

Understanding the Corrosion Behaviour of Graphite in Peat Environment for Environmental and Sustainability Applications

Nurettin ÇEK^{1,*}, Ayhan ORHAN²

Abstract

Graphite, which is one of the most remarkable carbon materials for industrial applications, is a heat exchanger component material in the phosphoric acid industry, bipolar layer or electrode material in vanadium redox flow batteries, electrode material for advanced oxidation processes, electrode material for lead-acid batteries, electrode material in fuel cells and microbial fuel cells, and more interesting, as moderator or core material in nuclear reactors used. Corrosion, one of the most important problems of graphite materials widely used in such important areas of the industry, has not been adequately addressed, although it is an issue of primary importance for the long-term stability of carbon materials. Moreover, since corrosion studies of graphite have mostly focused on its performance in artificial environments, studies on how they would perform in “real world situations” are quite poor. Based on these deficiencies, in this study, graphite corrosion was investigated in aqueous peat environment, which is a natural material, anaerobic, contains bacteria and provides a complex environment, in order to represent real world conditions. Experiments performed to determine corrosion behaviour show that graphite material is prone to corrosion in the peat environment and pitting corrosion is especially dominant. However, graphite exhibited good corrosion resistance in the peat environment and its corrosion rate was around 1.117×10^{-7} mm/year. This is in the very low corrosion category (≤ 0.025 mm/year) according to the corrosion rate classification of equipment in the chemical industry. Therefore, graphite is promising as a more sustainable, corrosion-resistant electrode material for fuel cells, microbial fuel cells, biosensors.

Keywords: Peat, graphite, corrosion, real world condition, corrosion category

INTRODUCTION

Graphite is one of the most remarkable carbon materials for industrial applications due to its self-

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lubricating properties, good mechanical properties, good electrical and thermal conductivity and biocompatibility, and chemical and electrochemical properties [1,2]. There is a strong interest in graphite in the manufacturing of component materials of some equipment. For example; graphite is available as the component material of heat exchanger required for various processes in the phosphoric acid industry [1]. Additionally, graphite is used as the bipolar layer, which is the electrode component in fuel cells [3,4]. Similarly, graphite material is used as a bipolar layer or electrode in vanadium redox flow batteries [5]. Additionally, graphite is used as the electrode material in microbial fuel cells [6,7]. Moreover, graphite is used as the electrode material required for electrochemical reactions in electrochemical

advanced oxidation processes [8]. Additionally, graphite has been used to make electrode materials called graphite-lead metallic composites in lead-acid batteries [9]. More interestingly, graphite has been used as moderator or core material in more than 85 nuclear reactors worldwide and specifically [10]. Moreover, the International Atomic Energy Agency report mentions that some graphite blocks from moderator repairs stored near nuclear reactors in Russia constitute waste [11].

Graphite, a material widely used in scientific research and industrial applications, has both strengths and weaknesses. One of these weaknesses is that graphite can corrode. Although corrosion, which occurs not only in graphite but also in other carbon materials, is an issue of primary importance for the long-term stability of carbon materials, it has not been adequately addressed [8]. Although corrosion is defined as the change in the chemical and physical properties of metal materials as a result of chemical or electrochemical interaction with their environment [12], it is a serious problem not only in metals but also in carbon-based materials and especially in graphite [8,13]. For example, Graphite electrodes used in electric arc furnaces create great costs and are subject to corrosion. To reduce this cost and corrosion damage, graphite electrodes were coated with SiC/TiO₂ [13]. In addition, the results of the study investigating the effects of graphite on the corrosion behaviour of aluminium-graphite composite in sodium chloride solution show that the aluminium corrosion rate augments with the increase in the presence and content of graphite [14]. In studies on coating [15], graphite is often stirred with particulate conductive materials or pre-processed as the conductive ambient in the procedure in order to provide outperform electrical conductivity. But there is a need for further research on the effect of non-graphite pretreatment and as a sole conductive ambient above conductive coatings feature. In a study conducted within the scope of this requirement, focused on the wear behaviour and corrosion resistance of graphite coating in order to provide electrical conductivity to micro arc oxidation coatings including magnesium alloy. Here, the sample with the graphite content of 80 wt% provided excellent corrosion resistance, exhibiting the maximum module of impedance, corrosion potential (-0.253 V) and the minimum corrosion current density of 1.644×10^{-7} A/cm². Increased graphite content (100 wt%) cause more surface roughness and minus compactness for coating, reducing the corrosion resistance for composite organic layer [15].

In studies talking about the corrosion properties of graphite material, the corrosive medium (electrolyte) is alkaline (e.g., Tri-Sodium Phosphate (Na₃PO₄.12H₂O) [1]), neutral (e.g., sodium chloride (NaCl) [14]) and acidic (e.g., 0.1 mol.dm⁻³ Na₂SO₄ + 2 ppm F⁻ solution [16]) artificial environments have been investigated. However, since corrosion studies of graphite are mostly focused on its performance in artificial environments, studies on how they will perform in “real world” situations are quite poor. Therefore, studying the corrosion of graphite in a real and complex environment becomes an essential topic. To investigate this essential issue, peat material and peat water, which are in acidic form and contain high amounts of organic material, are among the materials that stand out as examples of natural environments that reflect real-world conditions [17,18].

For this reason, in this study, graphite corrosion was investigated in the aqueous peat environment, which is a natural material, anaerobic, contains bacteria and provides a complex environment [17,18], in order to represent real world conditions.

Experimental

Square graphite electrode (10 mm diameter, 150 mm length) material was purchased from Mass Döküm (Konya/Turkey). The product sold in the market as Orkide Peat (Ideal Torf Ticaret, Bolu/Turkey) was used as peat material. Experiments were carried out in a glass container (55×55×55 mm). A potentiostat/galvanostat device (IVIUM Vertex.1A) was used for corrosion experiments. The silver/silverchloride (Ag/AgCl) reference electrode required as device equipment in the corrosion experiments was supplied from Landt Instruments (United States) and the platinum auxiliary electrode was supplied from BASi (United States). Weight measurements were made using an analytical balance (Ohaus PR523).

Inspired by previous studies [19,20], to prepare the appropriate electrolyte for corrosion tests, the peat was dried at 105°C for 5 hours and cooled in air before weighing. Then, the dry peat and distilled water ratio was mixed. For these 50 grams of dry peat material and 100 grams of pure water were used for the mixture. Corrosion tests were performed by creating a three-electrode electrochemical cell in an aqueous peat solution environment in a glass container using a potentiostat/galvanostat device. In the three-electrode electrochemical cell, graphite material was used as the working electrode, while platinum material was used as the auxiliary electrode and Ag/AgCl as the reference electrode [14,15]. In order to better understand the graphite material's corrosion behaviour in peat, open circuit potential, linear polarization resistance, potentiodynamic test, alternating polarization, galvanic corrosion, electrochemical impedance spectroscopy and Tafel analyses were carried out. All tests were performed at room temperature (298 K).

Open circuit potential (OCP) measurement was performed by recording the potential value every second against time and continued until a stable (fixed) potential value was achieved.

To determine linear polarization resistance (LPR), measurements were first made using a scan rate of 1 mV/s in the range of ± 20 mV against stable OCP. Potential and current values were determined, and current density was calculated by dividing the current value by the graphite electrode geometric surface area. The resistance of the graphite material was revealed from the slope of the potential curve versus current density [21].

Potentiodynamic testing was performed by recording data obtained using a scan rate of 1 mV/s in the range of ± 250 mV against stable OCP [22].

Cyclic polarization was started from 150 mV below the OCP value and continued up to 1500 mV above the initial value with a potential scanning rate of 1 mV/s, and the scanning was completed by reversing when the current density reached 5 mA/cm² [23].

Since galvanic corrosion results from the potential difference between materials, galvanic corrosion detection was carried out by monitoring the galvanic potential and galvanic current against time [24].

Electrochemical impedance spectroscopy (EIS) tests were performed in the frequency range from 100 kHz to 10 mHz, taking a potential amplitude of 10 mV and 10 frequency points per logarithmic decade [25]. EIS results were evaluated by creating Nyquist diagrams [26]. The equivalent circuit proposed by IVIUM software was used to estimate the resistance behaviour of the material [25].

Parameters such as corrosion potential (E_{corr}), corrosion current density (I_{corr}) and corrosion rate are obtained through Tafel tests, also referred to as Tafel extrapolation. This test was performed at a scan rate of 0.167 mV/s over a potential range of ± 250 mV against a stable OCP value [27]. After the Tafel tests, the graphite electrode was dried at 105°C and the mass change during the corrosion tests was recorded [8]. By applying the mass change data to the Tafel findings in the IVIUM software, important information about corrosion was obtained. Experiments were performed in triplicate and mean values are presented, considering standard deviations [8].

RESULTS AND DISCUSSION

OCP represents the corrosion susceptibility of the material. A shift of OCP towards more positive values indicates that it can increase the corrosion resistance of the material [22]. OCP of graphite material in peat environment was monitored for 36 hours (259200 seconds) and is shown in Figure 1. As can be seen here, the OCP value is initially approximately 130 mV, has a positive value and exhibits cathodic properties. Here, the graphite material showed electrochemical behaviour similar to a noble metal such as gold or platinum. However, towards the end of the OCP measurement, although the potential reached approximately -75 mV, it remained constant at approximately -43 mV and showed an

anodic feature. This shows that the corrosion resistance of the material decreases. In general, especially for the first 24 hours, the OCP curve obtained in this study was similar to the carbon-based material introduced as “Tepex-carbon fiber reinforced polymer/steel” [25]. In addition, the progression of the OCP value from positive to negative is similar to the behaviour of carbon steel coated with polypyrrole. A decrease in OCP indicates that the peat material penetrates into graphite and the polarization effect of graphite decreases [26]. Towards the end of the OCP measurement, the graphite material became susceptible to corrosion.

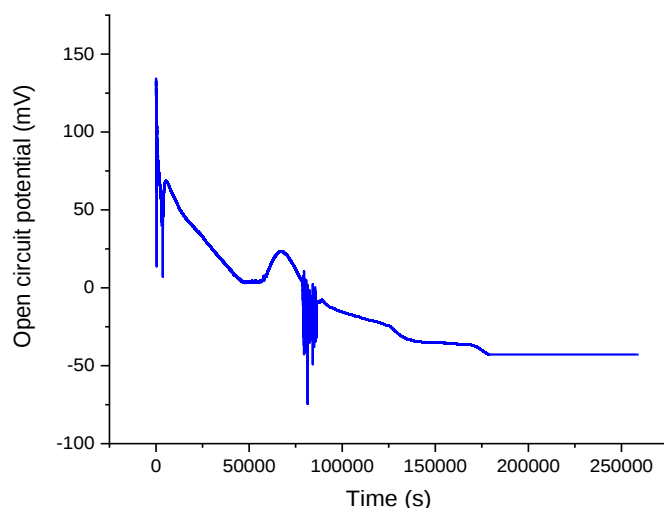


Figure 1. OCP curve of graphite material in peat environment.

LPR has provided remarkable information about the polarization resistance of graphite material in aqueous peat environment. Figure 2 shows the curve obtained from the LPR test of graphite material in peat. The slope of the curve in question was determined to be approximately 2.35365×10^7 . The LPR test provides information simply and quickly, showing that the polarization resistance of the graphite material in peat is $23.5365 \text{ M}\Omega/\text{cm}^2$. Since no numerical data on corrosion was found in the LPR test of graphite material in peat environment, the LPR finding in this study was compared with the LPR results of steel material in soil environment. This polarization resistance value is higher than the polarization resistance of carbon steel in the soil solution environment ($1301.7 \text{ }\Omega/\text{cm}^2$) [28]. Therefore, according to the LPR finding in this study, graphite material showed very high resistance to corrosion in the peat environment.

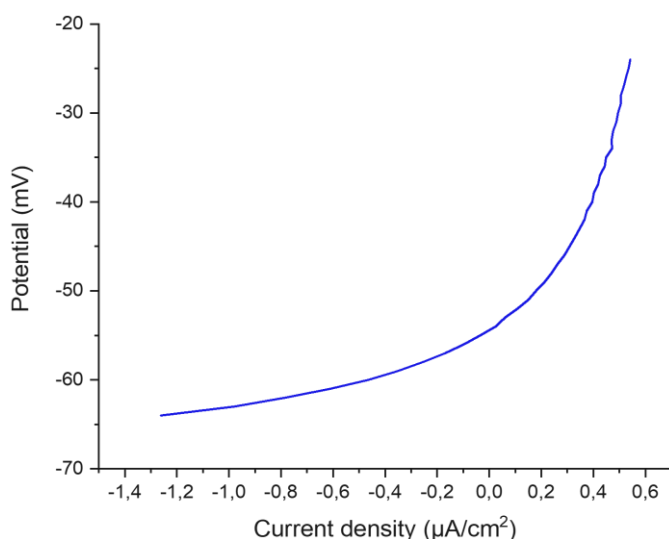


Figure 2. LPR curve of graphite material in peat environment.

Potentiodynamic testing provides information about oxidation or reduction reactions on the material surface. As can be seen from the potentiodynamic curves in Figure 3, the important findings are that the graphite material shows negative potential and that the negative current density of the graphite material increases when the potential value in the negative direction increases. These findings indicate that an oxidation reaction occurs in the graphite material and oxide formation. Therefore, the graphite electrode showed anodic properties in the peat environment. Electrochemical corrosion of graphite material in peat environment is thought to proceed by the agency of the carbon surface oxides formation. Additionally, at upward of anodic potential values, the current value increased quickly withal potential value. The resulting anodic oxidation is thought to be the amount of multiple stage such as the graphite electrode oxidation, graphite oxide's the decomposition into carbon dioxide (CO_2) and carbon monoxide (CO) and the oxygen emergence. All this information obtained is similar to the potentiodynamic test findings of graphite in dirty phosphoric acid environment [2,29].

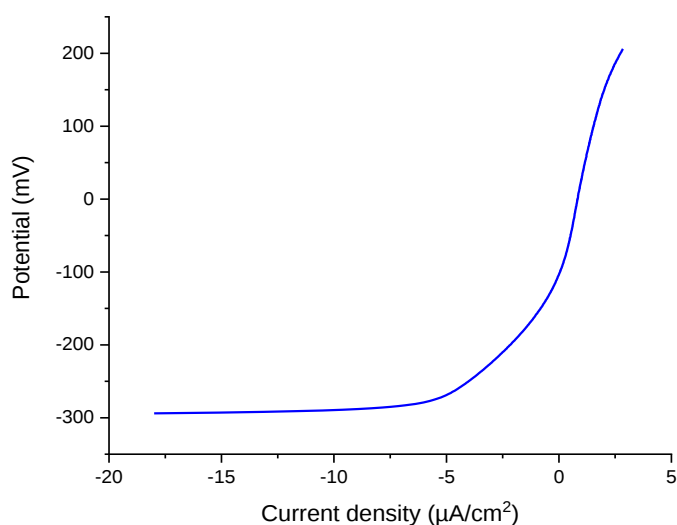


Figure 3. Potentiodynamic curve of graphite material in peat environment.

The cyclic polarization test provides information about the material's tendency to pitting corrosion. In Figure 4, it is obvious that the graphite material shows a remarkable level of hysteresis loop in the peat environment. This cycle shows that the graphite material has a tendency to pitting corrosion in a peat environment. Considering the necessary parameters to understand pitting corrosion, graphite material in peat environment could reach a reverse potential value of 1306 mV, a pitting potential of approximately 521 mV and a protection potential of approximately -0.054 mV was measured. Since the protection potential is more negative than the pitting potential, it is possible for pitting corrosion to occur here [30].

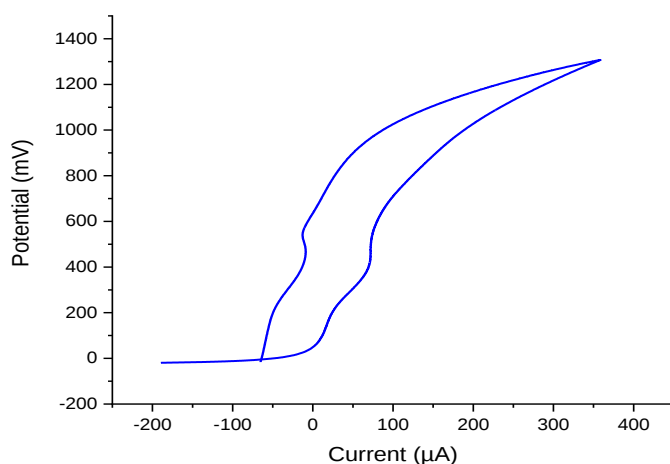


Figure 4. Cyclic polarization curve of graphite material in peat environment.

The potential and current curves obtained from the galvanic corrosion test of the graphite material in the peat are given in Figure 5. As can be seen here, changes have occurred in the galvanic potential and galvanic current values. Since the reaction kinetics at the graphite material-peat interface determines the galvanic corrosion potential, the higher (i.e. more positive potential) seen at the beginning showed that the graphite material has a more noble or more cathodic electrode potential in the peat environment. Towards the end of the measurement, the graphite material had a more anodic electrode potential in the peat environment and became prone to corrosion. Changes in galvanic current values may be due to oxygen diffusion around the graphite material in the peat environment [31].

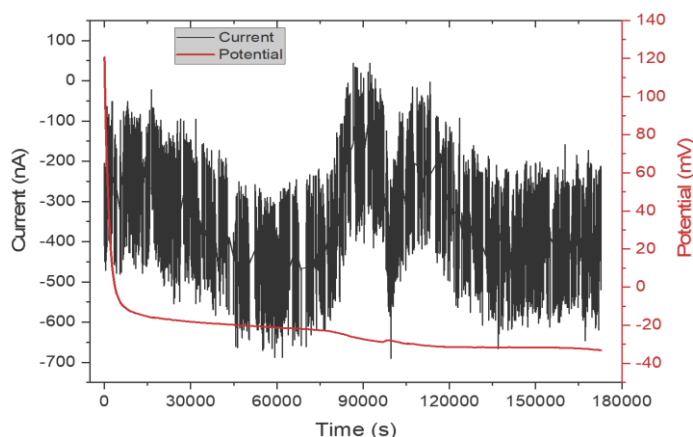


Figure 5. Galvanic corrosion of graphite material in peat environment.

The Nyquist diagram obtained from EIS findings is given in Figure 6. As can be seen here, the diagram generally consists of depressed semicircular and linear sections. The linear behaviour seen in the Nyquist diagram shows that diffusion processes occur in the graphite material in the peat environment [26]. Additionally, when looked at carefully, the semicircle appears to be a suppressed capacitive semicircle. It is known that the diameter of this semicircle is proportional to the load transfer resistance. Additionally, semicircular diameter has been associated with corrosion resistance and material dissolution during the corrosion process. In short, as the diameter of the semicircle increases, corrosion resistance increases. However, the linear line seen after this semicircle clearly reveals that corrosion processes of graphite material occur in the peat environment [32]. The equivalent circuit in Figure 7 provided by IVIUM software consists of R and C elements. R refers to the charge transfer resistance and C refers to the double-layer capacitance, which is essentially determined by the corrosion process. The equivalent circuit here is similar to the non-ideal behaviour of supercapacitors, generally attributed to the distributed electrical properties of porous materials [33]. It is thought that the graphite material turns into a porous form in the peat environment. This may have occurred due to pitting corrosion and is consistent with the cyclic polarization test.

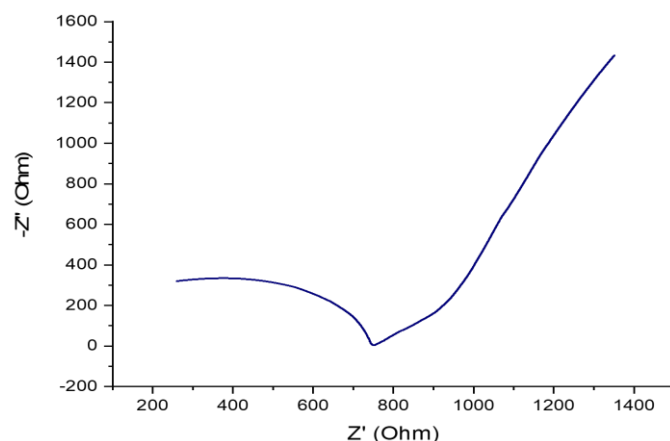


Figure 6. Nyquist diagram of graphite material in peat environment.

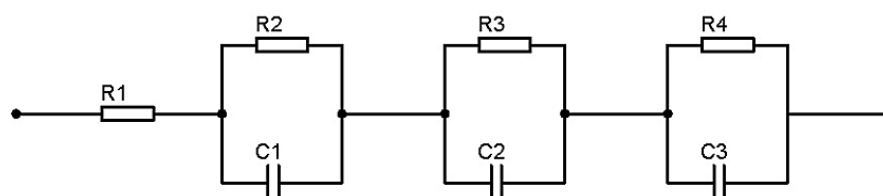


Figure 7. Equivalent circuit model of graphite material in peat environment.

The Tafel curve of graphite material in peat environment is shown in Figure 8 and some information obtained from this curve is given in Table 1-2. Considering Figure 8 and Table 1, the corrosion potential of graphite in the aqueous peat environment is positive and it can be said that the corrosion behaviour of graphite in the aqueous peat environment is noble. This indicates good corrosion resistance [34]. Corrosion current density is at very low levels. Compared to current practices, the corrosion current density value is well below the requirement limit ($<16 \times 10^{-6} \text{ A/cm}^2$) formulated by the United States Department of Energy (DOE) for proton exchange membrane fuel cells [16]. This indicates that the graphite material shows good corrosion resistance in the aqueous peat environment. Moreover, the corrosion rate of graphite in peat ratio falls into the very low corrosion category ($\leq 0.025 \text{ mm/year}$) according to the corrosion rate classification for equipment in the chemical industry [35]. The corrosion rate determined by extrapolation of Tafel sections indicates the high corrosion resistance of the graphite material in aqueous peat environment conditions. Despite this, corrosion is an inevitable natural process. This information can provide insight into the fabrication of various composite materials (e.g., metal matrix composites [36,37]), nanotechnology applications (e.g., artificial intelligence supported nanobots [38]) and open new research doors.

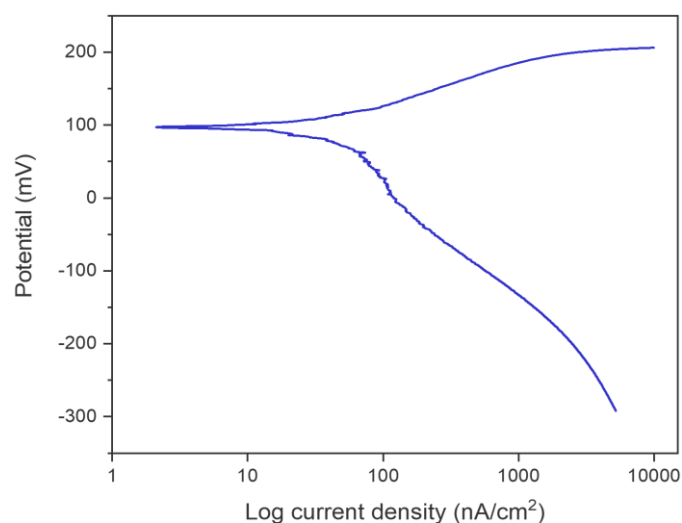


Figure 8. Tafel curves of graphite material in peat environment.

Table 1. Results from Tafel tests.

Parameter	Result
Corrosion potential (E_{corr})	88.9 mV
Corrosion current density (I_{corr})	25.72 nA/cm ²
Anodic Tafel slope (b_a)	-0.141 V/dec
Cathodic Tafel slope (b_c)	-0.062 V/dec
High anodic potential (E_{a1})	-33 mV
Low anodic potential (E_{a2})	-196 mV
High cathodic potential (E_{c1})	180 mV
Low cathodic potential (E_{c2})	134 mV
Corrosion rate (mm/year)	1.117×10^{-7}

Table 2. Comparison of corrosion behaviour of graphite material in different environments.

Environment	E _{corr}	I _{corr}	Corrosion rate	Ref.
3.5% NaCl solution	-253 mV	1644 nA/cm ²	-	[15].
0.1 mol.dm ⁻³ Na ₂ SO ₄ + 2 ppm F ⁻ (O ₂ saturated)	-20 to 112 mV	1510 to 3440 nA/cm ²	-	[16].
0.1 mol.dm ⁻³ Na ₂ SO ₄ + 2 ppm F ⁻ (H ₂ saturated)	25 to 192 mV	6180 to 8150 nA/cm ²	-	[16].
Peat (aqua)	88.9 mV	25.72 nA/cm ²	1.117×10 ⁻⁷ mm/year	This study

CONCLUSION

All test results expressing corrosion behaviour are consistent with each other. At the beginning of the experiments, the corrosion behaviour of the graphite material in the peat environment showed a more positive (noble) potential, but over time it shifted to a negative potential. This made it obvious that corrosion processes were occurring. An oxidation reaction occurred in the graphite material in the peat environment, triggering pitting corrosion. Moreover, the possible oxygen diffusion around the graphite material in the peat environment is thought to be a factor in the occurrence of corrosion processes. The diffusion processes noticeable in the Nyquist diagram and the fact that they are generally attributed to the dispersed electrical properties of porous materials, which are confirmed in the analysis in the equivalent circuit, suggest that the graphite material turns into a porous form in the peat environment. This may have occurred due to pitting corrosion and is consistent with other tests. It is understood that the corrosion occurring in graphite material in the peat environment is an inevitable natural process, and despite this, good corrosion resistance has been determined. The corrosion rate of graphite in the peat ratio falls into the very low corrosion category (≤ 0.025 mm/year) according to the corrosion rate classification for equipment in the chemical industry. All these findings show that graphite material is promising as a more sustainable corrosion-resistant electrode material for electrochemical devices such as fuel cells, microbial fuel cells and biosensors. Further advanced detail studies are needed on these issues separately. Also, the corrosion mechanism that occurs as anodic oxidation in the potentiodynamic tests of graphite, the decomposition of graphite oxide into CO₂ and CO, is an environmental problem that should be taken into consideration due to its potential to create a greenhouse effect. This is an indicator of the harmful potential of corrosion on the economy, health, ecosystem and living beings.

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