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EFFECTS OF CARBOXYLIC ACID ADDITIVE ON HARD ANODIZING OF AL7075 ALUMINUM ALLOY

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Keywords

Hard Anodizing,
Carboxylic Acid,
Al7075,
Vickers Microhardness.

Abstract

The effect of adding organic acid on the hard-anodizing coating on AA7075 aluminum alloy was investigated. Five different acid mixtures as electrolytes (Sulfuric acid (SA) + Acetic acid (AA), Sulfuric acid (SA) + Pyruvic acid (PA), Sulfuric acid (SA) + Malonic acid (MA), Sulfuric acid (SA) + Tartaric acid (TA), Sulfuric acid (SA) + Citric acid (CA)) were used as electrolysis bath. Scanning electron microscopy (SEM) analysis, EDS and elementary distribution analyzes were performed on the material surfaces after anodic oxidation, and Fourier Transform Infrared Spectroscopy (FTIR) analyzes were performed to determine the functional groups and molecular structures formed on the surfaces. As a result of the application, the highest coating thickness value was measured as 129.5 µm for tartaric acid. Fourier Transform Infrared Spectroscopy (FTIR) analysis generally shows that amorphous alumina contains Al-O bonds, Al-O-Al bonds and Al-OH bonds. The highest hardness value of 420 HV was obtained as a result of anodic oxidation by adding tartaric acid into a sulfuric acid bath

KARBOKSİLİK ASİT KATKISININ AL7075 ALÜMİNYUM ALAŞIMININ SERT ELOKSALLANMASINA ETKİLERİ

Anahtar Kelimeler

Sert Eloksal Kaplama,
Karboksilik Asit,
Al7075,
Vickers Mikrosertlik.

Öz

Al7075 alüminyum alaşımının sert eloksal kaplamasına organik asit ilavesinin etkisi araştırılmıştır. Elektrolit olarak beş farklı asit karışımı (Sülfürik asit (SA) + Asetik asit (AA), Sülfürik asit (SA) + Pirüvik asit (PA), Sülfürik asit (SA) + Malonik asit (MA), Sülfürik asit (SA) + Tartarik asit (TA), Sülfürik asit (SA) + Sitrik asit (CA)) elektroliz banyosu olarak kullanıldı. Malzeme yüzeylerinde anodik oksidasyon sonrası taramalı elektron mikroskobu (SEM) analizi, EDS ve elementer dağılım analizleri, yüzeylerde oluşan fonksiyonel grupların ve moleküler yapıların belirlenmesi amacıyla Fourier Dönüşümü Kızılötesi Spektroskopi (FTIR) analizleri yapıldı. Uygulama sonucunda en yüksek kaplama kalınlığı değeri tartarik asit için 129,5 µm olarak ölçülmüştür. Fourier Dönüşümü Kızılötesi Spektroskopisi (FTIR) analizi genel olarak amorf alüminanın Al-O bağları, Al-O-Al bağları ve Al-OH bağları içerdiğini göstermiştir. En yüksek sertlik değeri 420 HV ile Sülfürik asit (SA) + Tartarik asit (TA) elektroliz banyosu ile elde edilmiştir.

Alıntı / Cite

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Highlights

- Investigation of organic acid contribution in hard anodizing coating of Al7075 aluminum alloy.
- Examination of surface properties by SEM, EDS and FTIR analyses.
- It was found that the best coating thickness and highest hardness values were obtained with the addition of tartaric acid.

Graphical Abstract

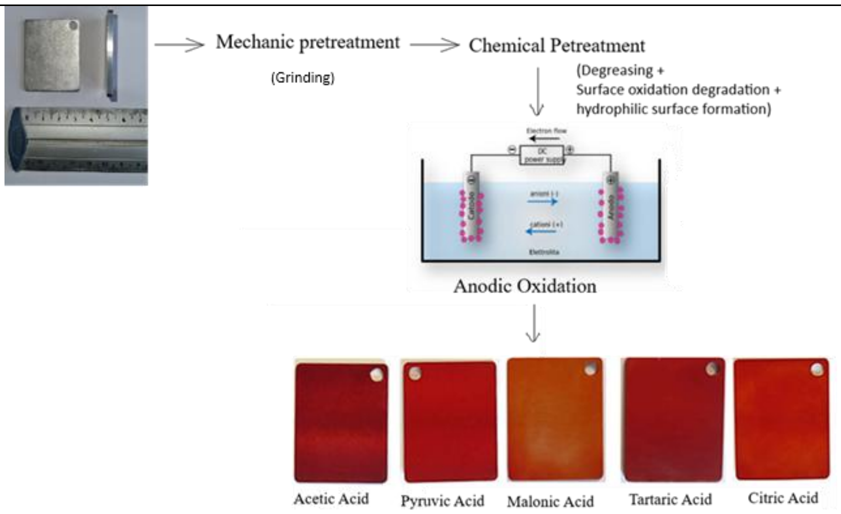

Figure. Effects of organic acid additive to the electrolyte bath

Purpose and Scope
In this study, it is aimed to obtain a high strength, porous and appropriately thick anodic oxide layer on the Al7075 aluminum alloy, which is preferred in many different areas such as food, chemical substance storage containers, heat exchangers, fuel tanks, furniture parts, especially in the space and aviation sector, by adding organic acid to the anodic oxidation process.

Design/methodology/approach
Five different acid mixtures as electrolytes (Sulfuric acid (SA) + Acetic acid (AA), Sulfuric acid (SA) + Pyruvic acid (PA), Sulfuric acid (SA) + Malonic acid (MA), Sulfuric acid (SA) + Tartaric acid (TA), Sulfuric acid (SA) + Citric acid (CA)) were used as electrolysis bath. Scanning electron microscopy (SEM) analysis, EDS and elementary distribution analyzes were performed on the material surfaces after anodic oxidation, and Fourier Transform Infrared Spectroscopy (FTIR) analyzes were performed to determine the functional groups and molecular structures formed on the surfaces. The average hardness value was measured by Vickers microhardness experimental tests.

Findings
The highest coating thickness of 129.5 µm was obtained by adding tartaric acid into the acid bath. As a result of the EDS analysis, it was determined that Al and O were the dominant elements in the structure and formed the dominant alumina-based structure. It has been determined that in addition to Al and O elements, S and C elements are also present in the structure and the ratios of these elements vary according to organic acid types. As a result of the hardness analysis, the highest hardness value of 420 HV was obtained as a result of anodic oxidation by adding tartaric acid into a sulfuric acid bath.

Originality
With this study, it has been seen that the hard-anodized coating on aluminum can be improved by adding tartaric acid to the electrolysis bath and thus a material with high mechanical properties can be obtained. It is thought that materials with different mechanical properties can be obtained by processes with different anodic oxidation process parameters, and these materials can be used in different engineering applications.

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1. Introduction

Aluminum and its alloys are used in a wide variety of areas, especially in the space and aviation industry, as well as in food, chemical storage containers, heat exchangers, fuel tanks and furniture parts. The high wear and corrosion resistance of aluminum materials is due to the naturally occurring oxide layer on it. There are techniques to increase corrosion resistance and strength by thickening this oxide layer formed on aluminum. Among these techniques, the most easily applicable, cheap and effective method is the anodic oxidation method. Anodic oxidation (anodizing) process is based on the classical electrolysis cell principle. The aluminum material to be coated forms the anode of this cell. An oxide layer forms on the anode surface due to the chemical reaction between the anode and cathode. Parameters such as the type and concentration of the electrolyte used during electrochemical coating, the temperature of the electrolyte in the bath, the voltage and current density of the anodizing bath, and the application time affect the coating thickness and structure. The type of electrolyte used is usually sulfuric acid solution (Ono, 2003; Mansfeld, 1998). With the additives added to this solution, the thickness, stability and hardness of the aluminum oxide layer that will form on the surface are increased.

Additives are usually organic acid solutions. Most organic acids dissociate poorly and form barrier layers when used as a pure anodizing bath for aluminum. The strongest organic acid belonging to this group is oxalic acid (Keller, 1953). Malonic acid also belongs to this class (Lee, 2007; Machado, 2020). Some other organic acids are unstable. However, they can form porous layers in malic (Kikuchi, 2013; Machado, 2020), citric (Ono, 2004; Cabral, 2021; Machado, 2020), phosphoric (Jagminas, 2007; Kushwaha, 2014), chromic (Machado, 2020; Akyıldız, 2021) and tartaric acid electrolytes (Surganov, 2004; Machado, 2020).

In addition to the addition of organic acid solutions, hard anodizing is the most popular application of recent times to improve the wear properties of aluminum alloys. Coating the surfaces of aluminum alloys with hard anodizing increases the hardness of the alloy and increases its wear resistance at a significant point. The coating obtained by the hard-anodizing process is harder and thicker than the coating formed as a result of the anodizing process (Goksahin, 2007). The hard-anodizing process is a process that occurs by applying high concentrations of sulfuric acid at low temperature, high voltage and current densities. As a result of the hard-anodizing process, dense, hard, wear- and corrosion-resistant oxide layers are obtained on the aluminum surface. To achieve high current density at low temperatures, the applied voltage helps create a small-sized and dense pore structure. Thus, it provides high hardness and high wear resistance (Yavuz, 2018). Research has generally been carried out on the properties of oxide films formed on pure Al.

In this study, hard anodizing was applied to Al7075 aluminum alloy, which is frequently used especially in the aviation industry, using five different organic acid solutions added to the electrolyte. Scanning electron microscopy (SEM/EDS) analyzes and Fourier Transform Infrared Spectroscopy (FTIR) analyzes were performed on the material surfaces after anodic oxidation. Hardness tests were applied to examine the mechanical properties of the coated samples.

2. Material and Method

2.1. Material

Samples with dimensions of 3.5 x 4.5 x 0.3 cm were prepared by precision cutting from Al7075-T6 aluminum alloy billet (Figure 1). The chemical composition of the Al7075-T6 aluminum alloy sample is given in Table 1.

Figure 1. Al7075-T6 aluminum alloy sample used in experiments

Table 1. Al7075-T6 Alloy chemical composition (wt. %)

Al	Zn	Mg	Mn	Si	Fe	Cu	Cr	Ti	Others
91,4	6,1	2.9	0,3	0,4	0,5	1,2	0,18	0,2	0,15

Before anodic oxidation, each sample surface was mechanically pretreated with 100-120-180-220 mesh paper sandpaper. After the ground sample surfaces were degreased with ethyl alcohol, they were subjected to surface oxidation degradation in 5% Sodium Hydroxide (NaOH) solution for 3-4 minutes, then washed with pure water and immersed in 20% Nitric Acid (HNO_3) solution. After chemical treatments, surface water retention was checked by washing with pure water and it was observed that the surface was hydrophilic.

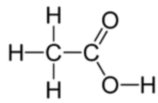
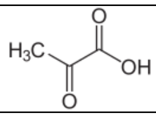
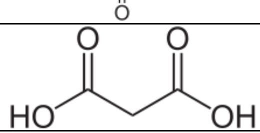
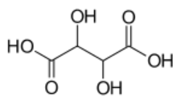
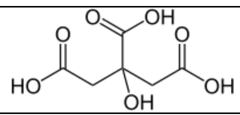
2.2. Organic Acids

Different organic acids (acetic acid, pyruvic acid, malonic acid, tartaric acid, citric acid) prepared at 0.5 M concentration were added into the 18% H_2SO_4 electrolysis bath. The organic acids used during the experiments are from the group of carboxylic acids and have the formula -C(=O)OH, which is also generally written as -COOH or CO_2H. Carboxylic acids are Bronsted acids, that is, they are proton donors. Carboxylic acids are typically weak acids, meaning they only partially dissociate into the H^+ cation and $RCOO^-$ anions. Chemical properties of the organic acids used in the experiments are given in Table 2.

2.3. Experimental Procedure

The samples, which were mechanically and chemically pretreated before anodic oxidation, were subjected to hard anodizing coating under low anodization temperature, low electrolyte and high current conditions. Experimental parameters were decided as a result of literature studies and preliminary experiments. 5 different baths were prepared by adding different organic acids (acetic acid, pyruvic acid, malonic acid, tartaric acid, citric acid) to the electrolyte containing 18% H_2SO_4.The hard-anodizing coating temperature was kept between -5/5° C, and the anodic oxidation process was applied for 120 minutes at 22V voltage value and 4 A/dm^2 current density. The applied voltage and current density during the experiments were provided by the DC power supply (UNI-T). The Al7075-T6 aluminum plate to be coated forms the anode of the classical electrolysis cell, and the lead plate used as the counter electrode constitutes the cathode. The chemical reaction between the anode and cathode causes the formation of an oxide layer on the anode surface. After anodic oxidation, the samples were colored using 5% dye solution. It is available in literature studies that the dye used must be at a temperature of 50-55°C in order for the coloring process to be of good quality. The processing time was determined as 15-20 minutes in order for the organic-based fabric dye to settle into the porous structure. In order to close the pores formed after the anodic oxidation and coloring processes, hot fixation was applied by keeping it in 100°C deionized water for 30-40 minutes. Thus, it is ensured that the anodizing layer is resistant to chemical and physical effects, color pigments do not bleed out and impurities do not enter the pores.

Table 2. Chemical properties of organic acids

Name	Class	Chemical Molecule Formula	Chemical Structure Formula	Acidity (pKa)
Acetic Asid	Monocarboxylic, C_2	$C_2H_4O_2$		4.76
Pyruvic Acid	Monocarboxylic, C_3	$C_3H_4O_3$		2.45
Malonic Acid	Dicarboxcylic, C_3	$C_3H_4O_4$		2.92
Tartaric Acid	Dicarboxcylic, C_4	$C_4H_6O_6$		2.93
Citric Acid	Tricarboxcylic, C_6	$C_6H_8O_7$		2.93

2.4. Microstructural Characterization

To examine the effect of organic acid contribution on the anodic oxidation process on Al7075-T6 aluminum alloy, the samples were examined using the NIKON ECLIPS LV150 brand optical microscope located in the Metallurgical and Materials Engineering Department of Atatürk University Faculty of Engineering. The measurement of the thickness of the coatings obtained after the anodic oxidation process applied by adding different organic acids into the electrolyte bath was examined using a scanning electron microscope (SEM). A section was taken from each sample, and after metallographic processing, the thickness measurement was carried out under 350X magnification conditions, from 3 different regions, and the coating thickness was determined by taking the average of the obtained data. ZEISS SIGMA 300 brand scanning electron microscope located at Atatürk University Eastern Anatolia High Technology Research Center (DAYTAM) was used for analysis. To determine the functional groups in the structure of the organic compounds, the state of the bonds in the structure, the binding sites and whether the structure is aromatic or aliphatic, the cross-sectional samples were examined using Fourier Transform Infrared Spectroscopy (FTIR). BRUKER VERTEX 70v brand Fourier Transform Infrared Spectroscopy (FTIR) located at Atatürk University Eastern Anatolia High Technology Research Center (DAYTAM) was used for analysis. Scans were performed at room temperature with a resolution of 0.4 cm^{-1} in the wavelength range of 4000 cm^{-1}–500 cm^{-1}. Spectra obtained from the FT-IR device were examined using the ORIGIN6 software program.

2.5. Mechanical Testing

Mechanical tests were performed using Wolpert Wilson-400 micro-hardness testing equipment comprising a Vickers indenter. The load applied on the specimens was about 0.01 kgf. Each microhardness value reported in this study is obtained by taking the mean of at least five individual readings.

3. Experimental Results

As a result of the examinations made on the samples after anodic oxidation, it was observed that different organic acids added to the 18% H_2SO_4 electrolyte bath caused different effects on the hard-anodized coating formed on the aluminum plate, depending on their chemical properties. Especially the color differences that occur after the coloring process can give a preliminary idea about the properties of hard anodized coating. The noticeable difference in tone on the color is an indicator of the extent to which the color pigments of the paint used have penetrated into the porous structure of the hard coating formed on the aluminum plate, thus giving a preliminary idea about the coating thickness obtained. Figure 2 shows the color tone differences of the coating formed on the aluminum plate by different organic acids added to the electrolyte bath.

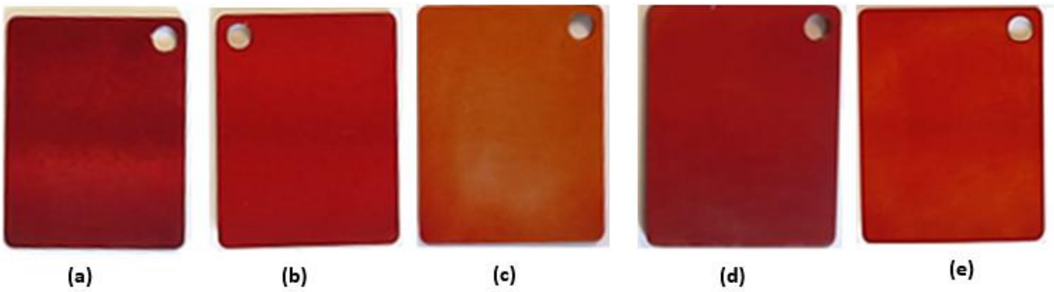


Figure 2. Effects of organic acid additive to the electrolyte bath ((a)Acetic acid, (b) Pyruvic acid, (c)Malonic acid (d) Tartaric acid, (e)Citric acid)

3.1. Microstructure

3.1.1. Optical microscope

Optical microscope images of hard anodized samples are given in Figure 3. When the optical microscope images are examined, grinding marks formed due to preliminary mechanical processes on the lower Al7075-T6 substrate layer are clearly visible. In the images, from bottom to top, the main material is in the form of an oxide layer. It was observed that the oxide layer formed on the substrate had an irregular structure and its thickness was not constant on the surface.

It was observed that the oxide layer formed on the substrate developed in a distinct, clear but irregular structure with the addition of organic acid. When the images were examined, it was seen that the irregularity in the structure increased with the addition of acetic ($C_2H_4O_2$), pyruvic ($C_3H_4O_3$), and malonic acid ($C_3H_4O_4$), and was relatively less with the contribution of tartaric ($C_4H_6O_6$), and citric acid ($C_6H_8O_7$).

3.1.2. SEM (Scanning Electron Microscopy) analysis

The thickness of the hard-anodized coating (total thickness of both the barrier oxide layer and the porous oxide layer) was determined by examining the sample under a scanning electron microscope (SEM). SEM images and EDS analyze of the samples hard anodized for 120 minutes at -5/5°C temperatures, 22V voltage and 4 A/dm^2 current density are shown in Figure 4. The coating thickness obtained without organic acid additive was measured as 57.78 µm. The lowest value was obtained with the addition of malonic acid at 27.12 µm, and the coating thicknesses of 70.82 µm, 77.52 µm and 79.11 µm were measured for adding pyruvic, citric and acetic acid into the acid bath, respectively. The highest coating thickness of 129.5 µm was obtained by adding tartaric acid into the acid bath. As a result of EDS analysis, the presence of aluminum (Al) and oxygen (O) elements in the coating indicates the oxide layer formed on the surface. Apart from oxygen and aluminum elements, the distribution of sulfur (S) and carbon (C) elements was also analyzed. It is thought that the carbon element comes from the organic acid content and the sulfur element comes from the electrolyte.

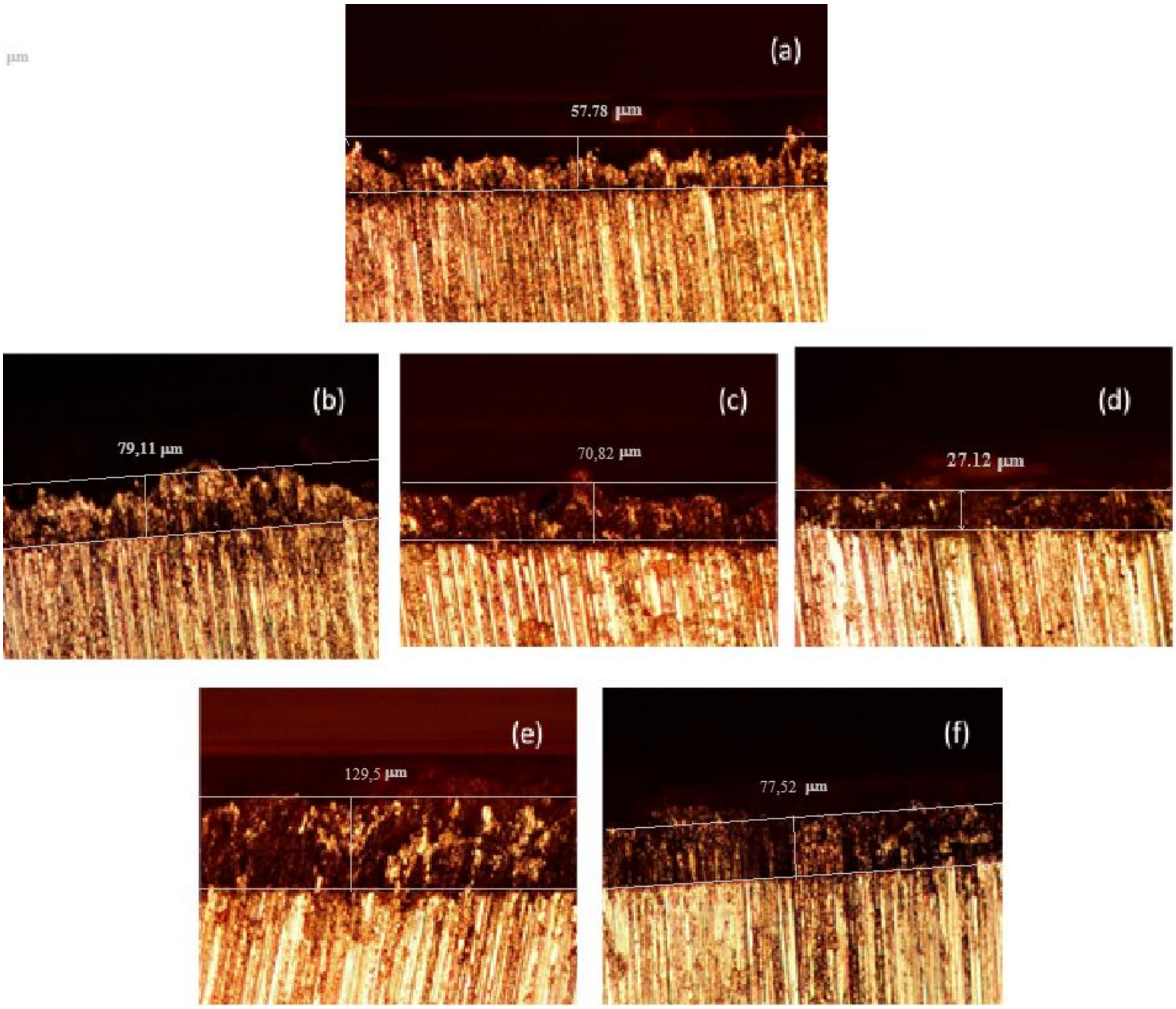


Figure 3. Optical microscope images after anodizing coating ((a): 18 % H_2SO_4, (b): 18 % H_2SO_4 + 0.5 M $C_2H_4O_2$(acetic acid), (c): 18 % H_2SO_4 + 0.5 M $C_3H_4O_3$ (pyruvic acid), (d): 18 % H_2SO_4 + 0.5 M $C_3H_4O_4$ (malonic acid), (e): 18 % H_2SO_4 + 0.5 M $C_4H_6O_6$ (tartaric acid), (f): 18 % H_2SO_4 + 0.5 M $C_6H_8O_7$(citric acid))

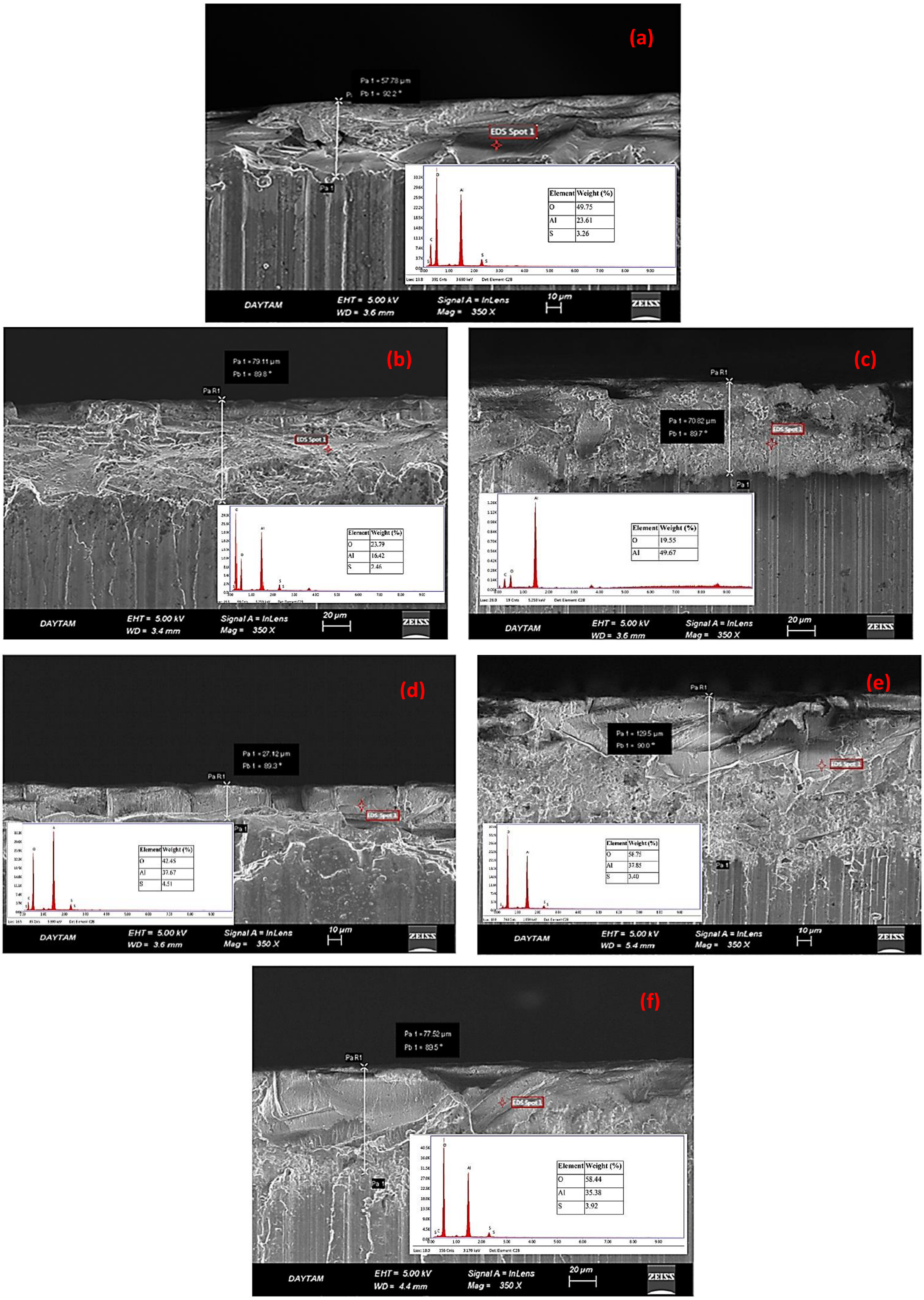


Figure 4. SEM images and EDS analyze of the hard-anodized samples ((a): 18 % H_2SO_4, (b): 18 % H_2SO_4 + 0.5 M $C_2H_4O_2$(acetic acid), (c): 18 % H_2SO_4 + 0.5 M $C_3H_4O_3$ (pyruvic acid), (d): 18 % H_2SO_4 + 0.5 M $C_3H_4O_4$ (malonic acid), (e): 18 % H_2SO_4 + 0.5 M $C_4H_6O_6$ (tartaric acid), (f): 18 % H_2SO_4 + 0.5 M $C_6H_8O_7$(citric acid))

3.1.3. FTIR (Fourier Transform Infrared Spectroscopy) analysis

FTIR analysis results are given in Figure 5. In order to determine the functional groups in the structure of the organic compounds in the oxide layer obtained on the surface, the state of the bonds in the structure, the binding sites and whether the structure is aromatic or aliphatic, the cross-sectional samples were examined using Fourier Transform Infrared Spectroscopy (FTIR) in the 4000-500 cm^{-1} band range. Al-O-H bonds are seen in sulfuric acid-based films at around 3750 cm^{-1}. It was stated in studies conducted by (Yaniv et al. 1985) that the spectral regions between 550-850 cm^{-1} belong to the Al-O bonds of amorphous alumina, and the spectral regions between 750-920 cm^{-1} belong to the Al-O-Al bonds. From the peaks obtained as a result of FTIR analysis, the formation of Al-O bonds proves the presence of hydroxyl groups. The intensity of the peaks shows how strong the bond formed in the FTIR analysis is (Koçer, 2019). The 1626 cm^{-1} band has been associated with O-H stretching and O-H deformation (Anjos et al. 2015, Se et al. 2018). It has also been reported that the weak bands between 1200-1500 cm^{-1} are associated with the deformation of $-CH_2$ and the angular deformation of C-C-H and H-C-O bonds (Gök et al. 2015, Se et al. 2018). 1150 cm^{-1} and 1380 cm^{-1} absorption bands; It is attributed to Al-O-S-O or Al-O-S-OH bonds and is also seen here.

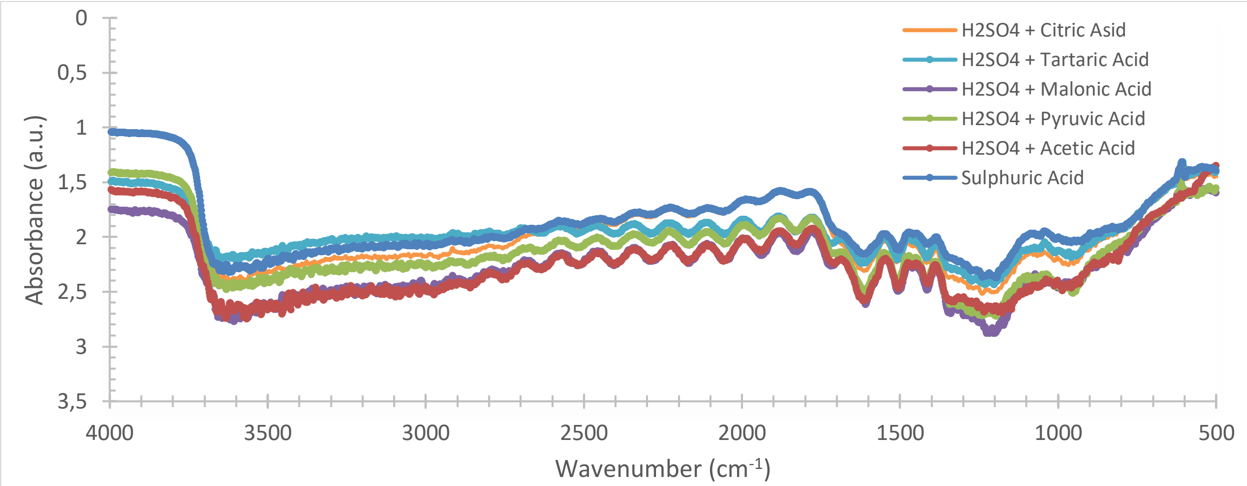


Figure 5. FTIR analyses results

3.2. Microhardness

The average microhardness values of uncoated samples and anodic oxidation-treated samples were measured by making 5 measurements of the cross-section surfaces under a 25 g load and a waiting time of 10 s. The hardness of the coatings was determined by taking the average of these 5 values. Figure 6 shows the average hardness values that were measured in at least five different points for each experiment.

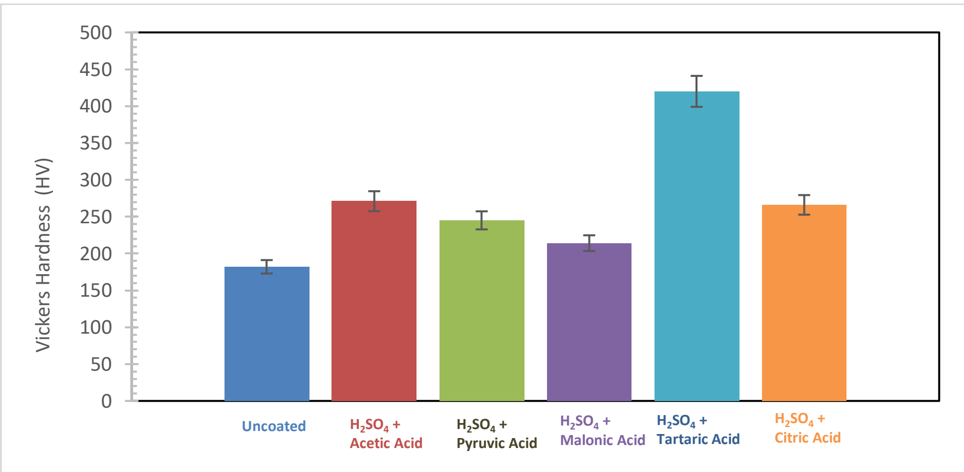


Figure 6. Vickers hardness results

4. Conclusion

The chemical content of the organic acids added to the sulfuric acid bath causes differences in the color tone of the resulting coating. The noticeable difference in tone on the color is a sign of the extent to which the color pigments of the paint used have penetrated the porous structure of the hard coating formed on the aluminum plate, thus giving a preliminary idea about the coating thickness obtained.

The highest coating thickness of 129.5 μm was obtained by adding tartaric acid to the acid bath. As a result of EDS analysis, it was determined that the dominant elements in the structure were Al and O and formed the dominant alumina-based structure. It is thought that the presence of tartaric acid in the acid bath reduced the intensity of oxygen evolution in the anodic film and weakened the dissolution rate of the anodic film. It was determined that in addition to Al and O elements, S and C elements were also present in the structure and the ratios of these elements varied according to the types of organic acids.

As a result of the hardness analysis, the highest hardness value of 420 HV was obtained as a result of anodic oxidation by adding tartaric acid into a sulfuric acid bath.

Conflict of Interest
No conflict of interest was declared by the authors.

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