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Characterization Membrane Composition Of PVA-Enzyme Coating PVC-KTpClPB As Urea Sensor With UV-VIS, SEM-EDX and XRD

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Abstract. Potentiometric ion sensor (ISE) is very attractive because of its small size, easy to carry, more durable, and miniature easy to use. ISE consists of an indicator electrode and a reference electrode located in an electrolyte solution. Potentiometric electrodes and electrochemical properties can be achieved through membrane synthesis and selectivity by the interaction of primary ions with respect to cavity size, molecular geometry and types of functional groups leading to the interaction between electrolyte solutions, membranes and indicator electrodes with reference electrodes. Indicator electrodes function as urea analytes. Potentiometric characteristics are significantly dependent on the composition of the membrane, the nature of the plasticizers. Characterization of membrane composition was carried out using (UV-Vis, SEM-EDX, and XRD). Characterization of PVA membrane : PVC in the composition of 1 : 1 of 0.0350 g PVA and plasticizer 0.0700 g KTpClPB variation of membrane electrode layer and enzyme 1 mg. The optimum amount of diffraction intensity is obtained from PVA E 4X PVC-KTpClPB 0.0700.

1. Introduction

Potentiometric sensors are devices that measure the voltage between two indicator electrodes and the reference electrode depending on the concentration of the analyte, without contradicting the electrochemical cell [1]. Analites bind the bioreceptor to the surface of the indicator electrode in a buffer solution, producing potential differences between the two electrodes. Measurement of urea concentration in the blood shows kidney indications and liver function, liver / heart failure, excessive protein or protein catabolism input, malnutrition, pregnancy, shock and pressure. Biological bond reaction selected urease enzyme with PVA and PVC substrate, its interaction in the form of molecular adsorption to increase the limit of detection and selectivity of biosensors [2]. PVA is soluble in water as well as the urease enzyme dissolves in water, while PVC is not soluble in water. PVA and PVC are polymer matrices that have branch C as a functional group.

Materials consisting of conductor polymers have responsive properties and are sensitive to small disturbances, conductor polymer material as one of the materials for making electrochemical sensors.

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The polymer is modified to bind biomolecules to biosensors. Biosensors use enzymes as recognition elements [3]. Electrochemical sensors and biosensors have advantages because they are simple, sensitive, fast and do not require sample preparation. Biosensors use enzymes immobilized into polyvinylchloride (PVC), polyvinyl alcohol (PVA) into membranes [4] (Narang at al, 2013), modification of electrodes has advantages such as ease of construction, fast response time and stability.

2. Materials and Methods

2.1. Materials

Materials used in this study are Urea-^{13} C (99 atom % ^{13}C) $H_2N^{13}CONH_2$ FW 61.05, the enzyme EC 3.5.1.5 (Urease) U4002-100KU (ix type), Tungsten (wire of 1.0 mm diameter, 99.99%, 7440-33-7) W, PVA [- CH_2CHOH-]n, PVC $(CH_2CHCl)n$, KTpClPB (potassium tetrakis 4-chlorophenyl borate), Tetrahydrofuran C_4H_8O.

2.2. Tools

Tools used in this resecarch are SEM-edx (Bruker-129 eV Zeiss), UV-Vis (Ray Leigh UV-1601), XRD-6100 (Shimazu), tungsten indicator electrode (W) and other supporting tools.

2.3. Methods

The membrane consists of membrane immobilization and membrane coating, matrix immobilization membranes and matrix membrane coating has a ratio of W_{PVA}: W_{PVC} is 1: 1 [5]. Membrane coating has a composition (PVC: plasticizer = 1: 2) [6], as plasticizer is KTpClPB. Preparation membrane solutions derived from a mixture of PVA solution and enzyme solution. PVA solution consists of 0.0350 g PVA which is dissolved in 10 mL of hot water until the PVA solution is in accordance with room temperature. Mixture of 50% water: 50% alcohol in three 10 ml sized beakers, input the EC 3.5.1.5 (Urease) enzyme 1.0 mg, 1.5 mg, and 2.0 mg, into each beakers. Preparation of the membrane coating consists of PVC-KTpClPB solution in 10 ml of THF with a composition of PVC: PVC-KTpClPB is 0.0350 g: 0.0700 g. Thus the PVA-enzyme solution and PVC-KTpClPB solution have been obtained which can be made into membranes and membrane electrodes. Membranes and electrode membranes were characterized using SEM-edx and XRD, while PVA-enzyme-KTpClPB membrane solution was characterized by UV-Vis.

3. Results and Discussion

3.1. Membrane 0.0350 g PVA-E PVC 0.0700 g

First, characterization of membrane solution 0.0350 g PVA-E PVC 0.0700 g from enzyme variation of 1.0 mg 1.5 mg and 2.0 mg, the results can be seen in Figure 1. Only a little addition of absorbance from the membrane solution 0.0350 g PVA-E PVC 0.0700 g on enzyme variations 1.0 mg of absorbance peak is 1.6347, 1.5 mg of absorbance peak is 1.4441 and 2.0 mg of absorbance peak is 1.4252 and there are shifts in wavelengths of 294 nm and 367 nm from 275 - 500 nm, maximum absorbance at 1.0 mg enzyme, used for manufacturing membrane and membrane electrode in 0.0350 g PVA enzyme. UV-vis is used to ensure that the mixture sample shows a broad absorption peak [7], characterized in the range 180 - 1000 nm. X-ray diffraction used has a voltage of 40 kV with a electric current of 30 mA, scan range is 7,000-70,000, scan speed is 2.0000 (deg/min), sampling pitch is 0.0200 (deg), preset time is 0.60 (sec), and drive axis is theta-2theta was used to determine the mineral composition of the electrode membrane. This membrane was also analyzed by SEM-EDX at an acceleration voltage of 20 kV and 10 kV, [8] the results can be seen in Figure 4.

Electrode membranes are made in three types, namely (1) electrode immersion 1x in 0.0350 g PVA enzyme after 30 minutes coting PVC-KTpClPB is el.1 PVA E PVC-KTpClPB 0.0700 g, (2) dyeing electrode 2x in 0.0350 g PVA enzyme 2 times in every 30 minutes one time dyeing then coating PVC-

KTpClPB is el.2 PVA E PVC-KTpClPB 0.0700 g, (3) dyeing 3x elktroda in 0.0350 g PVA enzyme is also done 3 times immersion in every 30 minutes one time dyeing then coating PVC coting-KTpClPB is el.3 PVA E PVC-KTpClPB 0.0700 g.

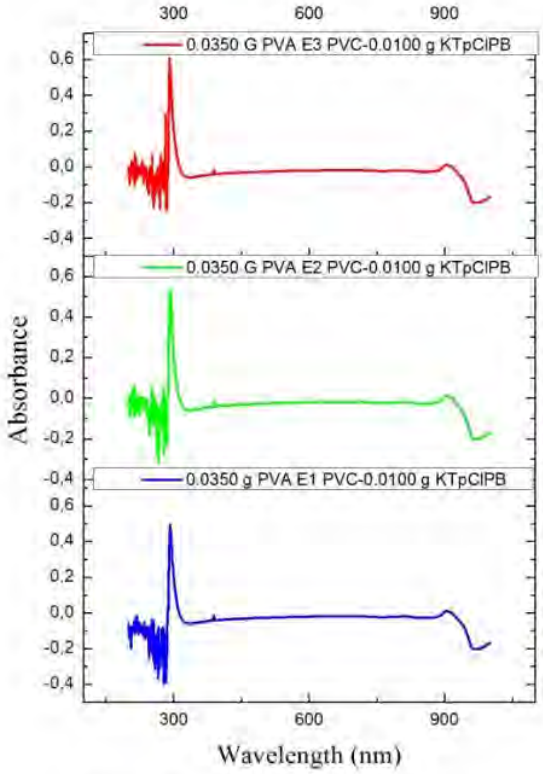
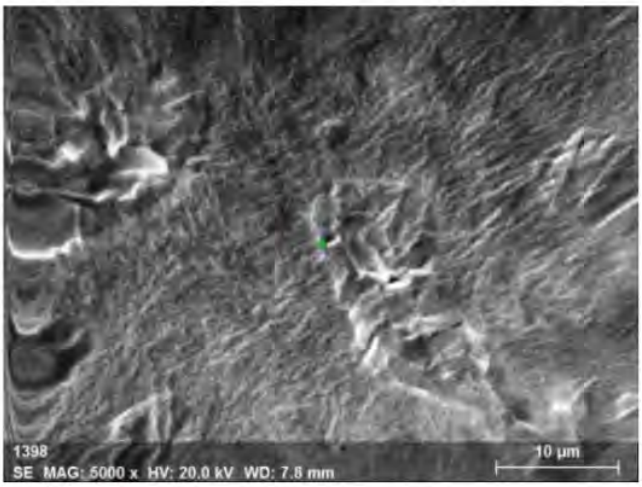


Figure 1. PVA-E PVC membrane solution of UV-Vis in composition (a) 0.0350 g PVA-E1 PVC-0.0700 g, (b) 0.0350 g PVA-E2 PVC-0.0700 g, (c) 0.0350 g PVA-E3 PVC -0.0700 g.

In Figure 2 the acceleration voltage of 20 kV for PVA-E 1X PVC membrane is 0.0700 g from (a) SEM obtained a small pore, (b) EDX obtained elements of elements C, Cl, K, O and P on the surface of PV 1-PVC PVA-E membrane 0.0700 g with a carbon C ratio of 2.19, chlorine Cl is 0.18, potassium K is 0.08, oxygen O is 0.51 and phosphorus P is 0.04 can be seen in table 1. There are differences in images 2 and 3 of the composition of the membrane 0.0350 g PVA-E 1x PVC 0.0700 g and PVA-E 2X PVC 0.0700 g more pores in Figure 3 at an acceleration voltage of 10 kV, as well as element elements of PVA-E 2X PVC 0.0700 g from EDX with a carbon C ratio of 9.23, oxygen O is 3.80, chlorine Cl is 0.24, phosphorus P is 0.08, magnesium Mg is 0.06 and sodium Na is 0.05. Based on the table 1 the composition of the membrane 0.0350 g PVA-E 1x PVC 0.0700 g and table 2 the composition of 2X PVC PVA-E membrane 0.0700 g shows that the carbon C ratio of 1x dyeing is 2.19 and 2x dyeing is 9.23, this is in accordance with the nature of PVA and PVC for a polymer matrix which has a C branch as a functional group.



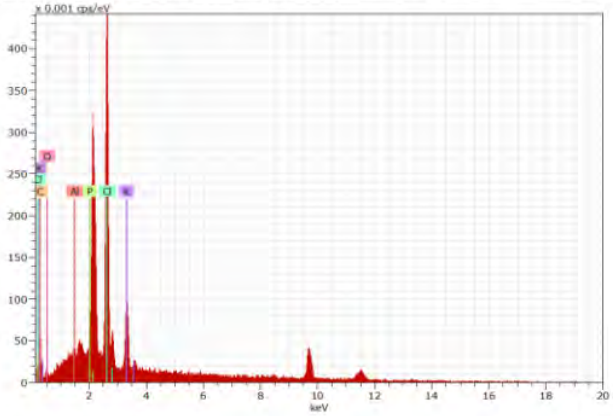


Figure 2. PVC 1-PVC PVA-E membrane 0.0700 g of (a) SEM, (b) EDX

Table 1. Ratio of PVA-E 1X PVC membrane element elements 0.0700 g using SEM-EDX

Element	Weight %	Atomic %	Ratio Atomic
C	54.80	75.14	2.19
Cl	27.83	12.93	0.18
K	8.23	3.47	0.08
O	7.25	7.46	0.51
P	1.88	1.00	0.04
Al	0.00	0.00	0.00

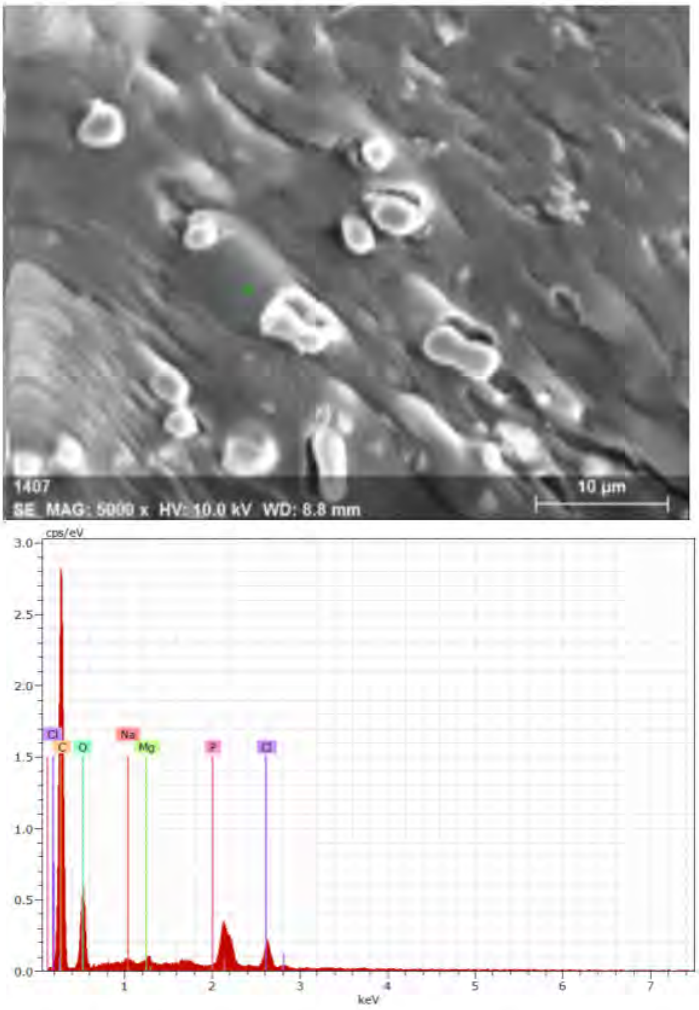


Figure 3. PVA-E membrane 2X PVC 0.0700 g from (a) SEM, (b) EDX

Table 2. Ratio of 0.0700 g PVA 2X PVC membrane element elements using SEM-EDX

Element	Weight %	Atomic %	Ratio Atomic
C	70.85	78.42	9.23
O	23.08	19.18	3.80
Cl	4.91	1.84	0.24
P	0.68	0.29	0.08
Mg	0.29	0.16	0.06
Na	0.20	0.11	0.05

Table 3. Ratio of element C elements from variations of enzyme PVA layers with PVC-KTpClPB coating only 1 time using SEM-EDX

Element	Membran	Ratio Atomic
C	PVA E 1 layer PVC-KTpClPB 0.0700 g	2.19
	PVA E 2 layer PVC-KTpClPB 0.0700 g	9.23

Based on table 3 and table 4 there is a proportional effect between the carbon C ratio and the amount of XRD diffraction intensity, namely PVA E 1 layer PVC-KTpClPB 0.0700 g and PVA E 2 layer PVC-KTpClPB 0.0700 g. According to Figure 4 the optimum amount of diffraction intensity is obtained from PVA E 4X PVC-KTpClPB 0.0700 g. There is a shift in wavelength through UV-Vis characterization in Figure 1, followed by a shift in the diffraction angle in Figure 4.

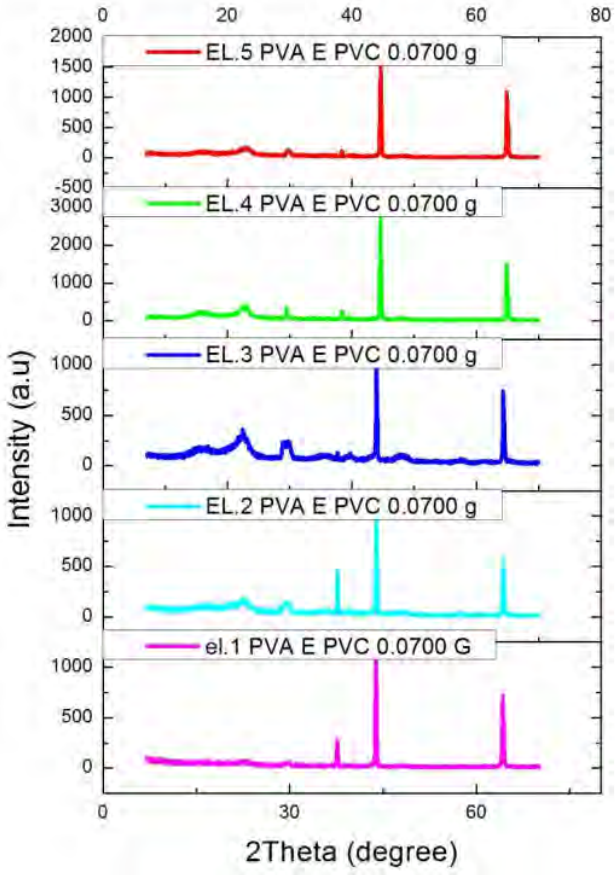


Figure 4. PVA-E PVC-KTpClPB membrane layer of XRD on indicator electrode (a) el.1 one layer, (b) el.2 two layer, (c) el.3 three layer, (d) el.4 four layer, (e) el.5 five layer

Table 4. Count Intensity C of the variation layer of KTpClPB on the electrode with PVA enzyme still using XRD

Membran Elektroda	2Theta (deg)	Count Intensity
PVA E 1X PVC-KTpClPB 0.0700 g	43.92	812
PVA E 2X PVC-KTpClPB 0.0700 g	43.92	1008
PVA E 3X PVC-KTpClPB 0.0700 g	43.92	1066
PVA E 4X PVC-KTpClPB 0.0700 g	44.56	2838
PVA E 5X PVC-KTpClPB 0.0700 g	44.6	1824

4. Conclusion

Characterization of PVA-enzyme PVC-KTpClPB with UV-Vis membrane solution was analyzed in a range of 275-500 nm and PVC-KTpClPB electrode mrmbrane with SEM-EDX to determine the carbon C and XRD ratios to determine the intensity of the diffraction degree membrane electrode. There is a comparison of the carbon C ratio with the amount of intensity to the degree of diffraction of the electrode membrane.

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