EINSTEIN (e-Journal)
Jurnal Hasil Penelitian Bidang Fisika
Available online http://jurnal.unimed.ac.id/2012/index.php/einsten
e-issn: 2407 – 747x, p-issn 2338 – 1981

ANALYSIS AND CHARACTERIZATION OF THE INDICATOR ELECTRODE MEMBRANE PVA-ENZYME/GA/PANI-BENZENE SULFONATE ACID/ PVC-KTPCLPB O-NPOE

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Submit: April 2026. Approved: May 2026. Published: June 2026.

ABSTRACT
This research aims to determine the characteristics of indicator electrode membranes using XRD, SEM-EDS and FTIR testing. Differences in the composition of the urease enzyme will affect the diffraction spectrum pattern of the indicator electrode and the use of a conductive polymer, namely PaNi, can increase the intensity of the crystal diffraction pattern. The method used is the potentiometric biosensor method with the urease enzyme immobilization technique. The indicator electrode membrane is composed of four layers, namely PVA-enzyme/GA/PANi-Benzene Sulfonic Acid/PVC-KTpClPB-o-NPOE. Based on XRD testing, sample II has the highest intensity of 39170 at $2\theta = 24.07°$; while sample II has an intensity of 8450 a.u. The resulting diffraction pattern shows that the electrode membrane is a polymeric material as indicated by the presence of crystalline and amorphous patterns. Based on SEM-EDS testing, it shows that the morphological structure of sample I has fewer pores with C elements of 9.99 ± 0.21 wt.% and O of 4.37 ± 0.18 wt.% compared to sample II with C elements of 16.41 ± 0.29 wt.%. Apart from that, the best electrode membrane is PVA-Enzyme 1 drop/GA/PANi-Benzene Sulfonic Acid/PVC-KTpClPB-o-NPOE 66% and the functional groups C – O, C = C, C – H, and N – H are obtained. with the highest transmittance in the wave number range 1423.84 cm^{-1} – 2914.77 cm^{-1}.

Keywords: Indicator electrode, Potentiometry, Polyaniline, Benzene Sulfonic Acid

INTRODUCTION

A device known as a biosensor is used to analyze concentrations by changing signals from biological components such as enzymes, bacteria and others. The converted signal is then combined with physical elements such as sensors and transducers to produce output signals such as current, potential, optics, density and so on. This biological component functions as an electroactive sensor involved in electrochemical half-cell reactions, so that the resulting potential is sensitive and selective towards certain ions. (Waruwu & Hakim S, 2020)

The Urea Biosensor is a tool developed using the biological reaction (biocatalyst) "urease" which functions to analyze urea. One method for determining urea levels is the potentiometric method. For the potentiometric method, the equilibrium potential difference between the indicator electrode and the reference electrode is measured (Tamba, 2016). The potentiometric method is an electrochemical analysis technique used to carry out quantitative measurements by measuring the potential of an electrode against a comparison electrode. In the potentiometric method there are several components such as the working electrode (indicator electrode), reference electrode, salt bridge and potential measuring device (voltmeter). The working electrode is an electrode that functions as a sensor that will analyze the analyte (Suheryanto et al., 2019). The reference electrode is a half-cell electrode whose potential value is known.

Various studies use this type of polymer as a urease immobilization matrix for electrode membrane parameters. (Begum, 2012). One type of polymer used is Polyvinyl Alcohol (PVA). PVA is a type of polymer that is soluble in water (Abd Hakim dkk., 2019) which is now known as a synthetic polymer that can be used as a research material for the immobilization of membrane-form biocatalysts. Meanwhile, PVC is waterproof and has good pores so it is quite good to use as a membrane. To increase the elastic properties of PVC, you can use a plasticizer. Then, to compare the composition of PVC membrane materials and plasticizers, it affects the sensitivity and selectivity of the ISE membrane because PVC will coat PVA. (Waruwu & Hakim S, 2020).

In research conducted by (Hakim S dkk.,2022) regarding the manufacture of indicator electrodes with membrane modification. The membrane modification involves coating PVA-enzyme using GA (Glutaraldehyde) which is then followed by coating PVC-KTpClPB-o-NPOE. GA also plays a role in increasing the stability and sustainability of enzyme function as well as enabling repeated use of the enzyme as an absorbent and contributing to increased strength and thermal stability. (Hakim S .,2021).

In research conducted by (Hakim S,A dkk., 2023) namely making and characterizing indicator electrodes with several modifications. One modification is an indicator electrode layer consisting of PVA-Enzym/GA-2,9%/PPy+Sulfonic Acid/PVC-KTpCIPB-o-NPOE. GA is used to increase the detection range, o-NPOE forms an amorphous structure and enzyme variations are used to increase the intensity of the XRD spectrum pattern. The development process for modifying the indicator electrode membrane was carried out in stages using GA for cross-linking, o-NPOE and conductive polymer. The best results were found on indicator electrodes PVA-Enzyme/GA-2.9 %/PPy + Sulfonic Acid/ PVC-KTpClPB-o-NPOE. This is because linear curve analysis of the sample shows a sensitivity of 41.56 mV per decade, a detection range of between 10-4 to 10-1 M, with a detection limit of 10-4 M, and an R2 of 97.51%.

In the research that has been carried out by (Simanihuruk, 2023) namely making and characterizing indicator electrodes with modifications, namely the indicator electrode layer from PVA-Enzyme/GA/PANi-/PVC-KTpCIPB-o-NPOE. The transmittance spectrum pattern from the FTIR test results shows that the amount of transmittance produced has decreased because the conductive polymer used is not PPy conductive polymer but rather polyaniline conductive polymer (PANi-HCl). So the properties of the acid still need to be studied. Because even though the concentration value of an acid is high, it does not necessarily mean that the intensity value is also high. So what makes the high intensity value is the nature of the conductive polymer. So this research requires polyaniline (PANI) which can be used as a conductive polymer which can increase the intensity value.

The electrode membrane was characterized using various tests including, Scanning Electron Microscopy-Energy Dispersive X-Ray (SEM-EDX), Fourier Transform Infrared Spectroscopy (FTIR) and X-Ray Diffraction (XRD). SEM-EDS is used to observe and understand surface morphology and particle size distribution. FTIR is used to identify functional groups present in compounds. Meanwhile, XRD is used to determine the membrane intensity ratio and to characterize the properties of the resulting membrane.

RESEARCH METHOD

The materials used in this research were polyvinyl alcohol (PVA), urease enzyme, glutaraldehyde (GA), polyaniline (PANi), polyvinyl chloride (PVC), potassium tetrakis p-chlorophenyl borate (KTpClPB), o-nitrophenylloctylether (o-NPOE), tetrohydrofuran, Benzene Sulfonic Acid, distilled water and wolframm electrode. 0.0350 g PVA-Urease enzyme was coated with 2.9% GA then 0.0350 g PANi and finally coated with 0.0350 g PVC-KTpClPB-o-NPOE. These layers will form 4 layers into a membrane electrode.

The indicator electrode was made using the potentiometric method, the urease immobilization technique. Electrode membrane samples were made by varying the urease enzyme droplets on the membrane layer, namely one and 1 drop each at the 61% o-NPOE solution composition and one drop variation at the 66% o-NPOE solution composition.

Analysis and characterization of the electrode membrane will be demonstrated using XRD, SEM-EDS and FTIR testing.

RESULT AND DISCUSSION

Characterization Results using XRD

Based on the XRD test results, the diffraction patterns of several samples can be shown as shown in Figure 1 below.

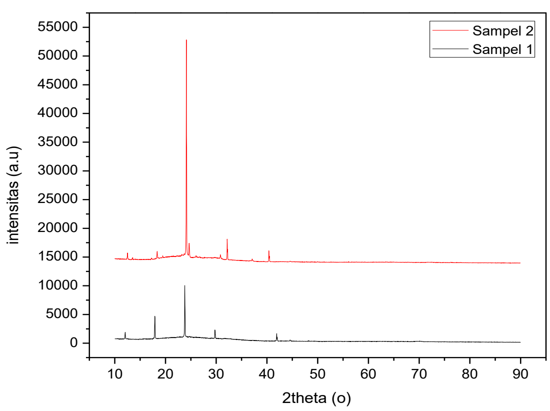


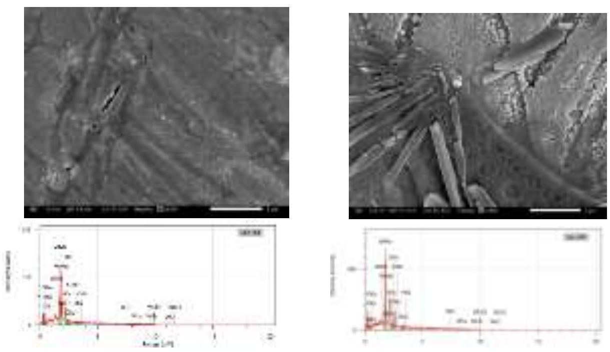
Figure 1. Diffraction patterns of samples I and II from XRD analysis

Tabel 1. Diffraction patterns in samples I and II using XRD

2theta (º)	Intensitas (a.u)	
	Sampel I	Sampel II
24,07	1255	16536

Characterization Results using XRD

Based on the SEM-EDS test results, the morphology and elements contained in the electrode membrane can be shown as shown in Figure 2 below.



(a) (b)

Figure 2. Surface morphology of the electrode membrane sample with a magnification of 5000× (a) 1 drop of urease enzyme in a 61% o-NPOE solution (b) 1 drop of urease enzyme in a 66% o-NPOE solution

Based on SEM-EDS testing, it can be shown that several elements can be identified on the surface of the electrode membrane sample. The following are some of the elements detected on the sample surface.

Table 2. Elements in Sample I (PVA_Enzyme 1 drop/GA/PANi-Benzene Sulfonic Acid/PVC-KTpClPB-o-NPOE 61%

Elements	Series	Mass %	Atom %
Carbon	K-Series	9,99	48,74
Oxygen	K-Series	4,37	16,0
Chlorine	K-Series	2,43	4,02
Nitrogen	K-Series	1,22	1,22
Wolframm	M-Series	81,98	26,12

Table 3. Elements in Sample II (PVA_Enzyme 1 drop/GA/PANi-Benzene Sulfonic Acid/PVC-KTpClPB-o-NPOE 66%

Elements	Series	Mass %	Atom %
Carbon	K-Series	16,41	56,55
Oxygen	K-Series	4,54	11,76
Chlorine	K-Series	10,88	12,07
Nitrogen	K-Series	0,79	2,35
Kalium	K-Series	1,77	1,88
Wolframm	M-Series	65,60	24,77

Analisis SEM-EDS dan XRD

In table 1 it can be seen that the composition of the elements C and O has a more prominent composition compared to other elements, namely with a mass of 9.99 wt.% and 2.43 wt.%. The Cl element shows that the fourth layer is composed of compounds containing oxygen. However, in table 2, the C element is more prominent compared to the other elements identified, namely 16.41 wt.%, which shows that PVA and PVC as polymer matrices have C branches as functional groups (Hakim S et al., 2019). However, the Boron element cannot be identified because the SEM-EDS test tool cannot identify elements with an atomic number below 11 where the atomic number of the Boron element is 5. Then in the SEM-EDS test table there is also Wolframm (W) which shows that the electrode was made on tungsten rods (Hakim S,2023).

Table 4. Membrane Indicator Electrode Compounds and Elements

Elektroda Membran	Formula	Jumlah (%)	Sistem Kristal	Unsur
Sampel I (PVA-Enzim 1 tetes/GA/PANi-HCl/PVC-KTpClPB-o-NPOE 61%	$C_{40}H_{108}Cl_4Cr_4N_{16}O_{22}W_4$	100 %	*Orthorhombic*	C H N O Cl Cr W
Sampel II (PVA-Enzim 1 tetes/GA/PANi-HCl/PVC-KTpClPB-o-NPOE 66%	$C_{40}H_{108}Cl_4Cr_4N_{16}O_{22}W_4$	22,1 %	*Orthorhombic*	C H Cr Cl O N W
	$C_4H_6O_4$	77,9 %	*Monoclinic*	C H O

On the PVA-Enzyme/GA/PANi-Benzene Sulfonic Acid/PVC-KTpClPB-o-NPOE membrane electrode, there are several components that are expected to be seen, including C, O, H, Cl, B, P, K, and W (Hakim S, et al., 2017). In table 4.7, there are several elements that can be identified using match software, namely C, O, H, N, K, Cl, Cr, and W. Several compounds in the XRD test can be reviewed in the SEM-EDS test to determine the intensity of the elements in the compound. – the compound.

According to research conducted by (Hakim S, 2023) with the PVA-Enzyme/GA/PPy + Sulfonic Acid/PVC-KTpClPB-o-NPOE indicator electrode, the energy intensity produced was 14.7 cps/keV. Sample I has an energy intensity of 12 cps/keV and sample II has an energy intensity of 12.5 cps/keV, which shows an increase in XRD intensity (Hakim S, 2023)

FTIR Test Analysis

The PVA-Enzyme/GA/PANi-HCl/PVC-KTpClPB-o-NPOE solution which has been characterized by XRD and SEM-EDS tests will then be tested again by FTIR test. The best sample chosen was sample II (PVA-Enzyme 1 drop/GA/PANi-Benzene sulfonic acid/PVC-KTpClPB-o-NPOE 66%) because it has a diffraction pattern that shows a regular crystal structure and a better morphological structure because it has fewer pores than other samples. FTIR testing was carried out to determine the functional groups contained in the sample membrane. The following is the spectrum pattern resulting from FTIR testing.

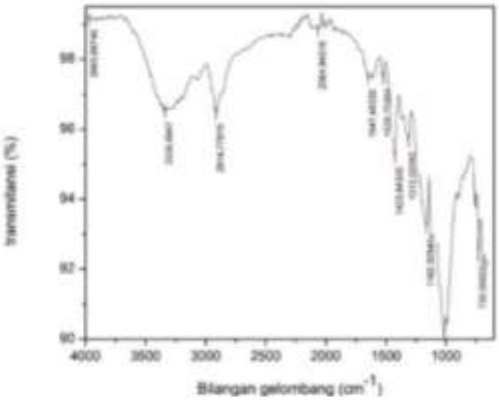


Figure 3. Spectrum Pattern via FTIR Testing

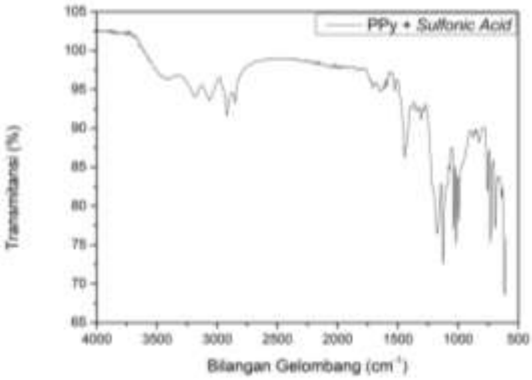


Figure 4. Spectrum Pattern of PVA-Enzyme/GA/Ppy+Sulfonic Acid/PVC-KTpClPB-o-NPOE

Based on Figure 3, it shows the spectrum pattern on the 1 drop PVA-Enzyme membrane/GA/PANi-Benzene Sulfonic Acid/PVC-KTpClPB-o-NPOE 66% containing the C-O functional group seen at a wave number of 1162.92 cm-1 with the type of alcohol compound, ethers, carboxylic acids and esters. The C=C functional group is found at wave numbers 1520.75 cm-1 – 1647.48 cm-1 with aromatic and alkene ring compound types. The C – H functional group is found at wave numbers 1423.84 cm-1 – 2914.77 cm-1 with the type of alkane compound. The N-H functional group is found at wave number 3339.69 with the amine compound type. Polyaniline (PANi) is the conductive polymer used. The FTIR test results show that in the wave number range 1433.00 cm-1 to 2866.81 cm-1 there is a decrease in transmission intensity compared to the indicator electrode using PPy + Sulfonic Acid, as shown in Figure 5. This shows that polyaniline has the properties good conductor. (Hakim S, 2023).

CONCLUSION AND SUGGESTION

The diffraction spectrum pattern in sample II shows a good pattern because there is a high crystalline pattern, namely at 2θ = 24.07º and the intensity of the amorphous pattern is lower when compared to sample I. The morphological structure in sample I has a smoother surface and does not have many pores on the surface of the sample while sample II has a rougher surface. This is caused by differences in the composition of the urease enzyme in the indicator electrode samples. The indicator electrode samples that have been tested show that there are several elements that make up the electrode. Some of the identified elements include Carbon (C), Nitrogen (N), Oxygen (O), Chlorine (Cl), Potassium (K).

The functional groups identified in the indicator electrode membrane samples are C – O, C = C, C – H, and N – H. The transmittance spectrum pattern from the FTIR test results shows that the amount of transmittance produced has decreased because the conductive polymer used is not PPy but rather polyaniline. (PANi) which is paired with Benzene Sulfonic Acid.

Characterization can be continued with potentiometric biosensor testing with immobilization techniques to see the linear curve pattern and response time so that the sensitivity and selectivity of the indicator electrode can be analyzed. Then variations in composition and number of drops of venzyme urease can be further developed to get better results.

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