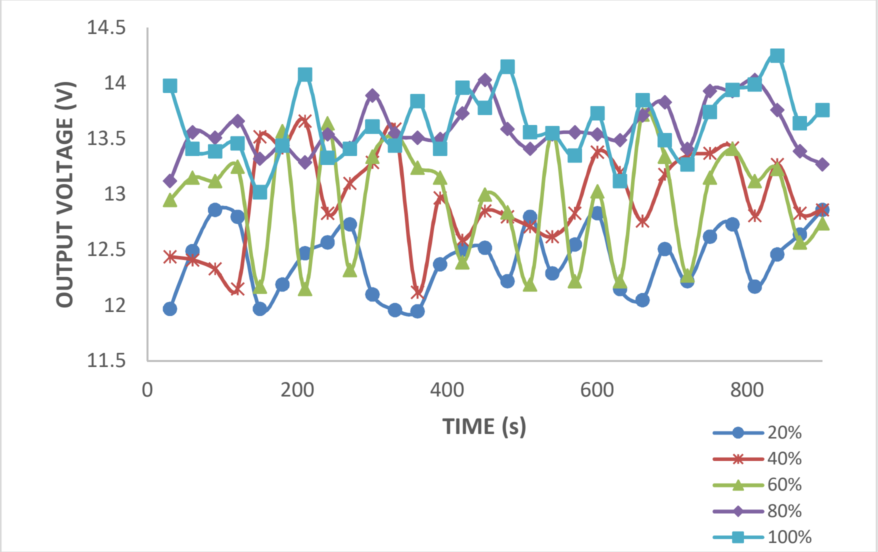

Figure 8 (b). Stability for acids; Malic Acid.

5. Conclusion

An acid sensor has been successfully demonstrated by developing a Eu2O3 coated thick film for acid sensing application using a simple and cost-effective screen-printing method. The result of experiment demonstrates that citric acid with linearity 99.28% has the best sensitivity when compared to the malic acid which is 98.76%. An acid sensor is effectively shown by applying citric acid to thick film coated Europium oxide. The resolution improved by a factor of 2.9 and the citric acid sensitivity rode by 1.1 when compared to the malic acid, with a standard deviation that was 2.7 times better. In comparison to malic acid, Europium oxide demonstrated improved performance in citric acid detection, indicating its promise as a dependable material for sensing application. Besides that, the findings demonstrated the sensor’s reliable operation by confirming a positive linear connection between output voltage and citric acid concentration. Its dependability for practical used was further shown by its repeatability and minimal hysteresis. Several enhancements should be taken into consideration for future work to improve the existing study on the fabrication of Europium oxide onto thick film. First, the uniformity and performance of the films may be greatly enhanced by refining the manufacturing processes, such as exact temperature control and thickness optimization. Finally, investigating different acids or acid mixes, especially environmentally friendly acid, might lead to better result.

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