

# THE CHARACTERISTICS MEMBRANE PVA-ENZYME AND COATING PVC- PLASTICIZER WITH SEM-EDX

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**Abstract:** *This study aims at PVA membrane-coating enzyme characteristics PVC-plasticizer through the SEM absorption spectrum and completeness of elements using EDX. The method used membrane immobilization at the indicator electrode derived from Wolfram material. Selection phase of indicator electrode by dyeing tungsten electrode with PVA - enzyme, then coating with PVC - plasticizer and dyeing tungsten electrode using PVA - enzyme, then in coating with PVC - plasticizer. Membrane and electrode membrane are varied by respectively (1) PVA 0.5040 g + enzyme, (2) PVA 0.0350 g + enzyme, (3) PVA 0.5040 g + enzyme and coating I, (4) PVA 0.0350 g + enzyme and coating I, (5) PVA 0.0350 g + enzyme and coating II. The approach used for analysis is the morphology and EDX spectrum analysis. The result of the research was obtained with a 0.0350 g PVA membrane of the best Coating II Coating on 2x 1x coating dyeing, to be shown by difference morpologi of coating II and coting I, where coting II completely flatten at the indicator electrode.*

**Keywords:** SEM-EDX, immobilization-membrane, PVA-enzyme, PVC-plasticizer, coating.

**Abstrak :** *Penelitian ini bertujuan karakteristik membran PVA-enzim coating PVC-plastisizier melalui spektrum absorpsi SEM dan kelengkapan unsur menggunakan EDX. Metoda yang digunakan immobilisasi membran dan immobilisasi membran elektroda indikator, elektroda indikator berasal dari bahan Wolfram. Tahapan pemilihan elektroda indikator melalui analisis immobilisasi membran PVA - enzim coating PVC - plastisizier dan pencelupan elektroda wolfram menggunakan PVA - enzim, kemudian dicoating dengan PVC - plastisizer. Masing-masing membran dan membran elektroda divariasi denga (1) PVA 0.5040 g + enzim, (2) PVA 0.0350 g + enzim, (3) PVA 0.5040 g + enzim dan coating I, (4) PVA 0.0350 g + enzim dan coating I, (5) PVA 0.0350 g + enzim dan coating II. Pendekatan yang digunakan untuk analisis adalah morpologi dan analisa spektrum EDX. Hasil penelitian yang diperoleh membran PVA 0.0350 g Enzim Coating II terbaik pada 2x pencelupan coating 1x, ditunjukkan oleh perbedaan morpologi coating II dan coting I, dimana coting II serba merata pada elektroda indikator.*

**Keyword :** SEM-EDX, immobilization membrane, PVA-enzyme, PVC-plasticizer, coating.

## 1. Introduction

Poly (Vinyl Alcohol) base material for biosensors using enzyme entrapment in PVA matrix [1]. PVA membranes can absorb water as well as salts can diffuse through the polymer layer in a hydrated salt ion solution [2]. Membrane electrode PVC, sensitized ionophore with low solubility of polymer film in conventional organic and non-organic solvents [3]. The enzyme immobilization method according to physical properties (adhesion, inclusion) and cross-linking, PVC as the urease medium on the glass electrode surface for direct enzyme installation on the membrane [4]. Adhesion is used for the isolation of the transducer and the electric part of the coating on the ion-selective membrane. Membranes are used to separate the liquid mixture, also the membranes are used based on the diffuse pore size and absorption through the membrane [5]. This morphology and pore size is a problem in this study, one of the tools used is SEM-EDX. PVA and PVC are polymers, polymers can act as electrode materials in redox activity connected to specific potentials [6], through oxidation (doping p) and reduction (doping n) reaching the carrier for conduction. Based on the above description the researcher chose the title "Characteristics of PVA Membrane-

Coating Enzyme PVC- Plasticizer with SEM-EDX".

## 2. Materials and Methods

### 2.1 Chemical and Materials

The materials used in the study were standard urea 56180 Sigma-Aldrich, EC enzyme 3.5.1.5 (Urease) U4002, 50 - 100 µg type, PVA  $[-CH_2CHOH-]_n$ , PVC  $(CH_2CHCl)_n$ , potassium tetrakis 4-chlorophenyl borate  $(ClC_6H_4)_4BK$ , Tetrahydrofuran  $C_4H_8O$ , is derived from Sigma-Aldrich and the method used is potentiometric method.

### 2.2 Tools

The equipment used is from Laboratory of Physics and Chemical Laboratory of State University of Medan in accordance with its use as follows: SEM Evo MA 10 Ziess, and Coating of SEM Q150RES Quorum.

### 2.3 Manufacture

#### 2.3.1 Procedure of membrane and membrane electrode manufacture with SEM-EDX

Coating I consists of 10 mL of THF mixed with PVC 0.5040 g and KTpClPB 0.0120 g. Coating II consists of 10 mL of THF mixed with PVC 0.0350 g and KTpClPB 0.0500 g. Membrane morphology using SEM with membrane composition and magnification as follows PVA 0.5040 g +

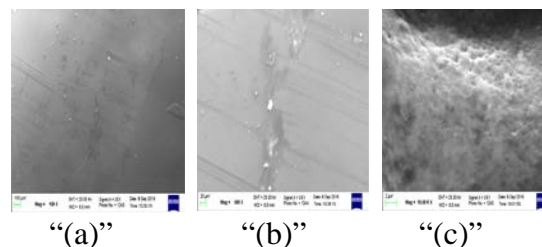
Enzymes and membranes PVA0.5040 g Enzyme-Coating I each magnification (a) 100x, (b) 500x, (c) 10000x, PVA membrane 0.0350 g + (A) 100x, (b) 10000x, 0.0350 g PVA membrane Coating I and PVA membrane 0.0350 g + 10000x. Morphologi membrane electrode with PVA composition 0.5040 g Dye enzyme 1x Coating I 1x, PVA 0.5040 g Dye enzyme 2x Coating I 1x, PVA 0.5040 g Dyeing enzyme 3x Coating I 1x, PVA 0.5040 g Dyeing enzyme 4x Coating I 1x, PVA 0.5040 g Dyeing enzymes 5x Coating I 1x, 0.0350 g Dye enzyme 1x Coating II 1x, 0.0350 g Dye enzyme 2x Coating II 1x, 0.0350 g Dye enzyme 3x Coating II 1x, 0.0350 g Dye enzyme 4x Coating II 1x, 0.0350 g Dye enzyme 5x Coating II 1x. The result of SEM morphology to determine membrane pore, SEM-EDX is used for morphology, absorption spectrum and elements present in the electrode membrane. SEM is used for morphology and EDX is used for analysis [7].

### 3. Result and Discussion

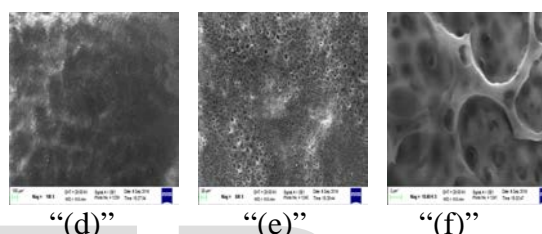
#### 3.1 Membrane characterization and membrane electrode with SEM-EDX

The morphological results of SEM for the composition of each membrane (1) PVA 0.5040 g + enzyme, (2) PVA 0.0350 g + enzyme, (3) PVA 0.5040 g + enzyme and

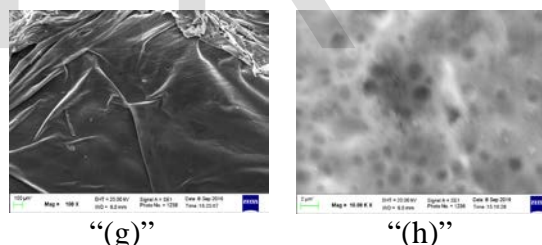
coating I, (4) PVA 0.0350 g + enzyme and coating I, (5) PVA 0.0350 g + enzyme and coating II can be seen in figure 1 s/d 5.



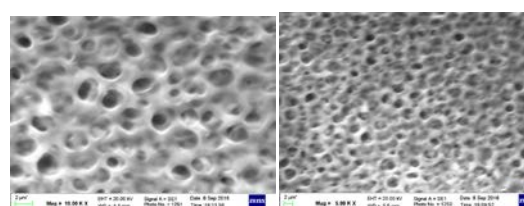
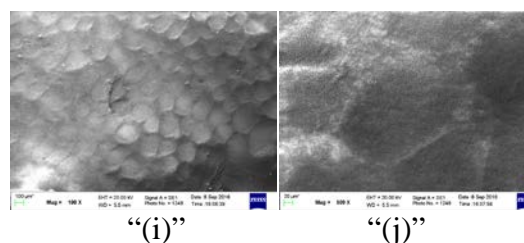
**Figure 1:** SEM PVA membrane 0.5040 g + Enzyme magnification (a) 100x, (b) 500x, (c) 10000x



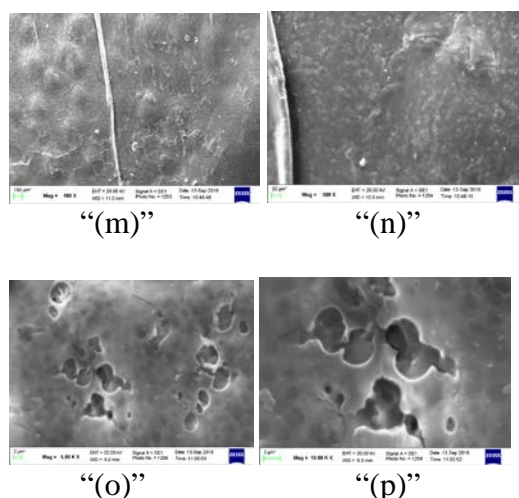
**Figure 2:** SEM membrane PVA0.5040 g Enzyme-Coating I magnification (a) 100x, (b) 500x, (c) 10000x



**Figure 3:** SEM PVA membrane 0.0350 g + Enzyme magnification (a) 100x, (b) 10000x



“(k)” “(l)”  
**Figure 4:** SEM PVA membrane 0.0350 g Coating I magnification (a) 100x, (b) 500x, (c) 5000x, (d) 10000x



“(o)” “(p)”  
**Figure 5:** SEM PVA membrane 0.0350 g Enzyme Coating II magnification (a) 100x, (b) 500x, (c) 5000x, (d) 10000x

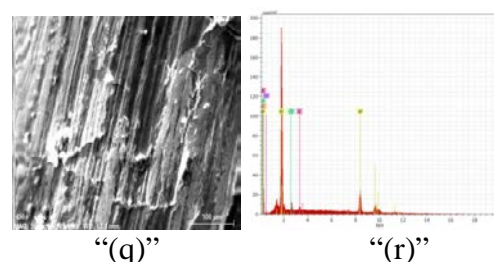
All membranes through SEM have pores at 10000x magnification appear clear but for 100x magnification there is not clear. The presence of pores provides an opportunity for electron transmission [8].

(3) PVA 0.5040 g + enzyme and coating I,  
(4) PVA 0.0350 g + enzyme and coating I,  
(5) PVA 0.0350 There is a difference of morphology (1) PVA 0.5040 g + enzyme, (2) PVA 0.0350 g + g + enzymes and coating II, due to changes in PVA concentration, PVC concentrations as coating I and coating II, fineness of the membrane surface. PVA [9] have chemical resistance both forming films / membranes and high hydrophilicas extensively used to separate water or hydrophilic elements. The membranes used are based on the size of the pores that can diffuse and absorb

(Jyoti et al, 2015). Coating layer thickness is estimated from SEM morphology and PVC properties [10].

### 3.2 Membrane Morphology on Electrode with SEM and EDX

After optimizing the concentration of the electrode layer in the ISE-urea potentiometry system, the membrane morphology test on the electrode and EDX observed the correctness of the element content of the electrode with SEM. The membrane morphology of the 1x, 2x, 3x, 4x, and 5x dipping electrodes can be seen in Figures 4.33 (a), 4.34 (a), 4.35 (a), 4.36 (a) and 4.37 (a) while the respective constituents (b), 4.36 (b) and 4.37 (b), through the EDX system elements can be seen in the table 4.8a, 4.8b, 4.8c, 4.8d , and 4.8e.

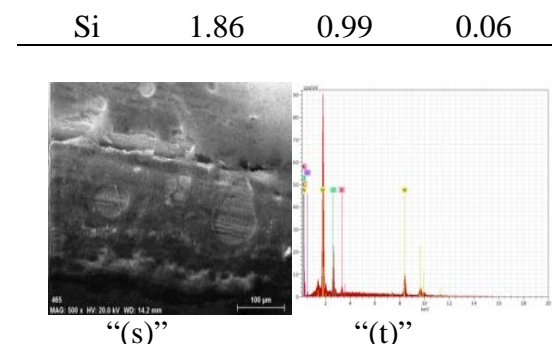


**Figure 6:** SEM and EDX analysis of PVA 0.5040 g Dyeing enzyme 1x Coating I 1x

**Table 1:** EDX Composition of PVA 0.5040 g Dye Enzyme 1x Coating I 1x

Element	Weight %	Atomic %	Ratio Atomic
C	65.65	81.59	2.59
O	13.50	12.60	0.82
Ni	13.49	3.43	0.15
Fe	3.10	0.83	0.06
Cu	2.40	0.56	0.06

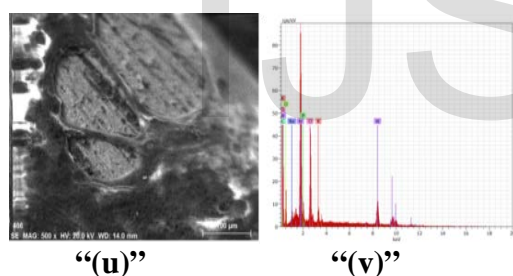




**Figure 7:** SEM and EDX analysis from PVA 0.5040 g Dyeing enzyme 2x Coating I 1x.

**Table 2:** EDX Composition of PVA 0.5040 g Dyeing Enzyme 2x Coating I 1x

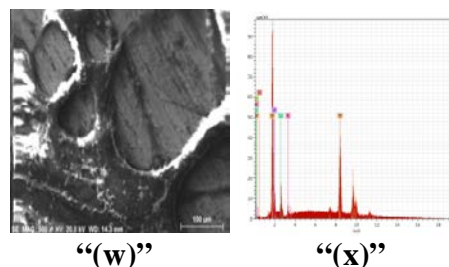
Element	Weight %	Atomic %	Ratio Atomic
C	65.65	81.59	2.59
O	13.50	12.60	0.82
Ni	13.49	3.43	0.15
Fe	3.10	0.83	0.06
Cu	2.40	0.56	0.06
Si	1.86	0.99	0.06



**Figure 8:** SEM and EDX analysis of PVA 0.5040 g Dyeing enzyme 3x Coating I 1x

**Table 3:** EDX Composition of PVA 0.5040 g Dyeing Enzyme 3x Coating I 1x

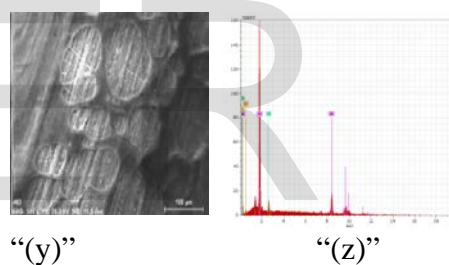
Element	Weight %	Atomic %	Ratio Atomic
C	65.65	81.59	2.59
O	13.50	12.60	0.82
Ni	13.49	3.43	0.15
Fe	3.10	0.83	0.06
Cu	2.40	0.56	0.06
Si	1.86	0.99	0.06



**Figure 9:** SEM and EDX analysis of PVA 0.5040 g Dyeing enzyme 4x Coating I 1x

**Table 4:** EDX Composition of PVA 0.5040 g Dyeing Enzyme 4x Coating I 1x

Element	Weight %	Atomic %	Ratio Atomic
C	65.65	81.59	2.59
O	13.50	12.60	0.82
Ni	13.49	3.43	0.15
Fe	3.10	0.83	0.06
Cu	2.40	0.56	0.06
Si	1.86	0.99	0.06



**Figure 10:** SEM and EDX analysis of PVA 0.5040 g Dyeing enzyme 5x Coating I 1x

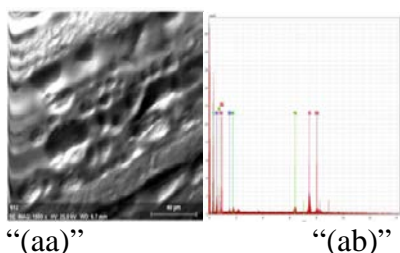
**Table 5:** EDX Composition of PVA 0.5040 g Dyeing Enzyme 5x Coating I 1x

Element	Weight %	Atomic %	Ratio Atomic
C	65.65	81.59	2.59
O	13.50	12.60	0.82
Ni	13.49	3.43	0.15
Fe	3.10	0.83	0.06
Cu	2.40	0.56	0.06
Si	1.86	0.99	0.06

Based on table 1 till table 5 above  
PVA membrane 0.5040 g Coating  
Enzymes I elements of electrode content

for 1x dyeing are W, C, Cl, O, K and enzyme, 2x dyeing are W, C, Cl, O, K and enzyme, 3x immersion is W, C, Cl, O, K, P, Na and enzyme, 4x dyeing are W, C, Cl, O, K, P, and enzyme, and 5x dyeing are W, C, Cl, O, and enzymes. The EDX analysis of the PVA 0.5040 g Coating I composition on the best W electrode by 3x ciating 1x dyeing [12] the C element content of 6.70% as an error (1Sigma) also according to the PVA  $[\text{CH}_2\text{CH}(\text{OH})]_n$ , PVC  $[\text{CH}_2\text{CH}(\text{OH})]_n$ , PVC  $(-\text{CH}_2\text{CH}(\text{Cl})_n)$ , plestesizier (KTPClPB), and THF  $(n\text{C}_4\text{H}_8\text{O} \rightarrow -(\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_2\text{O})_n)$ .

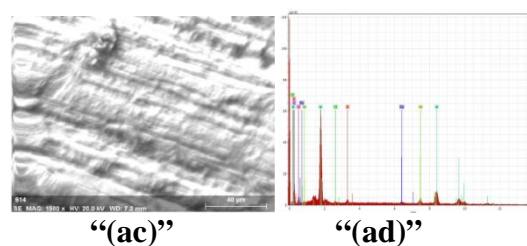
The following electrodes W (1x, 2x, 3x, 4x, and 5x) are immersed in a membrane made of (1) Enzyme (U4002-100 KU, type IX) of sigma 6 mg dissolved in an enzyme solvent composed of 50 % water and 50% alcohol (2) PVA  $[\text{CH}_2\text{CH}(\text{OH})]_n = 0.0350$  g, KTPClPB 0.0500 g (Potassium Tetra Kloro phenyl borate) as plastesiser, solvent = 10 mL tetrahydroforun THF  $(n\text{C}_4\text{H}_8\text{O} \rightarrow -(\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_2\text{O})_n)$ .



**Figure 11:** SEM and EDX analysis of PVA 0.0350 g Dyeing enzyme 1x Coating II 1x

**Table 6:** Composition EDX of PVA 0.0350 g Dye enzyme 1x Coating II 1x

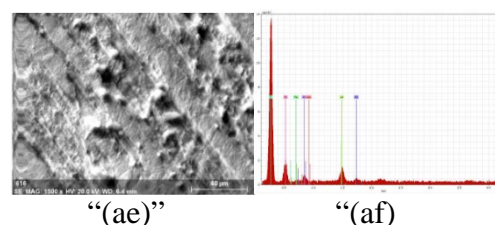
Element	Weight %	Atomic %	Ratio Atomic
C	65.65	81.59	2.59
O	13.50	12.60	0.82
Ni	13.49	3.43	0.15
Fe	3.10	0.83	0.06
Cu	2.40	0.56	0.06
Si	1.86	0.99	0.06



**Figure 12:** SEM and EDX analysis of PVA 0.0350 g Dyeing enzyme 2x Coating II 1x

**Table 7:** Composition EDX of PVA 0.0350 g Dye enzyme 2x Coating II 1x

Element	Weight %	Atomic %	Ratio Atomic
W	57.58	9.87	1.29
C	26.03	68.28	3.71
O	8.36	16.46	1.50
Ni	3.47	1.86	1.15
K	2.16	1.74	0.11
Cl	1.33	1.19	0.10
Fe	1.07	0.60	0.08

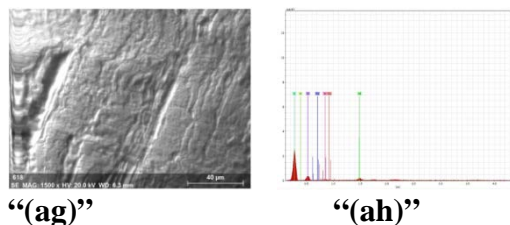


**Figure 13:** SEM and EDX analysis of PVA 0.0350 g Dyeing enzyme 3x Coating II 1x

**Table 8:** Composition EDX of PVA 0.0350 g Dye enzyme 3x Coating II

Element	Weight %	Atomic %	Ratio Atomic
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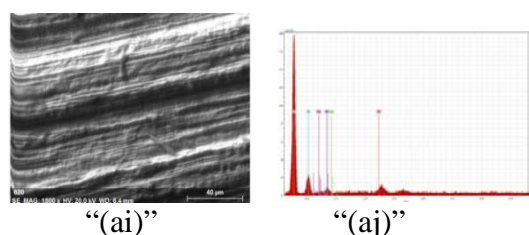
C	63.60	80.85	3.62
Ni	14.32	3.73	0.21
O	12.20	11.64	1.06
Al	3.53	2.00	0.12
Cu	3.28	0.79	0.09
Fe	2.49	0.68	0.07
Si	0.58	0.32	0.05



**Figure 14:** SEM and EDX analysis of PVA 0.0350 g Dyeing enzyme 4x Coating II 1x

**Table 9:** Composition EDX of PVA 0.0350 g Dye enzyme 4x Coating II

Element	Weight %	Atomic %	Ratio Atomic
C	58.63	74.52	3.70
O	14.06	13.41	1.27
Ni	13.96	3.63	0.22
N	5.10	5.56	0.87
Cu	3.51	0.84	0.10
Al	2.53	1.43	0.10
Fe	2.21	0.60	0.07



**Figure 15:** SEM and EDX analysis of PVA 0.0350 g Dyeing enzyme 5x Coating II 1x

**Table 10:** Composition EDX of PVA 0.0350 g Dye enzyme 5x Coating II 1x

Element	Weight %	Atomic %	Ratio Atomic
C	65.65	81.59	2.59
O	13.50	12.60	0.82

Ni	13.49	3.43	0.15
Fe	3.10	0.83	0.06
Cu	2.40	0.56	0.06
Si	1.86	0.99	0.06

Based on tables 1 to 6 of PVA membranes 0.0350 g Enzymes Coating II elements of electrode content for 1x dyeing are C, O, Ni, Fe, Cu, Si, Al and enzyme, 2x dyeing are W, C, O, Ni, K, Cl, Fe and enzyme, 3x dyeing are C, Ni, O, Al, Cu, Fe, Si and enzyme, 4x dye are C, O, Ni, N, Cu, Al, Fe and enzymes, and 5x dye are C, Ni, Fe, Cu, Si and enzymes. PVA membrane 0.0350 g The best Enzyme Coating II at 2x coating 1x by [12] element C content is 3.71% as ratio atomic, to be shown by difference morpologi of coating II and coting I, where coting II completely flatten at the indicator electrode.

#### 4. Conclusion

Based on the result and discussion of the research, the conclusion of the research is the need to repair the plasticizer composition of 0.0210 g (60%), 0.0231 g (66%), 0.0238 g (68%), 0.0242 g (69%) of 0.0350 g PVA.

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