International Journal of Applied Chemistry.
ISSN 0973-1792 Volume 15, Number 2 (2019) pp. 113-120

http://www.ripublication.com

Biosynthesis of Titanium Dioxide using Sargassum sp. Seaweed Extrac under Microwave

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Abstract

Biosynthesis is a new method used in the synthesis of nanoparticles by utilizing extracts of dyes from natural materials. The purpose of this study was to synthesize titanium dioxide (TiO_2) using Sargassum sp. extract. Sargassum sp. mixed with distilled water (1:4 w/v) then heated in the microwave for 1 minute with various power level at 10; 20; 30; 40 and 50. Extracts of the dyes produced were then characterized using UV-Vis and GC-MS spectrophotometers. The extract obtained was dropped in an TiO_2 solution with various concentration 0.001; 0.005; 0.01; 0.02 and 0.03 M. Then heated in the microwave for 5 minutes and obtained colloidal titanium dioxide. The results of the analysis using UV-Vis showed that the maximum dye extract was obtained at power level 30. The results of the analysis using GC-MS showed that the compound content in the Sargassum sp. Extract. using distilled water there are 15 peaks and the largest content is Hexadecanoic acid, methyl ester compound with a peak area of 79.72%. Based on analysis using UV Vis obtained that Sargassum sp. extract can be used as a bioreductor in the preparation of titanium dioxide. The Particle Size Analyzer (PSA) was used to obtain a particle diameter with z-average is 2322 nm.

Keyword: Biosynthesis; TiO_2; Sargassum sp. extrac; microwave.

INTRODUCTION

Sargassum sp. is a seaweed group of Phaeophyceae found in the tropics, including in Indonesia. Sargassum seaweed is widespread in Indonesia, one of which is along the coast of Sumbawa Island - Bima. Sargassum species that grow in Bima such as Sargassum sp., Sargassum duplicatum and Sargassum polycystum. During this time, the use of Sargassum sp. in Bima it is only sold as raw material for export [1].

Sargassum sp. contains alginate and iodine compounds used in the food, pharmaceutical, cosmetic and textile industries [2]. The chemical composition and pigment found in brown seaweed is the result of photosynthesis, the amount of which varies greatly depending on the type, the period of development and the conditions in which it grows. Sargassum sp. contains active compounds such as steroids, alkaloids, phenols, and triterpenoids that function as antibacterial, antiviral and antifungal [3]. In addition, the compound is able to act as a reducing agent because it has a strong tendency to reduce metals. Bioreductor is a reduction method by utilizing biological materials both microorganisms and plants as reducing agents for metal ions. The ability to chelate and reduce metals in phenol compounds because it has a high nucleophilic character from aromatic rings [4]. Seaweed Sargassum sp. has more ability than other natural materials as bioreductors. The use of bioreductors in the synthesis of nanoparticles is known as the biosynthetic method [5].

A variety of methods have been used in the extraction of dyes including maceration, reflux, and soxlets. Development of extraction methods to speed up extraction time and reduce the amount of solvent needed. Lately, the Microwave Assisted Extraction (MAE) extraction method has been widely used to extract active compounds in natural substances [3]. MAE extraction method utilizes microwave radiation to accelerate selective extraction through fast and efficient heating of solvents. The advantages of extraction using the MAE method are reduced extraction time and the use of fewer chemical solutions [4]. According to some research results, the MAE method increases the efficiency and effectiveness of extraction of active ingredients of various types of spices, herbal plants and fruits [5]. MAE method has advantages such as shorter time needed, less solvent needed, reducing energy consumption, suitable for thermolable components, giving higher extraction results, higher accuracy and precision [6].

In this research, TiO2 biosynthesis was carried out using Sargassum sp. Dye extraction from seaweed Sargassum sp. microwave method is used so that the extraction process can be short-lived. In the biosynthesis of TiO2 nanoparticles, microwave is also used because the process is easy, fast, very cheap and environmentally friendly.

EXPERIMENTAL METHOD

Extraction of Bioreductor

Sargassum sp. Seaweed powder mixed with distilled water at a ratio of 1:4 (w/v) then heated in the microwave for 1 minute at power level 10; 20; 30; 40 and 50. the mixture obtained is then filtered until Sargassum sp. extracts obtained were further analyzed using UV-Vis spectrophotometers and GC-MS.

Biosynthesis of TiO_2

TiO_2 synthesis was carried out by mixing 90 mL $1x10^{-3}$ M TiO_2 solution with 10 mL of Sargassum sp. Extract. then heated in the microwave for 5 minutes. The same treatment was used for synthesis at $5x10^{-3}$; $1x10^{-2}$; $2x10^{-2}$ dan $3x10^{-2}$. The mixture obtained was then analyzed using a UV Vis spectrophotometer and PSA.

RESULT AND DISCUSSION

This research was conducted to synthesize titanium dioxide using bioreductor from seaweed extract Sargassum sp. Synthesis methods that use reducing agents from natural materials are known as biosynthesis. The process of dye extraction and titanium dioxide biosynthesis in this study was carried out with the help of microwave radiation. In the process of extracting dyes from Sargassum sp. carried out using a microwave for 1 minute. The use of microwaves in the extraction process is due to the energy in the microwave extraction process directly to specific target samples and specific ways, so that no heat is lost to the environment, because the heating process takes place in a closed system [10]. This unique heating mechanism can significantly reduce the time needed for the extraction process [11]. Microwave heating involves three times the conversion of energy, namely electrical energy into electromagnetic energy, then kinetic energy and subsequently heat energy [12].

Based on the graph shows that the optimum conditions for the extraction of dyes from Sargassum sp. performed at power level 30 for 1 minute. The absorption peak occurs at a wavelength of 269 nm with an absorbance of 4.498. The use of microwaves in the extraction of dyes from brown algae Sargassum sp. can be said to be better because the extraction process takes place quickly and maximum results. Wiraningtyas research results (2019), explained the extraction of dyes from Sargassum sp by maceration method produced extract with absorbance of 3,883 with extraction time for 2 days while extraction by microwave method produced extract with absorbance of 3,371 with extraction time for 20 minutes [13]. The advantages of the microwave method being an alternative as a substitute for conventional heating, where heat transfer occurs through the heat gradient. Whereas in the microwave, heating occurs through direct collisions between polar material and microwaves which are regulated by two phenomena, namely ionic conduction and dipole rotation which take place simultaneously. As a result, energy transfer takes place faster and has the potential to improve product quality [14]. Microwave helps the process of extracting dyes by giving energy directly to the material. With the direct energy to the material, the direction of energy transfer and mass transfer becomes unidirectional, so that the extraction process is faster [15].

The heating process uses a microwave starting from outside the surface then conduction into the sample so that the inside of the sample will also be heated. However, the higher the microwave power level used in the extraction process will cause a decrease in the absorbance value of the extract. This is because high heating causes changes in the structure of compounds and bioactive compounds in the extract of Sargassum sp. Graph of measurement results of absorbance of Sargassum sp. on the variation of the microwave power level as shown in Figure 1. Based on the UV Vis graph in Figure 1 shows that the process of extracting seaweed Sargassum sp. produce extracts with optimum absorbance at level 30.

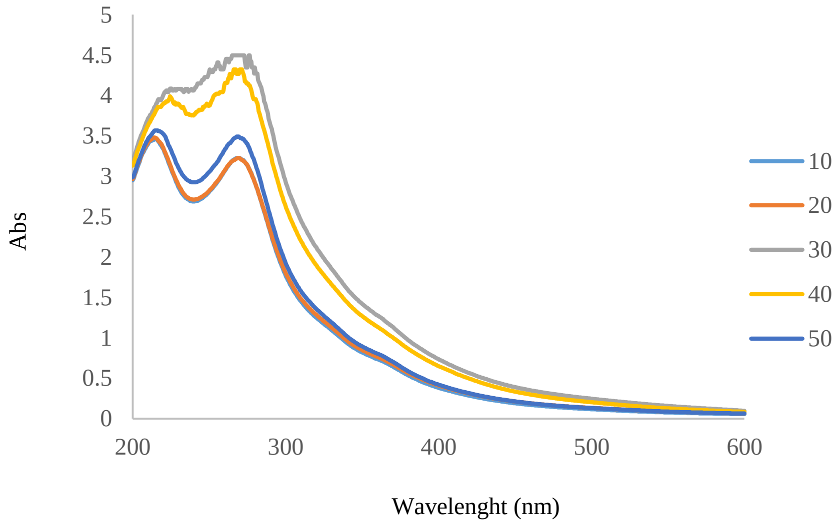

Figure 1. UV Vis graph of Sargassum sp. on Power Level Variations

While at the same wavelength obtained low absorbance values at power levels 40 and 50 are 4,323 and 3,492. The decrease in absorbance may be due to excessive heat degradation of the dye. The same was stated by Handayani (2014) that polyphenol compounds will degrade if excessive heating occurs [16]. At too high a power can cause degradation of dyestuff compounds so that the resulting product is getting smaller [17].

To find out the content and structure of compounds contained in the extract of Sargassum sp. the extraction results were carried out by analysis using Gas Chromatography-Mass Spectroscopy (GC-MS). The samples analyzed were extracted from Sargassum sp. using methanol and distilled water assisted microwaves at power level 30 for 1 minute. Chromatogram of Sargassum sp. using distilled water as shown in figure 2.

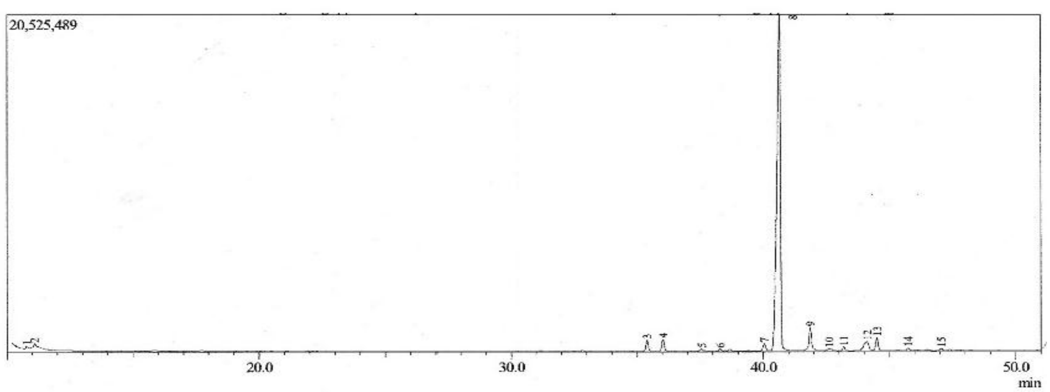

Figure 2. Chromatogram of Sargassum sp. Extract

Based on Figure 2, it was found that Sargassum sp. using aquades solvent shows 15 peaks detected. Based on the chromatogram, there is one of the most dominant peaks seen from the percent area, namely Hexadecanoic acid, methyl ester which is 79.72%. Data on the content of compounds in Sargassum sp. as in table 1.

Table 1. GC-MS Results Data of Sargassum sp. Extract.

Peak	Retensi Time	Compound Name	Formula	Mol Weight	Peak area %
1	10.783	1-nonena	C_9H_{18}	126	0,43
2	11.083	nonena	C_9H_{20}	128	1,34
3	35.383	hexadecane	$C_{16}H_{34}$	226	1,69
4	36.016	Decanoic Acid, methyl ester	$C_{11}H_{22}O_2$	186	1.99
5	37.540	1-Hexadecanethiol	$C_{16}H_{34}S$	258	0.54
6	38.288	Tetradecanoic acid, methyl ester	$C_{15}H_{30}O_2$	242	0.42
7	40.047	9-Octadecanoic acid, methyl ester	$C_{19}H_{36}O_2$	296	1.40
8	40.655	Hexadecanoic acid, methyl ester.	$C_{17}H_{34}O_2$	270	79.72
9	41.857	Nonadecanoid acid, methyl ester	$C_{21}H_{42}O_2$	326	4.10
10	42.602	1-Decanol	$C_{15}H_{30}O_3$	258	0.75
11	43.197	2-Norpinanol,3,6,6-trimethyl	$C_{10}H_{18}O$	154	0.67
12	44.130	9-Hexadecanoic acid, methyl ester	$C_{17}H_{32}O_2$	268	3.40
13	44.506	Octadecanoic acid, methyl ester	$C_{19}H_{38}O_2$	298	2.50
14	45.743	6-octadecrnoid acid, methyl ester	$C_{19}H_{36}O_2$	296	0.61
15	47.057	Methyl arachidonate	$C_{21}H_{34}O_2$	318	0.46

In the extraction process Sargassum sp. brown. The extract obtained was then mixed with a 1x10^{-3} M TiO_2 solution as a precursor. In the biosynthesis process, TiO_2 is also used by microwave to speed up the synthesis process. The synthesis process is carried out at power level 30 with variations in the concentration of TiO_2 solution. The result of the synthesis is obtained colloidal titanium dioxide and a white precipitate is formed. The results of TiO_2 colloidal characterization using UV Vis spectrophotometer as shown in the following figure 3.

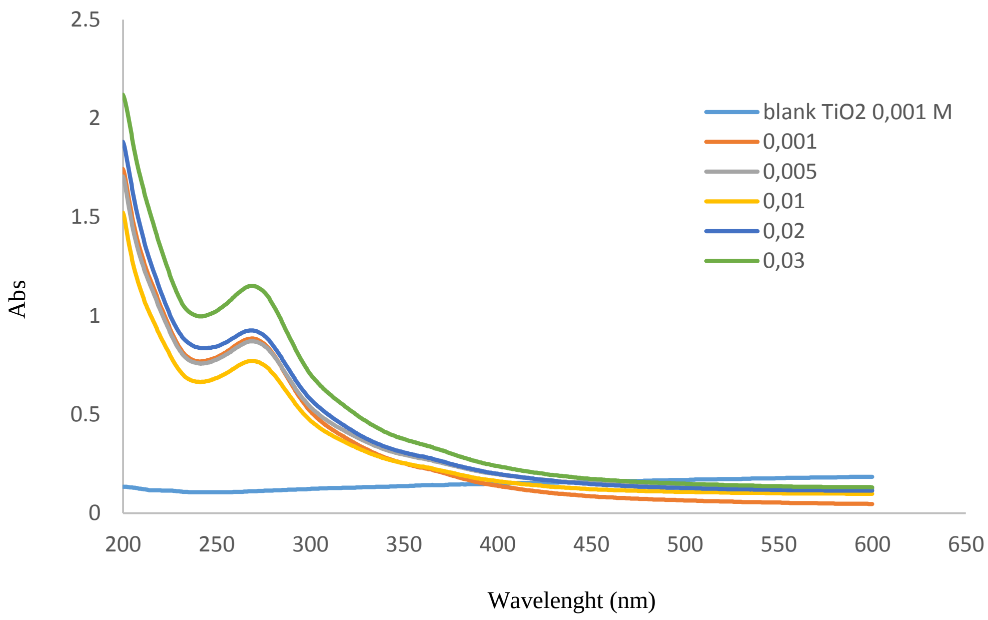

Figure 3. UV Vis graph of TiO2 colloids in various concentrations

Based on the graph in figure 3 shows that the UV Vis graph of the precursor of TiO2 solution did not produce a significant peak. While the UV Vis graph of the TiO2 colloid produces a peak at the wavelength having a peak shift at 271 nm. Shifting the absorbance peak indicates that the colloidal TiO2 nanoparticles have formed with varying sizes. In addition, the shift in the absorption peak is caused by the bond between the silver particles and the OH group in Sargassum sp. Extract. thus increasing the amount of energy needed at the time of electron excitation [13]. Biosynthesis of TiO2 nanoparticles was carried out with variations in the precursor concentration. Based on the UV Vis graph in figure 3, it was found that silver nanoparticles obtained the optimum product at a concentration of 0.03 M.

Particle size greatly affects the nature and performance of a material. The smaller the size of a particle, the better its properties and performance. TiO2 nanoparticles produced in this study were measured using Particle Size Analysis (PSA). Data from the measurement of the diameter of silver particles as shown in Figure 4. Based on the graph in Figure 4 that the size of the silver nanoparticles synthesized has a diameter that varies from 1363 to 7532 nm. While the average size of the particle diameter is 2322 nm.

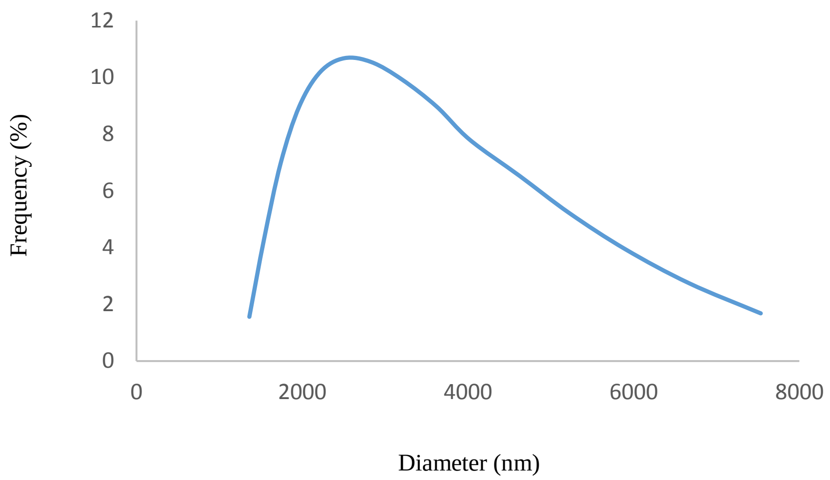

Figure 4. Particle Distribution of TiO_2 Nanoparticles

CONCLUSION

Titanium dioxide (TiO_2) can be synthesized using Sargassum sp. The extraction and biosynthesis process can be done with the help of a microwave. Sargassum sp. Seaweed extraction. produces extracts with optimum absorbance at level 30. Biosynthesis of TiO_2 at the variation of precursor concentration obtained optimum conditions at the synthesis of concentrations of 0.03 M. While the average size of the particle diameter is 2322 nm.

REFERENCES

[1] Amir, A. Wiraningtyas, A. Ruslan. 2019. Extraction of Sodium Alginate From Sargassum sp. using Microwave Assisted Extraction. The Journal of Pure and Applied Chemistry Research. 8 1 23-30

[2] Michalak, A. 2006. Phenolic Compound and Their Antioxidant Activity in Plants Growing under Heavy Metal Stress. *Polis Journal of Environmental Study*. 15 (4); 523-530.

[3] Abdel-Rauf, N. Al-Enazi, N.M. Ibraheem. I.B.M. Alharbi, R.M. Alkhulaifi, M.M. 2018. Biosynthesis of Silver Nanoparticle by Using of the Marine Brown Alga *Padina pavonia* and Their Characterization. Saudi Journal of Biological Science. https://doi.org/10.1016/j.sjbs.2018.01.007.

[4] Rajeshkumar, S., Malarkodi, C., Paulkumar, K., Vanaja, M., Gnanajobitha, G., and Annadurai, G. 2014. Algae Mediated Green Fabrication of Silver Nanoparticles and Examination of Its Antifungal Activity agaınts Clinical

Pathogens. *International Journal of Metals*. 2014; 1-8.

[5] Carrillo-Lopez, L.M. Zavaleta-Mancera, H.A. Vilchis-Nestor, A. Soto-Hernandez, R.M. Arenas-Alatore, J. Trejo-Tellez, L.I. Gomez-Merino, F. 2014. Biosynthesis of Silver nanoparticles Using Chenopodium ambrosioides. *Journal of nanomaterials*. 1-9.

[6] Silva, M., Gomes, F., Oliveira, F., Morais, S., Matos, C.D. 2015 *Inter. Journal of Chemical, Nuclear, Materials and Metallurgical Engineering* **9** 1 30-33

[7] Amir, A. Wiraningtyas, A. Ruslan 2019 The Journal of Pure and Applied Chemistry Research 8 1 23-30

[8] Ghallisa, K.N., Nugroho, W.A., Hendrawan, Y. 2014 *Jurnal Bioproses Komoditas Tropis* **2** 1 72-78

[9] Handayani, D., Mun'im, A., Ranti, A.S. 2014 *Traditional Medicine Journal* **19** 1 29-35

[10] Jain, T., Jain, V., Pandey, R., Vyas, A., & Shukla, S., 2009, *Microwave Assisted Extraction for Phytoconstituents – An Overvie,* Asian J. Research Chem, 2 (1), pp. 19-25.

[11] Mandal, V., Mohan, Y., & Hemalatha, S. 2007. *Microwave Assisted Extraction - An Innovative and Promising Extraction Tool for Medicinal Plant*, Research. Phcog Rev., 1(1), 7-18.

[12] Salari, Z., Danafar, F., Dabaghi, S., Ataei, S.A. 2016. Sustainable Synthesis of Silver Nanoparticles using Macroalgae Spirogyra varians and Analysis of Their Antibacterial Activity. *Jurnal of Saudi Chemical Society*. (2016) 20; 459-464.

[13] Wiraningtyas, A. 2019 *JURNAL REDOKS: JURNAL PENDIDIKAN KIMIA DAN ILMU KIMIA*, **2** 1 1-10

[14] Farida, R. Nisa, F.H. 2015 *Jurnal Pangan dan agroindustri* **3** 2 362-373

[15] Adiksana, A. Jumardi, A. Kusyanto. Andriani R. 2017 *Journal of Research and Technology* **3** 1 63-69

[16] Gunawan, E.R. Suhendra, D. 2012 *Molekul* **7** 2 95-104

[17] Widoretno, D.R. Kunhermanti, D. Mahfud. Qadariyah, L. 2016 *Jurnal Teknik ITS* **5** 2 237-241