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Corrosion Behavior of Nickel-Titanium Alloy in Simulated Body Fluids

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ABSTRACT

Nitinol, a nickel–titanium alloy, is widely used as a biomaterial because of its strong biocompatibility, ease of processing, and stable mechanical performance. However, its interaction with body fluids such as sweat, saliva, and tissue fluid is critical, since even relatively mild biological environments can still lead to corrosion and metal-ion release. In this study, the corrosion behavior of Nitinol was investigated electrochemically at 37 °C to simulate human body temperature. A naturally formed passive oxide layer was detected on the alloy surface, and its stability varied depending on the solution. EIS measurements showed impedance values of 6.85×10^5 ohm.cm² in Fusayama Meyer artificial saliva, 8.35×10^5 ohm.cm² in PBS, and 3.82×10^6 ohm.cm² in Hank's solution. Potentiodynamic polarization results supported these findings. Overall, the passive film demonstrated the greatest stability and corrosion resistance in Hank's artificial body fluid, indicating Nitinol's strong suitability for long-term biomedical use.

Yapay Vücut Sıvılarında Nikel-Titanyum Alaşımının Korozyon Davranışının İncelenmesi

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ÖZ

Nikel titanyum alaşımı olan Nitinol metali; biyouyumlu yapısı, işlenebilirliği, dayanıklı mekanik özellikleri ile biyomalzeme olarak tercih edilmektedir. Biyomalzemelerin insan vücudundaki sıvılarla (ter, tükürük, doku sıvısı vb.) etkileşimi, bu etkileşim sırasında vücuda iyon salımı ile oluşabilecek toksik etkilerin araştırılması açısından önemlidir. Vücut sıvıları çok aşırı saldırgan olmamalarına rağmen doğrudan ya da dolaylı olarak metalle etkileşmeleri halinde metalin korozyona uğraması kaçınılmazdır. Bu çalışmada Nitinol metalinin farklı vücut sıvılarında korozyon davranışları vücut sıcaklığı olarak kabul edilen 37°C'de elektrokimyasal olarak incelenmiştir. Sonuç olarak; Nitinol yüzeyinde doğal olarak pasif oksit filmi oluşumu gözlenmiş ve kararlılığı farklı vücut sıvılarında değişiklik göstermiştir. EIS sonuçları Fusayama Meyer'in yapay tükürük çözeltisi, PBS çözeltisi ve Hank'ın yapay vücut sıvısı için sırasıyla, 6.85 x10^5 ohm.cm^2, 8.35 x10^5 ohm.cm^2, 3.82 x10^6 ohm.cm^2 dir. Potansiyodinamik polarizasyon eğrileri, EIS sonuçları ile uyumludur. Nitinol alaşımının yüzeyinde oluşan pasif oksit filmin Hank'ın yapay vücut sıvısı içerisinde daha kararlı yapıya sahip olduğu ve korozyona karşı daha dirençli olduğu belirlenmiştir.

1. INTRODUCTION

Metals naturally occur as compounds in their lowest-energy, most stable states, such as oxides, sulfides, carbonates, and phosphates, in accordance with thermodynamic laws. Energy must be expended to produce metals or alloys from these compounds. As the energy of metals increases, their disorder decreases. All metals tend to return to their most stable state in nature. Corrosion is the process by which metallic materials revert to their primitive, stable state in nature by interacting with their environment [1,2]. This process causes significant problems, especially in biomaterial applications. [3-8]. Biomaterials are natural or artificial materials used to perform or support the function of the tissue or organ into which they are implanted in the living body [9-13]. Biomaterials are expected to coexist with the tissue they directly contact without adverse interactions. This expectation ensures long-term quality of life for the organisms that use them [8,9]. The use of metallic biomaterials in various orthopedic applications, including plate and screw materials, joint prostheses, hearing aids, jaw and facial components, heart valves, stents, catheters, artificial heart parts, and dental implants, is crucial for examining the corrosion of these materials [7,9,17-20]. The rate of corrosion of metals varies depending on many factors such as environmental conditions, duration of use, movement of the metal, design of the system, and the presence of microorganisms [4,7,17,19,21,22]. According to these factors, corrosion is divided into different types [1,2,23,24]. General corrosion, which occurs evenly across the metal surface, is called pitting corrosion. Galvanic corrosion, which occurs when metals that are electrochemically different come into contact, is called galvanic corrosion. Crevice corrosion, which occurs when a metal touches a narrow gap, is caused by insufficient oxygen. Stress corrosion, which progresses by creating cracks under tensile stress, is called intergranular corrosion, which progresses along the grain boundaries of the metal. Metallic biomaterials are typically exposed to complex, oxygen-rich physiological environments that contain various inorganic ions (such as chloride, carbonate, calcium, sodium, and potassium), as well as biological components including proteins, lipids, amino acids, and cells that tend to accumulate around the metal surface [7-9,25]. Nitinol (50%Ni, 50%Ti) stands out among the preferred metallic biomaterials due to its biocompatible structure, good processability, strength and durability in environmental conditions [26-28]. Nitinol, which has an elastic modulus similar to that of bone, is widely used in orthodontics, bone fracture treatment, and as bone suture anchors to attach soft tissues such as tendons and ligaments to bone. Its shape memory properties also make it suitable for use in medical devices that can be delivered to the patient through minimally invasive methods, such as via a thin catheter [27-29]. Considering these properties, constant contact with living body fluids is inevitable. Body fluids act as the electrolyte medium necessary for the electrochemical corrosion of Nitinol, and its contact with body fluids demonstrates the inevitability of corrosion. Titanium is known to have excellent corrosion resistance [3,30,31]. However, Nitinol tends to be coated with a TiO_2-based oxide containing nickel, which is both oxidized and metallic. It is useful to examine the relationship between the composition of the Nitinol surface oxide and its corrosion resistance.

In literature studies, corrosion studies were conducted on Nitinol, which has a porous surface, in a saltwater environment at 37°C, and it was determined that Nitinol exhibited pitting corrosion properties. Mott-Schottky analysis determined that the passive film formed on the surface exhibited n-type semiconductor properties. [26]. In another study, the corrosion behavior of Nitinol obtained from nickel and titanium elements by spraying method with bulk Nitinol was investigated in Hanks and Ringer solutions at 37°C and it was determined that Nitinol obtained in thin film form was more resistant to corrosion. [32]. In another study, the corrosion resistance of Nitinol was studied in an artificial body fluid that was not in contact with air and it was determined that the oxide film had a protective effect on the surface at high loads. [33].

In this study, electrochemical methods were used to determine the durability and corrosion behavior of Nitinol, a biocompatible, easily processable biomaterial preferred by many in environments such as Hank's simulated body fluid, phosphate-buffered saline, and Fusayama Meyer's simulated saliva solution[32-35] at 37°C (body temperature) [32,33]. For this purpose, EIS measurements and potentiodynamic polarization curves were taken. SEM (Scanning Electron Microscope) and EDS (Energy Dispersive Spectroscopy) techniques were used to examine surface morphology.

2. MATERIAL AND METHOD

The electrochemical measurements required for corrosion behavior were carried out using the three-electrode method in a single-compartment cell set up in a water bath (37°C). Nitinol was used as the working electrode, platinum (Pt) with a surface area of 2 cm^2 was used as the counter electrode and Ag/AgCl (3 M KCl) electrode was used as the reference electrode. Prior to each measurement, the surface of the Nitinol samples was mechanically polished using silicon carbide (SiC) abrasive papers with grit sizes of 600, 1000, and 1200, followed by ultrasonic cleaning for 5 minutes in ethanol and 5 minutes in deionized water. Test solutions are Hank's artificial body fluid (NaCI 8.00 g/L; KCI 0.40 g/L; $CaCl_2$ 0.18 g/L; $NaHCO_3$ 0.35 g/L; $Na_2HPO_4.2H_2O$ 0.48 g/L; $MgCl_2.6H_20$ 0.10 g/L; KH_2PO_4 0.06 g/L; $MgS0_4.7H_2O$ 0.10 g/L; Glucose 1.00 g/L) [34] Phosphate buffered saline (PBS solution) (NaCI 8.00 g/L; KCI 0.2 g/L; $Na_2HPO_4.2H_2O$ 1.42 g/L; KH_2PO_4 0.24 g/L) [37] and Fusayama Meyer's artificial saliva solution (KCl 0.40 g/L; NaCl 0.40 g/L; $CaCl_2.2H_2O$ 0.91 g/L; $Na_2HPO_4.2H_2O$ 0.69 g/L; $Na_2S.9H_2O$; 0.0005 g/L; Urea; 1.00 g/L) [38] was used. Measurements were made in triplicate. Electrochemical measurements were made using a CHI 660D instrument. EIS measurements were taken at an open-circuit potential with an amplitude of 5 mV in the frequency range of 10^5-10^{-3} Hz. Potentiodynamic polarization curves were obtained in the range of -1 to +1 V at a scan rate of 1 mV/s. The results were determined using the Zwiev program. Surface analyses of Nitinol were performed using a SEM-EDS (SEM/EDS FEI Quanta 650 Field Emission) instrument to investigate its structural properties.

3. RESULTS AND DISCUSSIONS

3.1. Corrosion Potential-Time Curves

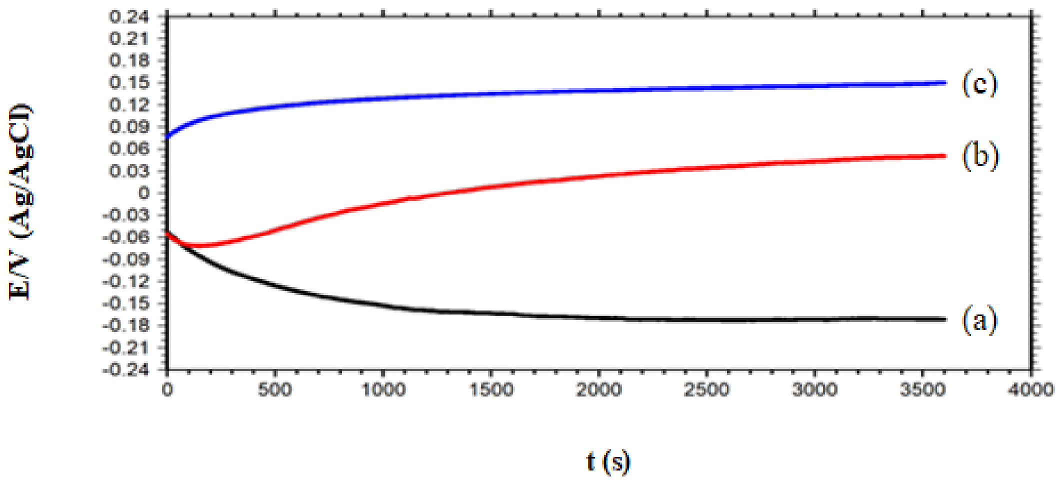

Figure 1. Corrosion potential with time for Nitinol alloy in different body fluids: Hank's artificial body fluid solution (Black (a)), PBS solution (Red (b)), Fusayama Meyer's artificial saliva solution (Blue (c))

Nitinol was compared in various body fluids by monitoring its open-circuit potential over time, which indicates its electrochemical stability. Nitinol has a natural and relatively stable oxide layer on its surface [39-41]. It was observed that this oxide layer thickens or degrades over time when Nitinol comes into contact with various body fluids. Corrosion potential indicates that the oxidation reaction within the metal's electrochemical cell is occurring, making the metal more easily oxidized. The shift of the corrosion potential to positive values indicates that the passive oxide film formed on the surface is more stable and more resistant to corrosion [42, 43]. Accordingly, it can be interpreted that the passive oxide film on the surface of Hank's artificial body fluid solution, which has a negative corrosion potential, is more unstable than PBS and Fusayama Meyer's artificial saliva solution.

3.2. Electrochemical Impedance Spectroscopy Measurements

Electrochemical impedance spectroscopy (EIS), a non-destructive and complementary technique based on alternating current (AC), is widely used to determine the corrosion rate of metal surfaces. Since the applied AC signal does not significantly alter the surface structure, this method is considered to yield more accurate and reliable information regarding the corrosion resistance and interfacial properties of the metal. In EIS, the polarization resistance representing the resistive components at the electrode electrolyte interface is determined using an equivalent electrical circuit model (Figure 2. (1)) [44-46].

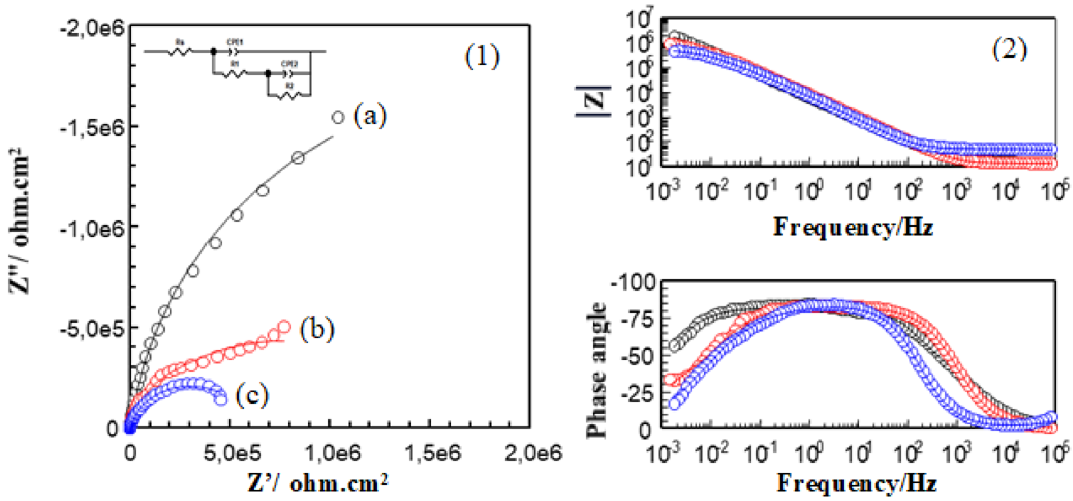

Figure 2. EIS results for Nitinol alloy in different body fluids; Nyquist Curves (1) Bode Curves (2); Hank's artificial body fluid solution (Black (a)), PBS solution (Red (b)), Fusayama Meyer's artificial saliva solution (Blue (c))

The Nyquist and Bode plots of Nitinol in different simulated body fluids were obtained using the Zwiew program based on the equivalent circuit shown in Figure 2. In this equivalent circuit, the capacitive behavior is modeled using a constant phase element (CPE); Rs represents the solution resistance; R1 corresponds to the charge transfer resistance; and R2 represents the resistance of the passive film presumed to form on the surface, which contributes to ion accumulation and provides physical protection to the metal, even in the absence of a fully developed passive layer. Nyquist curves, which are compatible with Bode curves, show a single loop that starts in the high frequency region, continues in the mid-frequency region, and closes in the low frequency region [44-46]. The resistance values listed in Table 1 for Hank's solution, PBS, and Fusayama-Meyer artificial saliva suggest that increasing total resistance correlates with enhanced TiO_2 formation on the Nitinol surface due to interaction with dissolved oxygen, thereby improving corrosion resistance.

Table 1. Parameters found from EIS measurements of Nitinol alloy in different body fluids.

Body fluids	CPE1 ($S\ s^n\ cm^2$)	n1	Rs (ohm cm^2)	CPE2 ($S\ s^n\ cm^2$)	n2	R2 (ohm cm^2)
Hank Sol.	$1.55x10^{-5}$	0.91	60.47	$1.31x10^{-5}$	0.91	$3.82\ x10^6$
PBS Sol.	$2.29x10^{-6}$	0.92	$5.64\ x10^5$	$3.58x10^{-5}$	0.72	$8.35\ x10^5$
Fusayama Meyer Sol.	$5.82x10^{-6}$	0.50	41.69	2.3110^{-5}	0.95	$6.85\ x10^5$

3.3. Potentiodynamic Polarization Measurements

Figure 3 shows the potentiodynamic polarization curves of Nitinol obtained in different body fluids. It is observed that there is no significant difference in the potentiodynamic polarization curves obtained from body fluids. The current is constant between -0.4 and 0.2 V in the cathodic direction. Oxygen in different body fluids comes to the surface from solutions by diffusion. Therefore, it is limited by mass transfer, and a significant oxygen reduction current limit is observed in the -0.4 to -0.2 V range. Reduction is observed between -0.4 and -1 V, where oxidation occurs. In the anodic region, a passivation zone is observed with a value close to the oxygen limit current. It can be concluded that the reaction occurring here is between the oxygen in body fluids and the Nitinol surface, causing spontaneous TiO_2 formation. More positive zero current potentials reflect higher corrosion protection [47,48]. According to the curves obtained in Hank's simulated body fluid, PBS, and Fusayama Meyer's simulated saliva solution, the current zero potentials are +0.47 V, +0.54 V, and +0.62 V, respectively. These potential values approaching positive values also show that Hank's artificial body fluid, PBS and Fusayama Meyer's artificial saliva solution are more resistant to corrosion, respectively.

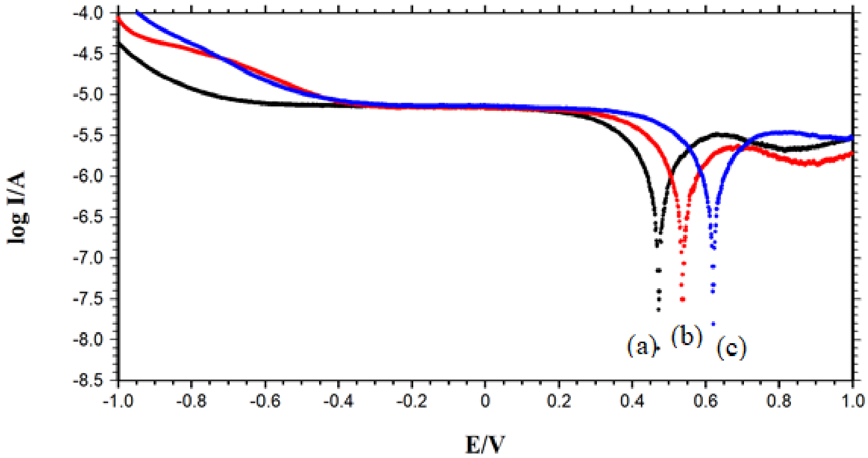

Figure 3. Potentiodynamic polarization curves for Nitinol alloy in different body fluids; Hank's artificial body fluid solution (Black (a)), PBS solution (Red (b)), Fusayama Meyer's artificial saliva solution (Blue (c))

3.4. SEM-EDS Results

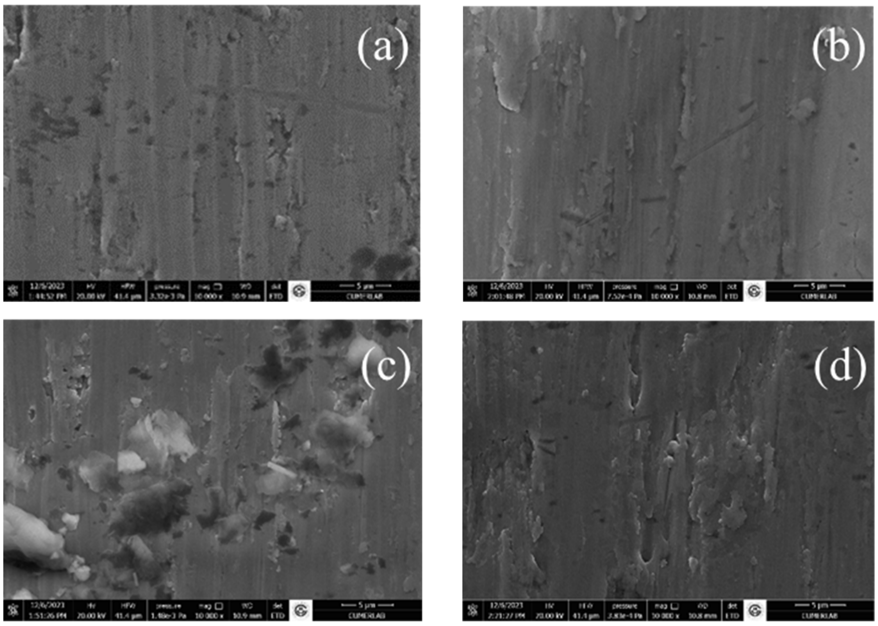

Figure 4. SEM images of Nitinol kept in different body fluids for 120 hours; Nitinol (a) Hank's artificial body fluid solution (b) PBS solution (c) Fusayama Meyer's artificial saliva solution (d)

Figure 5 presents SEM images of Nitinol surfaces after immersion in different simulated body fluids for 120 hours, revealing notable differences in surface morphology depending on the environment. The untreated Nitinol surface (Figure 5a) exhibited a smooth and homogeneous structure, with well-preserved linear polishing marks and no evidence of pitting, cracking, or particle deposition. After exposure to Hank's simulated body fluid (Figure 5b), the surface remained largely intact, showing only minor particle deposits and localized surface irregularities. These observations suggest that the mildly corrosive nature of Hank's solution led to limited interaction with the passive layer while preserving overall surface integrity. In contrast, the surface of Nitinol immersed in PBS (Figure 5c) showed severe degradation, including dense particle accumulation and amorphous precipitates. The complete disappearance of surface markings, indications of pitting corrosion, and disruption of the passive film imply that PBS created a highly corrosive environment, potentially leading to leaching of titanium and nickel from the alloy surface. Nitinol exposed to Fusayama-Meyer artificial saliva (Figure 5d) exhibited moderate corrosion, characterized by surface roughening, partial loss of linear features, and scattered precipitate formation. This suggests partial degradation of the passive layer and a moderately aggressive interaction with the solution [41].

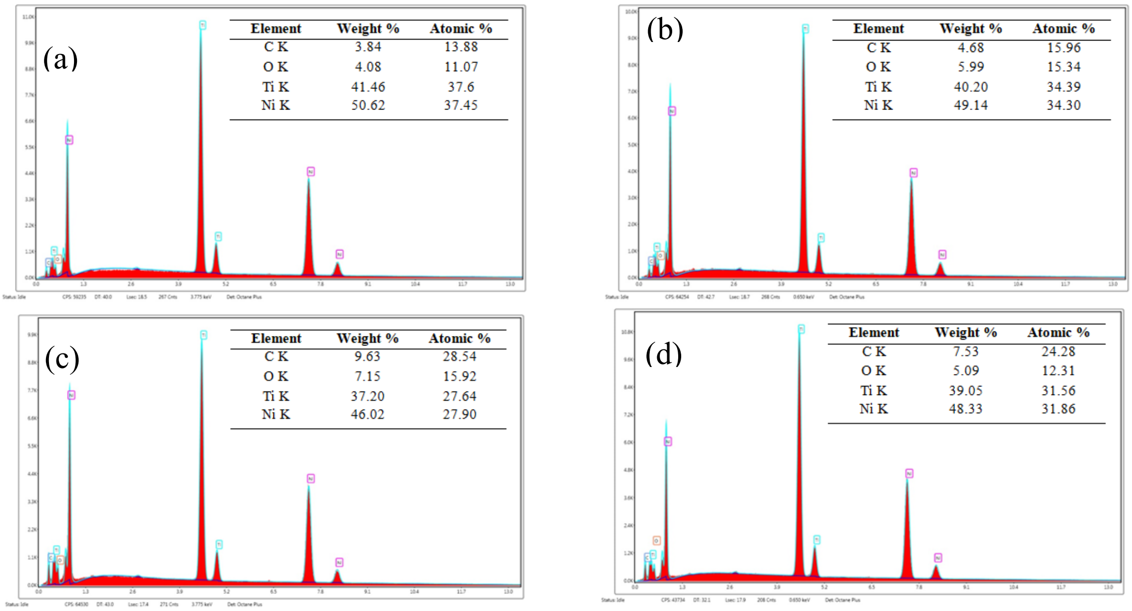

Figure 5. EDS result of Nitinol kept in different body fluids for 120 hours; Nitinol (a) Hank's artificial body fluid solution (b) PBS solution (c) Fusayama Meyer's artificial saliva solution (d)

In the EDS analyses given in Figure 5, parallel to the SEM images, the decrease in Ti and Ni percentages was clearly seen especially in the PBS solution, while the increase in O and C percentages supports the oxidation and organic/inorganic precipitation processes on the surface.

4. CONCLUSIONS

The corrosion behavior of Nitinol, used as a metallic biomaterial in various body parts, was electrochemically investigated in various body fluids and supported by surface analyses. As a result of this study, it was determined that:

- ✓ Titanium, one of the elements constituting Nitinol, is reduced by oxygen in body fluids, forming a passive film on the surface.
- ✓ Corrosion resistance was determined to increase by 685011 ohm cm^2 , 1399490 ohm cm^2 ve 3817460 ohm cm^2 in Fusayama Meyer's artificial saliva solution, PBS solution, and Hank's artificial saliva solution, respectively. These data indicate that the increased resistance of the passive film formed on the surface increases, and the lower emissivity indicates a higher polarization resistance.
- ✓ Potentiodynamic polarization curves show zero-current potentials of +0.47 V, +0.54 V, and +0.62 V in Hank's artificial saliva, PBS, and Fusayama Meyer's artificial saliva solutions, respectively. These potential values, approaching positive values, indicate that Nitinol exposed to Hank's simulated body fluid is more resistant to corrosion.

SEM-EDS results show that the passive film formed on the Nitinol surface by various body fluids significantly protects the metal, but over time, it breaks down and pitting corrosion begins.

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