

Decreasing of Mangan (II) in the Water Using Membrane of Moringa Seed Powder-TiO₂ with Variation of Mass TiO₂

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Received: September 2022
Accepted: January 2023
Published: March 2023

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Abstract

Mn (II) is a metal ion commonly used in steel alloys, pigment industries, welding, fertilizers, pesticides, ceramics, and electronics. According to the Regulation of the Minister of Health No. 32 of 2017, the permissible content of Manganese in dug well water is 0.5 mg/L. The purpose of this study was to determine the concentration of Mn (II) ions in water before and after passing through a Moringa Seeds Powder (MSP)-TiO₂ membrane 20:1; 20:3; 20:5; 20:7; 20:9 and measure the percentage decrease in the concentration of Mn (II) ions in water after through the MSP-TiO₂ membrane. The object of this research is a 55 ppm Mn (II) ion artificial sample at a flow rate of 0.56 mL/minute for 90 minutes with UV light 366 nm. The concentration of Mn (II) ion was measured by visible spectrophotometric method, the morphology of MSP, TiO₂, and MSP-TiO₂ membranes was characterized by SEM-EDX, and its diffraction spectra by X-Ray diffraction. The results obtained that the initial Mn(II) was 55.06 ± 0.031 ppm, the concentration of Mn (II) ions with the MSP-TiO₂ membrane of mass MSP-TiO₂ were 20:1; 20:3; 20:5; 20:7; 20:9 respectively 36.47±0.00; 44.16±1.15; 44.31±1.04; 44.94±0.94; 42.27±2.61 ppm. The percentage of decrease concentration of Mn (II) ion are 33.76±0.44%; 19.80±0.43%; 19.52±0.85%; 18.38±0.86%; and 23.23±0.86%. The highest percentage decrease in Mn (II) ion concentration was 34.15±0.44% in the variation of mass MSP-TiO₂ 20:1. The conclusion of this research is the the MSP-TiO₂ membrane has the potential to reduce the concentration of Mn (II) ions in water.

Keywords: *Moringa Seeds Powder (MSP), TiO₂, Mangan (II), membrane MSP-TiO₂*

Introduction

Water is one of the most important things that all living things need. Almost all of the fresh water in the world is ground water which can be utilized in daily life such as drinking,

agricultural irrigation, and for industrial activities^[1].

One of the sources of water used by the community to meet their water needs is healthy water is susceptible to contamination due to seepage leading to increased contents of

chemicals such as manganese and other organic materials. The presence of Manganese (Mn) in the water causes the color of the water to yellow-brown after some time in contact with air. According to the Regulation of the Minister of Health of the Republic of Indonesia No. 907/Menkes/SK/VII/2010, the permissible content of Manganese in dug well water is 0.5 ppm^[2]. Mn (II) is an ion of metal that is needed by humans but in small amounts. The rate of absorption of manganese is low and is decreased by phosphate, phytate, calcium, and iron. Manganese is quite high in erythrocytes, is transported in plasma by albumin, and is excreted in bile and pancreatic secretion. Symptoms of manganese deficiency are extremely rare, but in very high dosages, except by inhalation are non-toxic^[3]. Manganese is commonly used in steel alloys, pigment industries, welding, fertilizers, pesticides, ceramics, and electronics^[4].

Research of Munfiah^[5], healthy water in the working area of the Guntur II Demak Regency, the minimum-maximum value is 0.00-5.26 ppm and an average content of Mn (II) ions is 1.02 ± 1.33 ppm with an average pH of 6.45 ± 0.2 . The high concentration of Mn (II) ions requires a way to decrease Mn (II) concentrations. Therefore, it is to decrease the concentration of Mn (II) in water with metal degradation methods that can be biological, chemical, and physical. The adsorption process uses ZSM-5^[6], ZSM-5 membrane^[7], ZSM-5/TiO₂ powder^{[8][9][10]}, and ZSM-5/TiO₂ membrane^[11]. The price of the material to make ZSM-5 powder is relatively high, it is necessary to substitute ZSM-5 from natural adsorbents such as *Moringa oleifera* seeds^{[8][9]}, activated charcoal Moringa seed powder^[14], teak sawdust^[15], TiO₂ photocatalyst process with gauze buffer media^[16], and membranes MSP/TiO₂^[17], and Polyvinylidene fluoride (PVDF) Blending TiO₂-MSP membrane^[18].

The use of *Moringa oleifera* seed powder-TiO₂ membrane is to prevent the weakness of MSP-TiO₂ which is still present in water after coagulating metals such as Mn(II) water. TiO₂ or membrane of TiO₂ photocatalyst is used to decrease the concentration of Mn (II) ions in

solution in the presence of light, because TiO₂ photocatalysts can produce e⁻ (ecb⁻) and holes (hvb⁺) or •OH radicals easily. The number of •OH radicals from the photocatalyst TiO₂ and H₂O causes the number of electrons to be accepted by the Mn (II) ion so that the Mn (II) content in the water decreases^[19]. MSP/TiO₂ membrane with 20 g MSP and variation mass of TiO₂ 1, 3, 5, 7, and 9 g^[11].

The purpose of this study was to determine the concentration of Mn (II) ions in water before and after passing through an MSP-TiO₂ membrane with mass TiO₂ are 1; 3; 5; 7; 9; measure the percentage decrease in the concentration of Mn (II) ions in water, to determine the effective MSP/TiO₂ ratio that can reduce the maximum Mn(II) level, and characterization of surface shape and morphology by Scanning Electron Microscope (SEM), membrane composition using Energy Dispersive X-Ray (EDX).

Experimental

Materials

The materials used in this study were *Moringa oleifera* seed powder from Demak, MnSO₄ H₂O 100% (Merck, Germany), Toluene 99.5% (Merck, Germany), HCl 37% (Merck, Germany), Acetic Acid 100% (Merck, Germany), Aquades, Titanium dioxide (TiO₂) anatase 98.0-100.5% (Merck, Germany), Amylum GR for Analytic (Merck, Germany), NaOH 99.0% (Merck, Germany), HNO₃ 65% (Merck, Germany), AgNO₃ (Technical), and K₂S₂O₈ 99% (Merck, Germany).

Instruments

The instrumentation used in this study were visible spectrophotometer (Thermo Scientific, Genesys 20), SEM-EDX (Jeol JSM 6510 La), XRD (Shimadzu 7000), magnetic stirrer (Faithful M.S SH-3), oven (Mettler), a reactor with UV light 30 watts.

Methods

Gauze Pretreatment

Stainless-steel 304-300 gauze with a size of 3 cm x 3 cm were soaked in 95% toluene solution for

12 hours, then soaked in 15% HCl solution for 6 hours then rinsed with distilled water and stored in a dry place^[20].

Preparation of MSP/TiO₂ membrane A, B, C, D, E with 20 g MSP and TiO₂ mass variation 1; 3; 5; 7; 9 g

Moringa seed that is old and the brown seeds are peeled to get the cotyledons then mashed using a blender and dried for 24 hours. MSP was put in an oven for 1 hour at 90°C and sieved through a 100 mesh sieve ^[21]. Precursors of MSP/TiO₂ with TiO₂ mass 1g consist of a mass of MSP 20 g and TiO₂ mass 1 g made by 20 grams of MSP is dissolved in 40 mL of 10% acetic acid and then stirred with a magnetic stirrer at 900 rpm for 5 hours. Then 1 g of TiO₂ powder and 1 mL of 2% Amylum were added and stirred for 24 hours. MSP-TiO₂ precursors were coated on the gauze and put into a polypropylene plastic container, then dried in an oven at 105°C for 5 hours. The procedure was repeated 3 times and repeated with the membranes with a mass TiO₂ 3; 5; 7; 9 g^[22]. The thickness of the membrane was measured with a caliper.

Decrease Concentration Mn(II) Ions

The decreasing Mn (II) ion concentration was carried out using 50.0 mL Mn (II) ion samples 55.06 ppm through the MSP-TiO₂ membrane with a mass of ZSM-5: TiO₂ = 20:1 in a photoreactor Batch system equipped with a ZSM-5: TiO₂ = 20:1 with a flow rate of 0.56 mL/minute for 90 minutes and transferred quantitatively in 50 mL volumetric flask. This procedure was repeated 3 times and repeated with the MSP-TiO₂ membrane 20:3; 20:5; 20:7; and 20:9.

Determination of Mn (II) Ion Concentration after through the MSP-TiO₂ membrane

2.5 mL Mn (II) ion samples after through MSP/TiO₂ membrane A with a mass TiO₂ 1g added aquadest to 50.0 mL and transferred quantitatively pour into an erlenmeyer and added HNO₃ solution 1:1 and 5 mL of AgNO₃

0.1 N in erlenmeyer and was heated to a boil, then added K₂S₂O₈ powder and heating was continued for 5 min and transferred to a 50 mL volumetric flask and homogenized. The absorbance of the Mn (II) sample was analyzed using a Visible spectrophotometer at a maximum wavelength of 525 nm and stability time of 5 min^[6]. This procedure was repeated 3 times and repeated with the B, C, D, dan E MSP-TiO₂ membrane.

Results and Discussion

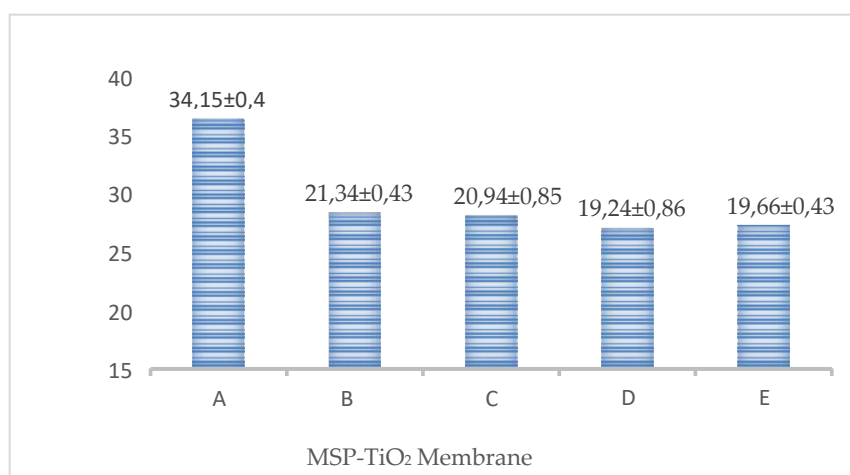
The purpose of this research was to decrease Mn (II) concentration samples by passing through the A, B, C, D, and E MSP-TiO₂ membrane with a mass TiO₂ of 1; 3; 5; 7; 9 by the gauze support 304-300 with a flow rate of 0.56 mL/min for 30 minutes UV light. The initial concentration of Mn (II) ion artificial samples is 55.06 ± 0.31 ppm. Absorbance and Mn (II) concentration before and after passing through variation of MSP-TiO₂ membrane presented in Table 1 and the percentage decrease in Mn (II) concentration after filtering using MSP-TiO₂ membranes can be seen in Figure 1.

Table 1 shows the absorbance data of the concentration initial Mn (II) artificial samples and after through the MSP-TiO₂ membrane. The concentration of Mn (II) ions early and after passing through membranes A, B, C, D, and E were calculated by the standard curve equation Mn (II) $y=0.0425x-0.0075$ with $R^2=0.9964$ (y=absorbance, x=Mn (II)) concentration obtained by interpolating absorbance vs standard Mn(II) 0.5-5.0 ppm with a visible of Spectrophotometer with a maximum a length wave 525 nm and maximum stability time of 5 min.

Table 1 showed the concentration of Mn (II) ions decreased after passing through the A, B, C, D, and E MSP-TiO₂ membrane and the lowest Mn (II) ion concentration was obtained after passing through the A MSP-TiO₂ membrane.

Table 1. Absorbance and Mn (II) concentration after passing through of MSP-TiO₂ membrane

MSP-TiO ₂ Membrane	Repeat	Absorbance	Mn (II) Ion Concentration (ppm)	Mean of Mn (II) Ion Concentration (ppm)
Initial	1	0.110	55.29	55.06±0.031
	2	0.108	54.35	
	3	0.109	55.06	
A	1	0.071	36.94	36.47±0.00
	2	0.069	36.00	
	3	0.070	36.47	
B	1	0.090	45.88	44.16±1.15
	2	0.085	43.53	
	3	0.084	43.06	
C	1	0.090	45.88	44.31±1.04
	2	0.086	44.00	
	3	0.084	43.06	
D	1	0.090	45.88	44.94±0.94
	2	0.086	44.00	
	3	0.088	44.94	
E	1	0.086	44.00	42.27±2.61
	2	0.084	44.47	
	3	0.074	38.35	

**Figure 1.** replaced are mass of MSP: TiO₂ membrane A 20:1; B 20:3; C 20:5; D 20:7; E 20:9.

This is because MSP has a carboxyl (-COOH) and alkyl (R-) functional group, which is negatively charged and will interact with the charged Mn (II) positive, so there is an attractive force and forms a precipitating floc.^[18, 23] The added mass of anatase TiO₂ functions as a photocatalyst, which speeds up the reaction indicated by the light because it has a semiconductor structure. The more the added mass of TiO₂, the more hydroxide (OH⁻) produced so that the ability to decrease Mn (II) ion concentration in the sample ^[19]. However

the increasing mass of TiO₂ on B, C, D, and E of the MSP-TiO₂ membrane resulted in a lower percentage decrease of Mn (II) ions than in MSP-TiO₂ membrane A because on the MSP-TiO₂ membrane with a mass of TiO₂ 1g, TiO₂ coats the surface of the Moringa seed powder, so that the contact between ultraviolet light and TiO₂ occurs maximally and the formation of OH radicals is also increasing, so the ability to reduce Mn(II) to Mn(0) is getting better. so that the level of Mn(II) in the water is reduced to the maximum. As the amount of TiO₂ (3; 5; 7; and 9

g) was added, the TiO_2 in addition to coating the surface of the moringa seed powder also entered the pores of the moringa seed powder and resulted in a reduction in the concentration of TiO_2 in contact with ultraviolet light, so that the decrease in the level of Mn(II) in the water is decreasing (Slamet et al., 2006). According to Xue et al (2011), the photocatalytic process is adsorption that occurs on the surface of the particles.

Based on research on decreasing Mn (II) concentration in water using MSP- TiO_2 membrane with mass variations of TiO_2 1; 3; 5; 7; and 9 g, the results shown in Figure 1 that there was a decrease in Mn (II) concentrations non-significant because MSP- TiO_2 membrane thickness is not the same which is between 0.059-0.067 mm. The morphology of scanning electron microscopy (SEM) of MSP, TiO_2 , and a mixture of MSP- TiO_2 membranes with TiO_2 mass 1, 3, 5, 7, and 9 g are presented in Figure 2 and EDX in Figures 3-5.

Figure 2 presents the morphology of MSP, TiO_2 , MSP- TiO_2 membrane with TiO_2 mass (1; 3; 5; 7; 9 g). The MSP has many open pores, while in the MSP- TiO_2 membrane, however with an increase in TiO_2 mass more TiO_2 particles enter the pores of the MSP, thus blocking UV light that reacts with the surface of TiO_2 . The contact between TiO_2 and ultraviolet light against MSP powder is not maximal because TiO_2 not only coats the surface of the MSP but the open gap (pores) in the MSP is also covered by TiO_2 . TiO_2 , which covers the open gap of the MSP, can reduce contact between ultraviolet light and TiO_2 , so that the OH radicals formed are decreasing, and causing a decrease in Mn (II) concentrations also getting smaller [15].

Based on Figure 3-5 EDX spectrum mass % compound of MSP, TiO_2 , and MSP- TiO_2 membrane made Graph of Mass element percentage Carbon and TiO_2 on are presented in Figure 6.

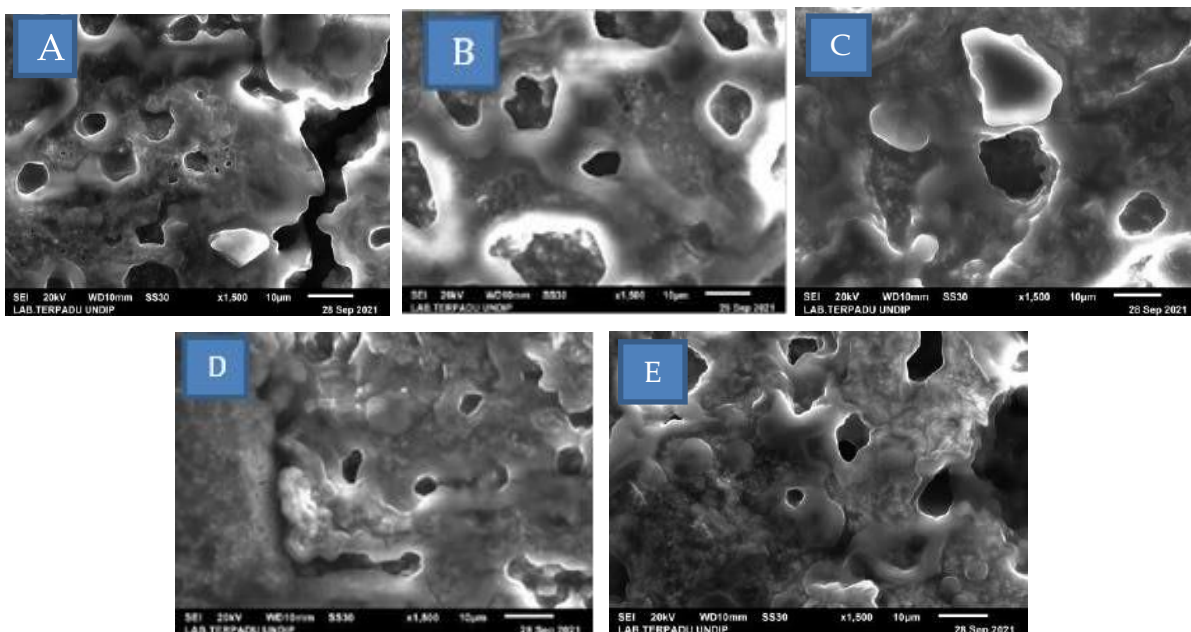
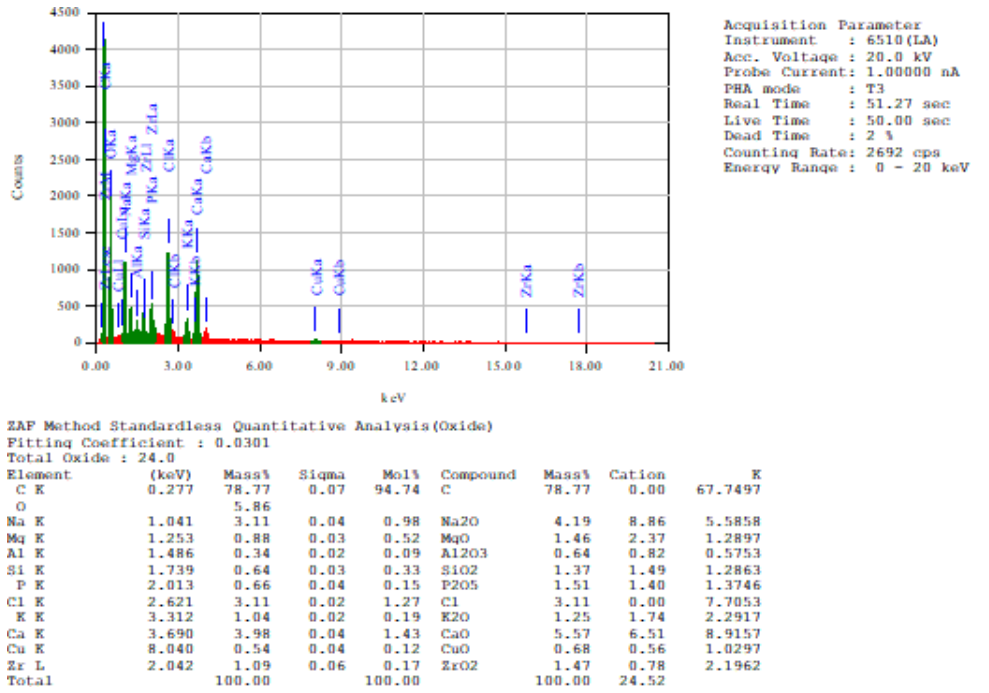
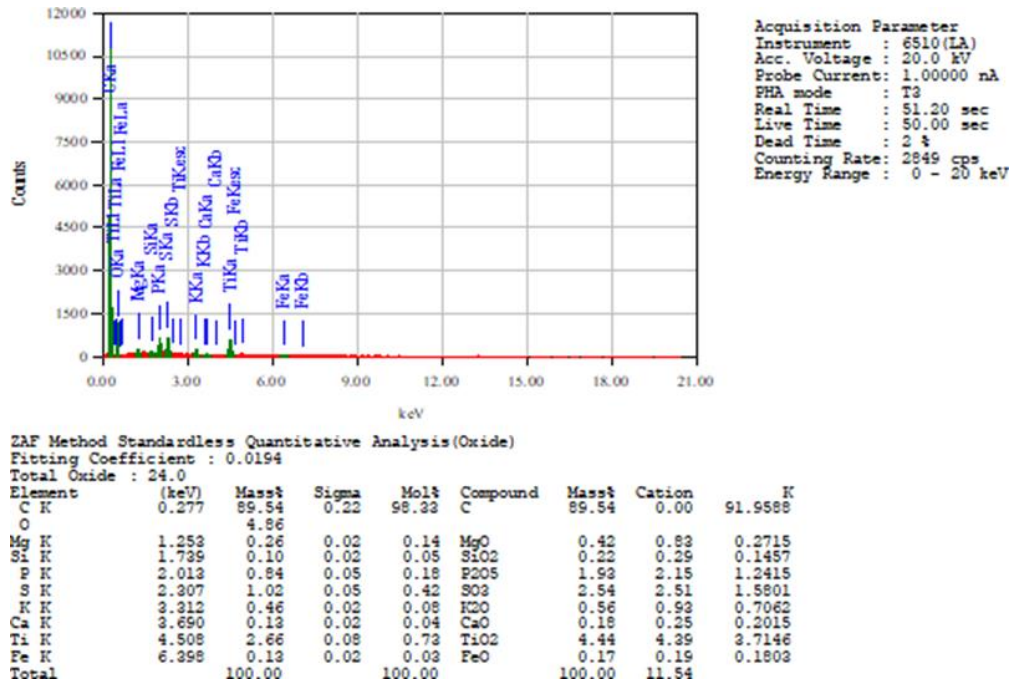


Figure 2. Morphology SEM with 1500x OF MSP, TiO_2 , and MSP- TiO_2 Membrane with TiO_2 mass A 1g; B 3g; C 5g; D 7g; E 9g (The integrated Laboratory of Diponegoro University Semarang)

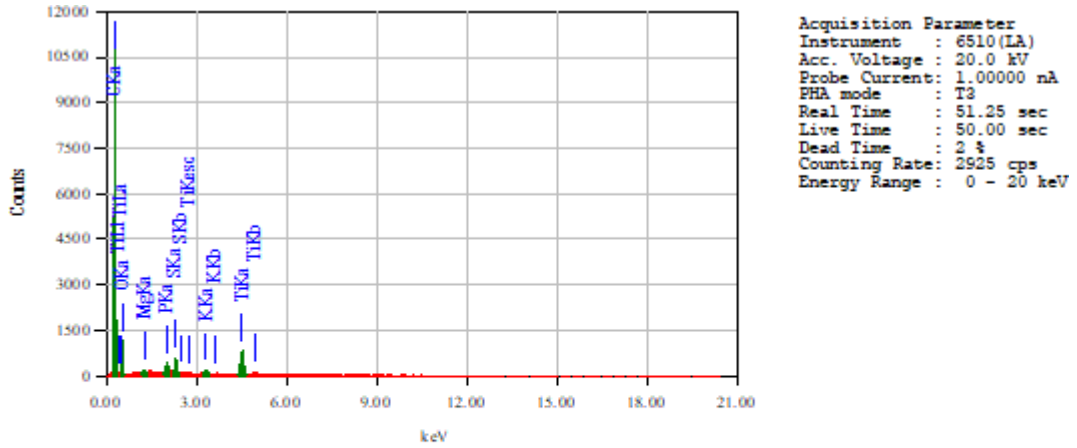


(a)



(b)

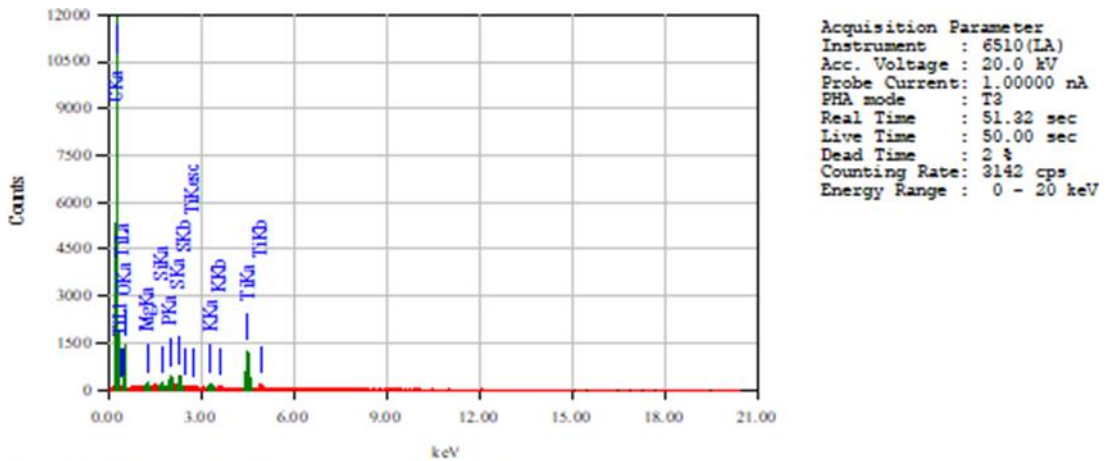
Figure 3. EDX MSP/TiO₂ with variation TiO₂ mass: a) Moringa Seed Powder, b) with the addition of 1 g TiO₂ (JEOL 2300 from UPT Laboratorium Terpadu Universitas Diponegoro, 2021).



ZAF Method Standardless Quantitative Analysis(Oxide)
 Fitting Coefficient : 0.0192
 Total Oxide : 24.0

Element	(keV)	Mass%	Sigma	Mol%	Compound	Mass%	Cation	K
C K	0.277	89.86	0.03	98.39	C	89.86	0.00	92.5648
O		4.61						
Mg K	1.253	0.15	0.01	0.08	MgO	0.25	0.52	0.1514
Si K	2.013	0.54	0.02	0.11	P2O5	1.24	1.45	0.7479
P K	2.307	0.87	0.02	0.36	SO3	2.17	2.26	1.2781
S K	2.312	0.30	0.01	0.05	K2O	0.36	0.63	0.4275
Ti K	4.508	2.67	0.04	1.01	TiO2	6.12	6.38	4.8305
Total		100.00		100.00		100.00	11.24	

(a)

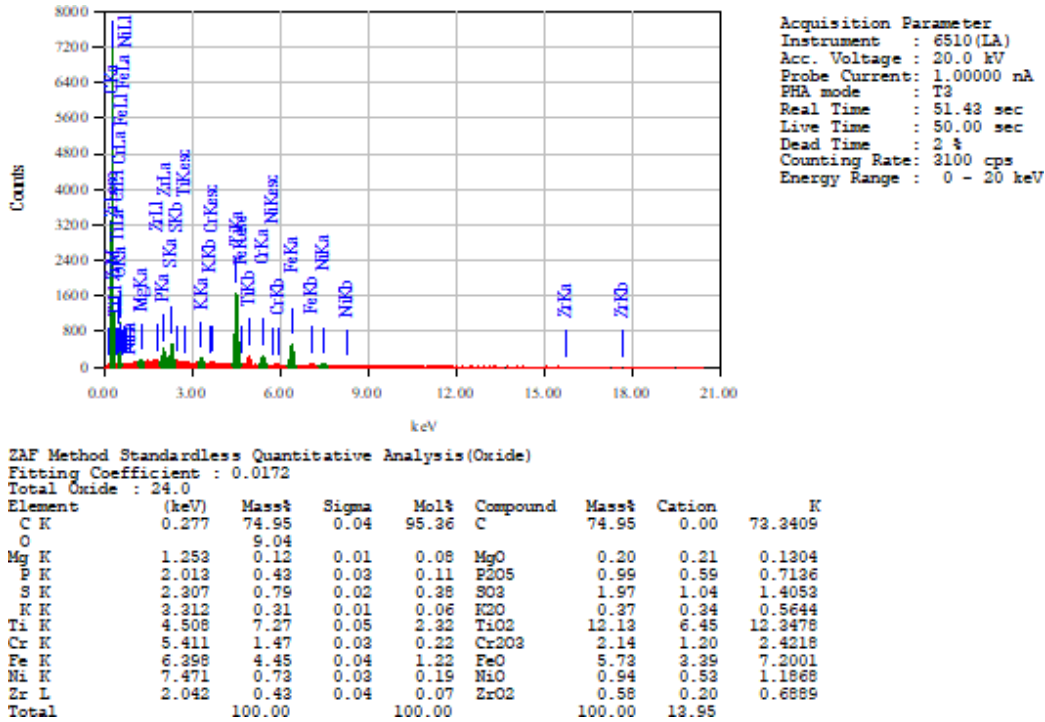


ZAF Method Standardless Quantitative Analysis(Oxide)
 Fitting Coefficient : 0.0165
 Total Oxide : 24.0

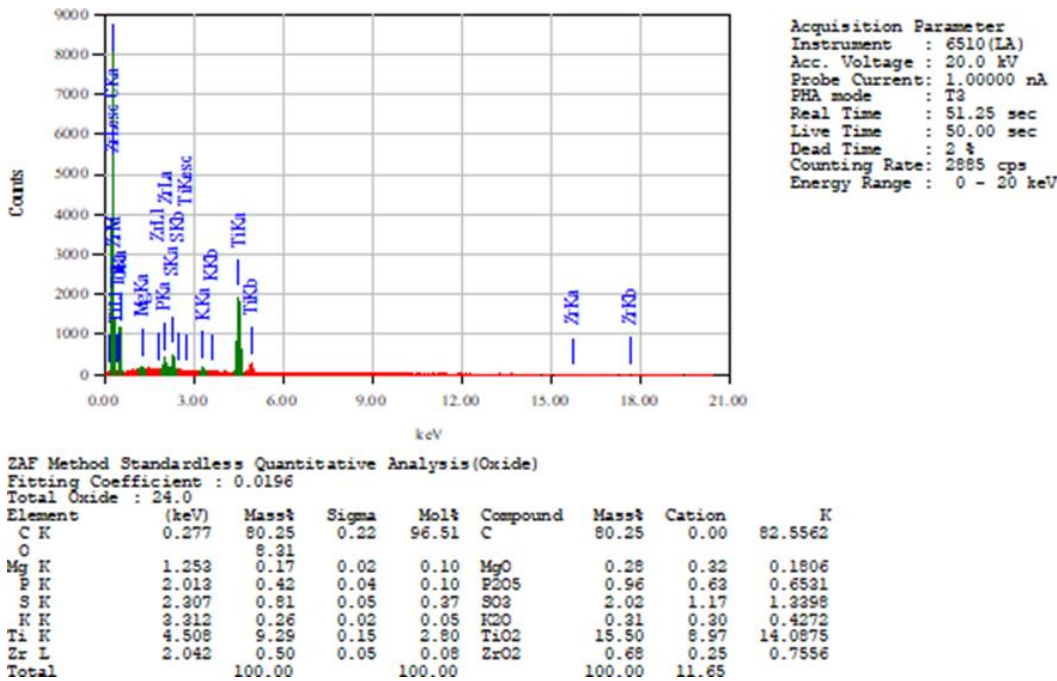
Element	(keV)	Mass%	Sigma	Mol%	Compound	Mass%	Cation	K
C K	0.277	87.61	0.03	97.96	C	87.61	0.00	90.5657
O		5.44						
Mg K	1.253	0.15	0.01	0.08	MgO	0.25	0.44	0.1505
Si K	1.739	0.10	0.02	0.05	SiO2	0.21	0.25	0.1229
P K	2.013	0.52	0.02	0.11	P2O5	1.19	1.18	0.7227
S K	2.307	0.67	0.02	0.28	SO3	1.68	1.48	1.0018
K K	2.312	0.29	0.01	0.05	K2O	0.35	0.53	0.4311
Ti K	4.508	5.22	0.04	1.46	TiO2	8.71	7.70	6.9953
Total		100.00		100.00		100.00	11.58	

(b)

Figure 4. EDX MSP/TiO₂ with variation TiO₂ mass: a) with the addition of 3 g TiO₂ b) with the addition of 5 g TiO₂ (JEOL 2300 from UPT Laboratorium Terpadu Universitas Diponegoro, 2021).



(a)



(b)

Figure 5. EDX MSP/TiO₂ with variation TiO₂ mass: a) with the addition of 7 g TiO₂ b) with the addition of 9 g TiO₂ (JEOL 2300 from UPT Laboratorium Terpadu Universitas Diponegoro, 2021).

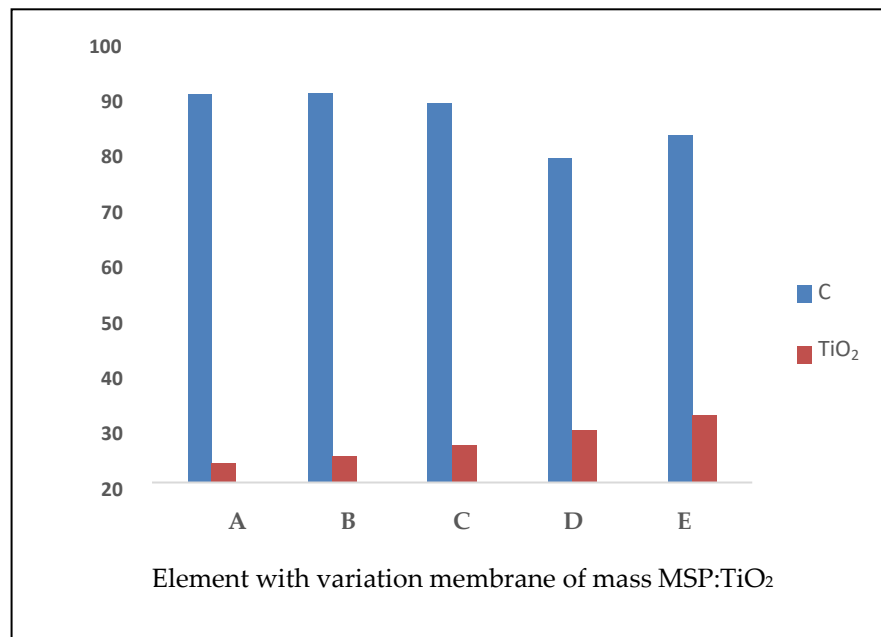


Figure 6. Graph of Mass element Percentage Carbon and TiO₂ on with MSP-TiO₂ membrane with TiO₂ mass A, B, C, D, dan E 1; 3; 5; 7; and 9 g.

Figure 6 shows the highest component of MSP 95.59% carbon on MSP/TiO₂ membrane with variation mass TiO₂. The MSP-TiO₂ membrane with a fixed MSP mass of 20 g and a TiO₂ mass of 1, 3, 5, 7, and 9 g, the percentage of TiO₂ mass increased from 4.44 to 15.50%, while the amount of carbon in MSP decreased from In 95.59% to 80.25 %. According to research [23], MSP has a functional group (active site) that can bind/adsorb metal ions, the result is that the MSP powder biosorbent has 5 main functional groups, namely carbonyl groups (C=O), hydroxyl (OH), amides (NH), alkenes (C=C), and Nitro (NO₂) which negatively charged which can bind to the positively charged Mn(II) ion.

This research is supported by [17] that the mass ratio of zeolite-TiO₂ is 100:1; 100:3; 100:5 can degrade the dye rhodamine B has the highest surface area of 73.913 m²/g at a ratio mass zeolite-TiO₂ 100:5. At a ratio of zeolite-TiO₂ 100:5 (100:5 if simplified to 20 g zeolite and 1 g TiO₂) is the highest increase was due to the increase in pores in the catalyst after the addition of zeolite. The more use of TiO₂ will cause more holes that react further with water Therefore (>Ti IV OH•)⁺ will reduce Mn (II) to Mn (0) or reduce the colored solution to

to form •OH radicals^[12]. The steps in the mechanism of the TiO₂ photocatalysis reaction can be written as follow:

1. Formation of charge carrier by photons (light)
 $\text{TiO}_2 + h\nu \rightarrow \text{Ti (IV) OH} + h\nu^+ + \text{ecb}^-$ (1)
2. Trapping of charge carrier
 $h\nu^+ + >\text{TiIV OH} \rightarrow (>\text{Ti IV OH}\bullet)^+$ (2)
 $\text{ecb}^- + >\text{Ti IVOH} \rightarrow (>\text{Ti III OH})$ (3)
 $\text{ecb}^- + >\text{Ti IV} \rightarrow >\text{Ti III}$ (4)
3. Recombination of charge carrier
 $\text{ecb}^- + (>\text{Ti IVOH}\bullet)^+ \rightarrow >\text{Ti IVOH}$ (5)
 $h\nu^+ + (>\text{Ti III OH}) \rightarrow >\text{Ti IVOH}$ (6)
4. Transfer of charge interface
 $(>\text{TiIV OH}\bullet)^+ + \text{Red} \rightarrow >\text{TiIV OH} + \text{Red}\bullet + (7)$
 $\text{ecb}^- + \text{Oks} \rightarrow >\text{TiIV OH} + \text{Oks}\bullet$ (8)

Note:

>TiOH = the hydrated form of TiO₂

(>Ti IV OH•)⁺ = the surface of the trap
 $h\nu^+ + (\text{Radikal } \bullet\text{OH})$

(>Ti III OH) = the surface of the trap
 ecb^-

Red (reductant) = electron donor

Oks (oxidant) = electron acceptor ^[25]

colorless. The use of TiO₂ in a certain amount will also cause the entry of TiO₂ into the pores

of Moringa seeds which blocks the incoming UV rays so that the incoming •OH radicals will reduce the photocatalyst process of TiO₂ and cannot work optimally. The lack of maximum contact between TiO₂ and UV light on Moringa seeds results in the ability to decrease Mn (II) ions concentration^[20].

Conclusions

Moringa Seed Powder (MSP)-TiO₂ membranes with variations in the mass of TiO₂ can decrease Mn (II) ions concentration in water. The highest decrease in Mn (II) contents using MSP-TiO₂ membranes at a ratio of MSP-TiO₂ 20:1 during 90 minutes of irradiation at a flow rate of 0.56 mL/minute is 33.76±0.44% with initial Mn (II) concentration 55.06±0.31ppm. Moringa Seeds Powder (MSP)-TiO₂ membrane has the potential to reduce heavy metal ion solution, especially manganese.

Acknowledgments

Thank you to the Institute for Research and Community Service, the University of Muhammadiyah Semarang for funding the Primary Lecturer Research in 2021, and the Ministry of Research, Technology and Higher Education for the Scientific Article Publication Assistance program for the fiscal year 2021.

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