ARTIKEL RISET

Study of Salty Taste Sensor Based Graphite PVC Electrode Modified with Polypyrrole-Carbon Black (PPy-CB)

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Abstract

A successful salty taste sensor was developed using solid-contact electrodes with graphite PVC composite substrates. The fabricated sensor was characterized using cyclic voltammetry (CV) and electrochemical impedance spectroscopy (EIS). The characterization results showed the addition of polypyrrole-carbon black (PPy-CB) material increased the capacitance, conductivity, and enlarged the graphite PVC electrode surface area. The selectivity of the sensor was measured on an electrode that has been coated with a lipid membrane mixture of tetradodecyl ammonium bromide (TDDAB) and 1-hexadecanol against basic taste samples using the impedimetric method and processed with PCA analysis. The result showed good salty taste selectivity with a silhouette coefficient value of 0.90. The constructed sensor demonstrated linear behavior towards standard salty substances (NaCl) with a sensitivity of -4.36 kΩ/decade and R2 = 0.99 in the sensing range of 0.1 to 30 mM. The study's findings suggest that graphite PVC electrodes modified with PPy-CB and lipid membrane combined with the impedimetric measurement method exhibit good selectivity and sensitivity in detecting salty taste.

Keywords: taste sensor; lipid membrane; solid-contact electrode; impedimetric; polypyrrole-carbon black; salty sensor

1 INTRODUCTION

Salty taste is one of the five basic tastes that can be detected by the human tongue, along with sweet, sour, bitter, and umami. The presence of salty taste is often associated with mineral salt content such as sodium chloride (NaCl) and has been widely used in the food industry as a preservative, flavor enhancer, and to increase water absorption in food [1]. However, excessive addition of salt can cause health problems such as high blood pressure and stroke [2]. Therefore, the quality of salty taste in food needs to be evaluated to determine the quality and standards of the resulting products.

Conventional methods for evaluating salty taste involve the use of trained human panelists. However, this method cannot provide quantitative taste differences and is prone to subjectivity, as well as having low reproducibility due to sensory fatigue [3]. Another alternative approach is to use taste sensors. Taste sensors consist of sensing instruments and ion-selective lipid membranes used to detect the taste of specific taste substances based on the electrochemical mechanism between the membrane and the test solution. Lipid membranes can behave similarly to human gustatory cells, where the electrochemical response of the membrane can be used to extract taste information [4]. Several decades of research and development through experimentation with various lipid membrane compositions have eventually led to the development of specific membranes that selectively respond to basic tastes, such as salty, sour, sweet, umami, bitter, and also astringent [5].

In their application, almost all taste sensors adopt a liquid-contact electrode design consisting of a combination of Ag/AgCl electrode and inner filling solution as an ion-electron transducer [6]. In taste sensor applications, liquid-contact electrodes demonstrate good capability in measuring primary ion activity in various types of samples. However, these electrodes have complexities in attaching the membrane to the electrode body and require maintenance of the filling solution during storage [7]. Solid-contact ion-selective electrodes are ion-selective electrodes developed without the use of an inner filling solution. These electrodes utilize solid materials as

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ion-electron transducers, which are then coated with a membrane or other sensing layers [8]. The use of these electrodes overcomes the limitations of conventional liquid-contact electrodes and offers advantages in many aspects, including lower cost, reduced material usage, adjustable measurement positions, and rapid fabrication. Moreover, solid-contact electrodes also enable sensor miniaturization for greater compatibility [9].

Solid-contact electrodes are made using conductive substrates such as glassy carbon, graphite, gold, platinum and more. Another potential material as a conductive substrate for solid-contact electrodes is the graphite-PVC composite. This material has good mechanical resistance and stability, is economical and easy to produce, and has a readily renewable surface area [10]. In recent years, the development of solid-contact electrodes has involved functional materials as ion-electron transducers, including conductive polymers and nanomaterials. These materials are used to enhance the double-layer capacitance between the electrode and the membrane, positively impacting the quality of electrochemical measurement responses. Recent studies have found that the use of polypyrrole-carbon black (PPy-CB) composite material can improve the performance of solid-contact sensors for taste detection, with good stability and performance [7].

The process of measuring the response between the electrode and the test solution in this research is using impedimetric method. The principle of the impedimetric method is works by measuring the impedance change between the working electrode and the test sample. The results of this measurement can be displayed in Nyquist or Bode plots [11] and provide more complex information about ion transfer and charge phenomena at the electrode surface with the test solution [12]. The use of impedimetric methods in the field of sensory research has been widely applied in various research applications, ranging from sensors for detecting ions in solutions to detecting biological specimens such as antigens and antibodies [12][13][14]. In some studies, impedance data analysis is combined with chemometric machine learning analysis methods, such as principal component analysis (PCA), linear discriminant analysis (LDA), and cluster analysis (CA) [15].

Based on the description above, we conducted a study on solid-contact electrode-based sensors for the detection of salty taste. The sensor electrode was fabricated using graphite PVC composite material, which was then coated with a conductive polymer, PPy-CB. The electrochemical properties of the formed electrode were characterized using cyclic voltammetry (CV) and electrochemical impedance spectroscopy (EIS). The formed electrode was subsequently coated with a lipid membrane solution composed of tetradodecyl ammonium bromide (TDAB) and 1-hexadecanol, which was deposited on the electrode surface using the drop casting method. The salt taste sensor's selectivity was validated by testing EIS on basic taste samples and evaluating its selectivity using principal component analysis (PCA). Additionally, the sensor's sensitivity was evaluated for salty taste components, NaCl. Thus, this study aims to develop a lipid membrane-based sensor with good selectivity and sensitivity for the testing of salty taste samples.

2 MATERIALS AND METHODS

2.1 Materials

PPy (doped with organic sulfonic acid anion) composite with CB at 20% wt loading (PPy-CB), di-n-octyl phenylphosponate (DOPP), tetradodecylamonium bromide (TDAB), 1-hexadecanol, polyvynil chloride (PVC), L(+)-tartaric acid, L-glutamic acid monosodium salt hydrat (MSG), quinine monohydrocloride dihydrate and tetrahydrofuran (THF) were purchased from Sigma-Aldrich (USA). Potassium chloride (KCl), sodium chloride (NaCl), potassium chloride (KCl), potassium nitrate (KNO3), and N,N-dimethylformamide (DMF) were purchased from Merck. Graphite powder was purchased from Dingshengxin (China). Alumina slurry (0.3 μm and 0.05 μm) were purchased from Electron microscopy science (Hatfield, PA, USA). All solutions were prepared using distillated water.

2.2 Methods

Fabrication of Salty Taste Sensor

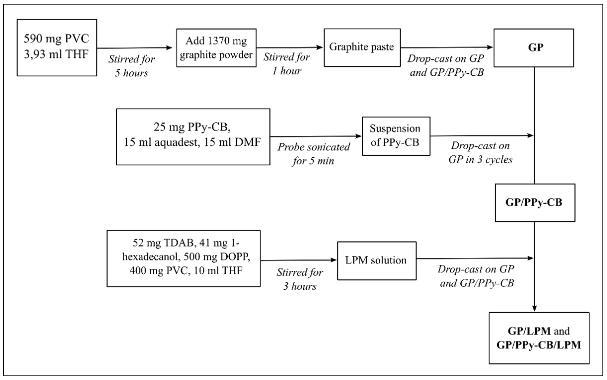
The sensor fabrication process consists of four stages: 1) creating the graphite-PVC paste, 2) preparing the conductive polymer PPy-CB suspension, 3) producing the lipid membrane, and 4) fabricating the electrode. A schematic representation of the sensor fabrication process can be seen in Figure 1.

Preparation of PVC graphite paste

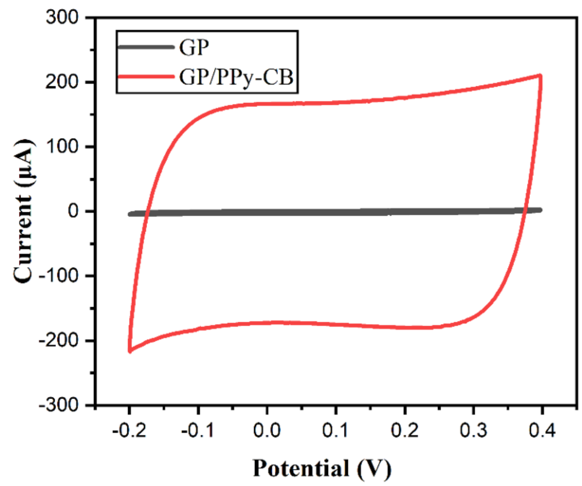
Pasta grafit PVC was prepared by dissolving 590 mg of PVC in 3.93 mL of THF. The mixture was stirred for 5 hours using a magnetic stirrer at a temperature of 40–50 °C. After complete mixing, 1370 mg of graphite powder was added and stirred again until homogenized.

Preparation of PPy-CB suspension

A Ppy-CB suspension was prepared in 1:1 (v/v) water:DMF. A mixture of 1:1 water:DMF was



Gambar 1: Diagram of sensor fabrication



Gambar 2: The cyclic voltammograms of GP and GP/PPy-CB electrodes in a 100 mM KCl solution with a scan rate of 50 mV/s are shown in the figure

prepared in a volume of 30 mL, and then 25 mg PPy-CB was added. Finally, a suspension was obtained after ultrasonication using Intelligent Ultrasonic Generator (19.9 kHz) of the mixture for 5 minutes. The suspension was stored at $2-8\,^{\circ}$C to use.

Preparation of lipid/polymeric membrane (LPM)

The LPM solution was synthesized by dissolving lipid components consisting of TDAB (5.24%) and 1-Hexadecanol (4.13 wt%), plasticizer DOPP (50.35 wt%), and PVC (40.28 wt%) in THF to form a 10 wv% lipid solution. Specifically, 52 mg of TDAB, 41 mg of 1-Hexadecanol, 500 mg of DOPP, and 400 mg of PVC were dissolved in 10 ml of THF. The mixture was stirred with a magnetic stirrer at a temperature of $35-45\,^{\circ}$C until it became homogeneous.

Preparation of salty taste sensor

The salt taste sensor was fabricated through several fabrication stages as follows: Firstly, the fabrication of graphite PVC (GP) electrode was conducted. The sensor electrode was made by coating the graphite PVC paste onto the surface of copper (Cu) electrode with a thickness of 5 mm, then dried overnight. The dried graphite PVC electrode was then polished using 0.3 μm and 0.05 μm alumina slurry on a microfiber cloth. The graphite PVC electrode was subsequently washed using distilled water, sonicated in distilled water for 10 minutes, sonicated in ethanol for 10 minutes, rinsed again with distilled water, and dried at room temperature. Next, 30 μL of Ppy-CB suspension was dropped onto the surface of GP electrode every 4 hours for 3 times repetition (GP/PPy-CB). After the PPy-CB deposition was dried, 30 μL of LPM was dropped onto the surface of GP/PPy-CB electrode to obtain GP/PPy-CB/LPM electrode. Additionally, LPM was also coated onto the surface of GP electrode without PPy-CB suspension (GP/LPM) as a comparison see in Figure 2

Electrochemical Characterization

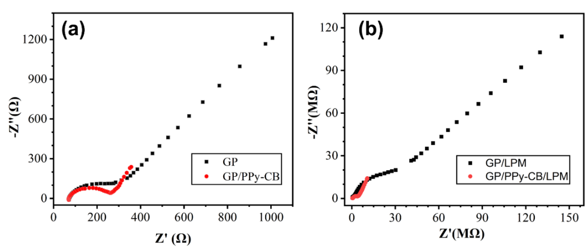
The formed electrode was characterized using cyclic voltammetry (CV). CV was carried out using a three-electrode system with GP and GP/PPy-CB electrodes as the working electrodes, Hanna HI 5315 as the reference electrode, and a Platinum plate as the counter electrode. CV was then run at a potential range of -0.2 V to 0.4 V with a scan rate of 50 mV/s for 3 cycles. After the CV measurement, impedance spectrocopy were performed using a three-electrode system, similar to the CV measurement, on GP, GP/LPM, GP/PPy-CB, and GP/PPy-CB/LPM as the working electrodes. The frequency range set at 1 Hz to 100,000 Hz with an AC voltage of 300 mV and a DC voltage of 0 V versus RE. Prior to the measurement, GP/PPy-CB and GP/PPy-CB/LPM electrodes were conditioned by soaking them in a reference solution (30 mM KCl + 0.3 mM tartaric acid) for 4 days

Sensor Selectivity and Sensitivity Testing

Testing the selectivity and sensitivity of the salty taste sensor using the impedimetric measurement method by measuring the electrode impedance in the frequency range of 1-100,000 Hz with an AC voltage of 300 mV and DC voltage of 0 V versus OCP. The measurement is repeated 5 times. For selectivity test, the electrodes GP, GP/LPM, GP/PPy-CB, and GP/PPy-CB/LPM were tested using the basic taste samples in Table 1. The data was collected by performing five repetitions, and the EIS data were analyzed using PCA analysis. In this study, PCA was used to reduce the size of the obtained data into a smaller number of data with new variable size, which is called the principal component. After that, the sensor sensitivity was evaluated by observing the impedance response of the GP/LPM and GP/PPy-CB/LPM electrodes to NaCl samples with various concentrations ranging from 0.03 mM to 1000 mM. All sampels inclueded 1 mM as the supporting electrolyte.

3 RESULT AND DISCUSSION

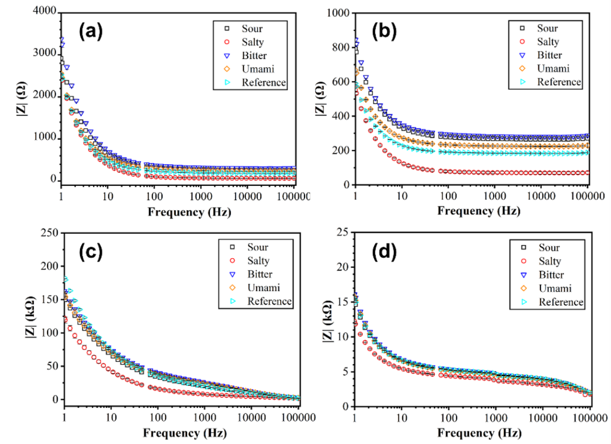
Electrochemical Characterization



Gambar 3: The Nyquist plot of EIS measurements for (a) GP and GP/PPy-CB electrodes and (b) GP/LPM and GP/PPy-CB/LPM electrodes

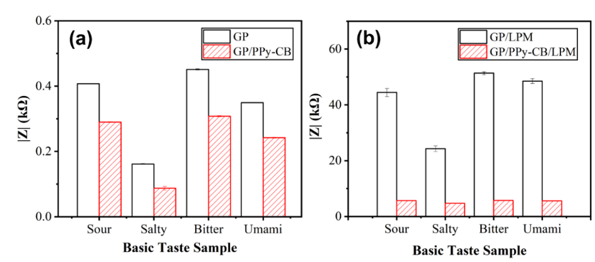
The cyclic voltammetry measurement before the disposition of the membrane aims to see the characteristics of the solid contact electrode and the result shown in Figure 3. The cyclic voltammogram curves formed indicate the presence of a non-faradaic mechanism, where the reaction is dominated by double-layer capacitance [16]. The difference in the areas of the graphs between the two electrodes demonstrates the variation in capacitance values. Considering the relationship between voltage and capacitive current, it can be identified that the GP/PPy-CB electrode exhibits a significantly higher capacitive current compared to the GP electrode. The capacitance comparison at 0 V reveals that the GP/PPy-CB electrode has a capacitance value 400 times greater than that of the GP electrode. The increased capacitive value in the GP/PPy-CB electrode is attributed to the presence of the carbon black component in the PPy-CB material, which possesses higher capacitance and conductivity values compared to graphite material [7].

The impedance measurement results for the GP, GP/PPy-CB, GP/LPM, and GP/PPy-CB/LPM electrodes are presented in Figure 4. Figure 4 shows the Nyquist plots obtained from the measurements, in which semi-circles or arcs represent the electrochemical system's impedance response to sinusoidal oscillating signals. Nyquist plots comprise two impedance components: the real part (Z') on the x-axis and the imaginary part (Z") on the y-axis. The real component



Gambar 4: Bode plots of electrode selectivity testing of (a) GP, (b) GP/PPy-CB, (c) GP/LPM, and (d) GP/PPy-CB/LPM on basic taste samples using the impedimetric method

(Z') arises due to the influence of resistance during the measurement process, originating from the electrode, electrolyte, or electrode-electrolyte interactions, and dominates at lower frequency ranges. Conversely, the imaginary component (Z") is caused by inductance, capacitance, and mass diffusion processes (Warburg impedance). However, in this study, Z" exhibits negative values, indicating the predominance of capacitance contributions as the largest impedance contributors. This phenomenon occurs in regions with high frequencies [17].



Gambar 5: Impedance response $|Z|$ electrodes of (a) GP and GP/PPy and (b) GP/LPM and GP/PPy-CB/LPM in the basic flavor sample at 35.6 Hz

The Nyequist plot was then fitted using the CS Studio6 software, the result indicated that the electrodes are influenced by the solution resistance (RS) and the bulk electrode resistance (Rbulk). The GP electrode exhibits a solution resistance of 72 Ω and an electrode resistance of 193 Ω, while the

Tabel 1: Components of samples solution for basic taste

Basic taste samples	Compositions
Reference solution	30 mM KCl + 0.3 mM tartaric acid
Sour	30 mM KCl + 3 mM tartaric acid
Salty	300 mM KCl + 0.3 mM tartaric acid
Bitter	30 mM KCl + 0.3 mM tartaric acid + 0.1 mM quinine
Umami	30 mM KCl + 0.3 mM tartaric acid + 10 mM monosodium glutamate

GP/PPy-CB electrode displays a solution resistance of 71 Ω and an electrode resistance of 184 Ω. These findings demonstrate that the addition of PPy-CB material to the GP electrode surface results in a decrease in electrode resistance. This effect is also observed for membrane-coated electrodes. The GP/LPM electrode has a solution resistance of 672 Ω and an electrode resistance of 58,900 Ω, whereas the GP/PPy-CB/LPM electrode exhibits a solution resistance of 451 Ω and an electrode resistance of 1,819 Ω.

The results indicate the presence of a capacitance circuit (C), which suggests the formation of double-layer capacitance (Cdl) due to the membrane on the electrode surface. The capacitance values obtained are 0.05 μF for the GP/LPM electrode and 200 μF for the GP/PPy-CB/LPM electrode. Moreover, there was a significant change in impedance values in the low-frequency region, which is a characteristic of the solid contact sensor. The absolute impedance value of GP and GP/PPy-CB electrodes at a frequency of 1 Hz was 1,575 Ω and 430 Ω, respectively, while the addition of a membrane reduced the impedance to 189 kΩ and 18 kΩ, respectively. This indicates that the addition of PPy-CB significantly enhances the capacity of the solid contact layer and reduces ion transfer between the electrode and test solution [7].

***Sensor Selectivity on Basic Taste Samples* Measurement with impedimetric method**

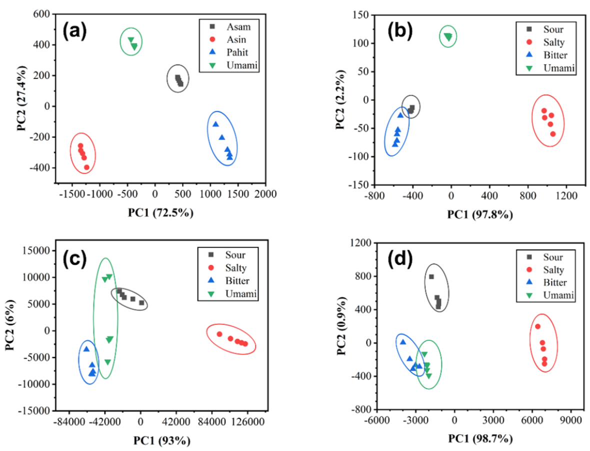
The sensor measurement results using the impedimetric method for the basic taste samples are displayed as Bode plots in Figure 4. These results show that all four sensors exhibit similar response patterns to each of the basic taste samples, with the impedance response differences more clearly demonstrated by the GP/PPy-CB electrode (Figure 5.b). Based on Figure 5.b, it is evident that each sample generates Bode plots with varying absolute impedance $|Z|$ values, with the impedance values arranged from lowest to highest as follows: salty, umami, sour, and bitter. This variation is influenced by the concentration of ions in the test solution; higher ion concentrations result in smaller impedance values and vice versa. The KCl component in the salty sample has stronger electrolytic properties compared to the MSG component in the umami sample and the tartaric acid in the sour sample, which tend to exhibit weak electrolytic properties. Meanwhile, the quinine component in the bitter sample tends to be covalent, resulting in a larger impedance value compared to the other samples [18].

The sensor response differences to the basic taste samples were further investigated by taking the impedance $|Z|$ value at a single frequency that produced an optimal impedance response, specifically at a frequency of 35.6 Hz, and displayed in a diagram in Figure 5. The diagram shows that the addition of the membrane results in an increase in the impedance value, with the GP electrode response rising from 161 Ω to 24,299 Ω for the GP/LPM electrode in the salty taste sample. However, the addition of PPy-CB material increases the conductivity of the electrode and lowers the resistance value, resulting in a decrease to 88 Ω for the GP/PPy-CB electrode and 4,729 Ω for the GP/PPy-CB/LPM electrode.

The impedance measurement results in Figure 5 also show that the responses generated by the GP and GP/PPy-CB electrodes display noticeable differences in sensor responses between each basic taste sample. However, for the GP/LPM and GP/PPy-CB samples, there is a more striking difference in the salty taste sample response compared to the other taste samples, which tend to have similar sensor responses. This is due to the lipid membrane coating on the electrode surface, which only allows specific ions to pass through. The use of a membrane consisting of TDAB + 1-hexadecanol, which is a type of negative membrane, only permits the passage of negative ions (in this case, Cl-). The salty taste sample has the highest number of negative ions among the other samples [19]. This indicates that the lipid membrane coated on the electrode surface tends to show selectivity towards salty taste, which is further confirmed by PCA analysis.

Principal Component Analysis (PCA)

PCA analysis is used to obtain a visualization of the sensor response that shows a tendency towards selectivity for a particular taste. The data used in the PCA analysis process consists of the impedance sensor response to basic taste samples (salty, sour, umami, and bitter). The response data consists of frequency, impedance, and phase difference from 50 data points with 5 repetitions. The principle of PCA is to cluster



Gambar 6: PCA plot of GP, (b) GP/PPy-CB, (c) GP/LPM, and (d) GP/PPy-CB/LPM in basic taste samples

Tabel 2: Silhouette coefficient value

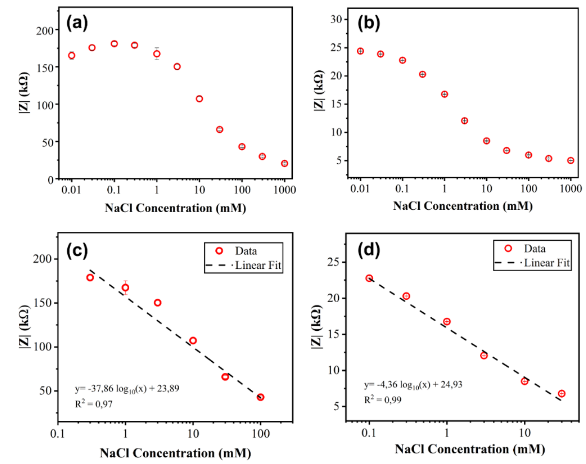
Sensor electrode	Silhouette coefficient
GP	0.59
GP/PPy	0.83
GP/LPM	0.84
GP/PPy-CB/LPM	0.90

the data taken. The number of data repetitions will become the points in each cluster, and each cluster indicates a taste group. The PCA results are displayed with PC1 and PC2, which show the main variation components on the x-axis and y-axis, respectively, as shown in Figure 6.

Based on the PC1 and PC2 plot of all sensors, it can be seen that the total variance is good, reaching approximately 99%, indicating that the sensors can differentiate between different tastes. In the spatial distribution of the PCA graph, there is a clear separation between the taste clusters on the GP and GP/PPy electrodes. Meanwhile, the GP/LPM and GP/PPy-CB/LPM electrodes show a tendency towards grouping for umami, bitter, and sour tastes. The degree of separation between the salty taste group and the other taste groups is determined by calculating the silhouette coefficient [20]. The silhouette coefficient values in Table 2 indicate that the electrodes coated with a membrane have the highest values, with the highest degree of separation found on the GP/PPy-CB/LPM electrode, which has a coefficient value of 0.90. This indicates that the addition of a lipid membrane provides good selectivity for the salty taste.

Sensor Sensitivity to Salty Taste

The impedance sensor response measurements on the GP/LPM and GP/PPy-CB/LPM electrodes to NaCl



Gambar 7: P The relationship between NaCl concentration and sensor impedance response (a) GP/LPM and (b) GP/PPy-CB/LPM at a frequency of 35.6 Hz with the results of fitting the linearity graphs of (c) GP/LPM and (d) GP/PPy-CB/LPM to determine the sensitivity of the sensor

solutions with varying concentrations are displayed in Figure 7 (a) and (b). Both sensors show a tendency of decreasing impedance values with increasing NaCl concentration. This decrease in impedance values is due to the increase in the concentration of ions in the solution as the concentration of NaCl increases, which leads to an increase in the solution conductivity. This increase causes more Cl- ions to be detected by the membrane layer, thus reducing the impedance values. The figure also shows a region with a pattern that indicates the sensor's working range, where the sensor has high sensitivity in detecting changes in NaCl concentration. The working range of the GP/LPM electrode is in the concentration range of 0.3-100 mM, while that of the GP/PPy-CB/LPM electrode is in the range of 0.1-30 mM. This indicates that the addition of PPy-CB material on the surface of the GP electrode results in a shift in the working range of the sensor towards lower concentrations.

The sensitivity values of the sensor are determined by calculating the gradient of the sensor graph with respect to the impedance change in the sensor's working area, and the results are shown in Figure 7 (c) and (d). The fitted graph results show that the sensitivity value of the GP/LPM electrode is about -37.86 kΩ/decade, while that of the GP/PPy-CB electrode is at -4.36 kΩ/decade. From this data, it can be seen that each sensor has an average correlation coefficient (R2) value close to 1, indicating a good linearity between the impedance response values and NaCl concentration.

4 CONCLUSION

This work presents the study of a lipid membrane-based salty taste sensor was developed on a PVC-modified graphite solid-contact electrode that had been modified with PPy-CB. The addition of polypyrrole-carbon black (PPy-CB) material can increase the capacitive properties, surface area, and conductivity of the graphite PVC electrode. Based on the sensor response measurement using impedimetric methods, the addition of TDAB and 1-Hexadecanol membrane showed a tendency for the electrode to be selective towards salty taste. This was confirmed through PCA analysis, which showed that the separation of salty taste from other tastes on the GP/PPy-CB/LPM sensor electrode had a silhouette coefficient value of 0.9. The developed sensor's selectivity showed promising results, with optimal responses obtained at a frequency of 35.6 Hz, and provided good sensitivity to NaCl samples in the range of 0.1 to 30 mM, with a sensitivity value at -4.36 kΩ/decade. Further development can optimize the amount of PPy-CB conductive polymer material added to the sensor surface to improve the sensor's performance.

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