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DETERMINATION OF TITANIUM IN ZINC ASH BY FAAS AFTER DIGESTED USING ULTRASOUND-ASSISTED EXTRACTION

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ABSTRACT

In this study, it is aimed to determine the amount of Ti (titanium) in waste ash samples taken from galvanizing factories by using flame atomic absorption (FAAS). Various time periods and different solvent mixtures were examined by using ultrasound-assisted extraction for digestion of zinch ash which taken from various processes of galvanizing metal sheet or pipe. The best solvent mixtures for Ti metal in galvanized waste was found $HCl:HNO_3:H_2O_2$ (1:3:2, v:v:v)>HCl>HNO_3:HCl (1:3, v:v)>HNO_3>$HNO_3:H_2SO_4$ (3:1, v:v)>$HNO_3:H_2SO_4$ (1:1, v:v)>$HNO_3:H_2SO_4$ (3:2, v:v)>$HNO_3:H_2SO_4$ (2:1, v:v), respectively.

Keywords: Ultrasound-assisted extraction, Waste ash, Titanium, Solvent mixtures.

ULTRASON DESTEKLİ EKSTRAKSİYON KULLANILARAK ÇÖZÜNÜRLEŞTİRMEDEN SONRA ÇİNKO KÜLÜNDEKI TİTANYUMUN FAAS İLE TAYİNİ

ÖZ

Bu çalışmada, alevli atomik absorpsiyon (FAAS) kullanılarak galvaniz fabrikalarından alınan atık kül örneklerindeki Ti (titanyum) miktarının belirlenmesi amaçlanmıştır. Çeşitli galvanizleme sac veya boru proseslerinden elde edilen çinko külünün çözünürleştirilmesi için ultrases destekli ekstraksiyon kullanılarak çeşitli zaman periyotları ve farklı çözücü karışımları incelenmiştir. Galvanizli atıkta Ti metali için en iyi çözücü karışımları sırasıyla, $HCl:HNO_3:H_2O_2$ (1:3:2, h:h:h) > HCl > HNO_3:HCl (1:3, h:h) > HNO_3 > $HNO_3:H_2SO_4$ (3:1, h:h) > $HNO_3:H_2SO_4$ (1:1, h:h) > $HNO_3:H_2SO_4$ (3:2, h:h) > $HNO_3:H_2SO_4$ (2:1, h:h) olarak bulunmuştur.

Anahtar Kelimeler: Ultrases destekli ekstraksiyon, Atık kül, Titanyum, Çözücü karışımları.

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INTRODUCTION

The method of zinc coating applied to the surface in order to prevent corrosion in metals is called galvanization. Zinc ash is formed during the various processes of galvanizing metal sheet or pipe. In the galvanizing process, the casting, smelting and other metal industries generate large amounts of waste containing high amounts of zinc [1-4]. In the galvanizing process, while zinc and nickel are commonly used for metallic coating; Cadmium, copper, tin, chrome, gold and silver metals are also used [5]. Since the waste ash generated after the galvanizing process may contain precious metals, it should be evaluated.

Ti element, which is rare in the nature, is the most abundant element in the earth's crust, after aluminum, iron and magnesium at the rate of 6 per thousand. Ti is one of the transition metals with atomic number 22, located in the 4B group 4th period d orbital in the periodic table [6]. The sparse distribution of ore and the difficulty of obtaining Ti from its ore makes it an expensive metal. The element Ti, which is very distributed in the earth's crust, is rutile (TiO_2), ilmenite ($FeTiO_3$), titanomagnetite ($Fe_3O_4.TiO_2$), perovskite ($CaTiO_3$), etc. It is one of several important ossicles and is also found in alluvial-volcanic rocks and sediments formed from them. Sediments can generally contain the heavy metals in the range of 3-12%, consisting of ilmenite, rutile, leucoxene, zircon and monazite [3-4]. Ti, which is a 57% lighter metal compared to steel, has low density (4.5 g/cm^3) and good hardness, has low thermal conductivity and expansion coefficient, high melting point (1660 °C). As it is easily processed between -240 °C and 540 °C and has the highest corrosion resistance of all metals against atmospheric corrosion, is more resistant than steel and resistant to almost all chemical effects, titanium is a sought-after metal in aircraft, space, medical and many other fields material. In addition, it is widely used in the plastic industry and electrical applications as it increases the durability of materials against UV rays and extreme temperatures [8,9].

Petry et al. determined Ti and V in Ziegler-Natta polymerization catalysts by inductively coupled plasma optical emission spectrometry (ICP-OES). They determined 0.80%-1.00% Ti in the catalysts used in polymerization. In addition, they determined that Ti in catalysts did not show homogeneous distribution with scanning electron microscope-energy dispersing X-ray (SEM-EDX) device [10]. Mirzaei and Naeini conducted a study to determine the trace amount of Ti by

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flame atomic absorption spectrometer after cloud point extraction. They determined the RSD (relative standard deviation) value for Ti as 3.8% [11].

Solvent extraction, ionexchange, ultrasound-assisted extraction and adsorption were investigated the digestion technique for removal of heavy metals from natural samples by many researchers [12-18]. In this study was used the ultrasound-assisted extraction as digestion technique.

In study, it is aimed to perform method validation and optimization by using flame atomic absorption spectroscopy (FAAS) for the analysis of total Ti in waste ash used in galvanizing process. To prepare the ash samples for analysis, different solvent mixtures were tried and the best solvent/solvent mixture was determined. During the preparation of the samples for analysis, the fast and effective SAE device (with time optimization) was used. The optimum results found were applied to the real samples.

MATERIAL AND METHOD

Preparation of Standard Solutions

In order to obtain the calibration plots used in quantitative analysis, stock and dilute solutions were prepared for Ti at known concentrations and metal contents were analyzed with FAAS. The standard of Ti is 1000 mg/L standard NIST (National Institute of Standard and Technology, primary standard material) stock solution. A suitable appropriate dilute (50 mg/L) solution was prepared from a 1000 mg/L stock solution. Then, standard solutions for Ti in the range of 1-25 mg/L were prepared from these appropriate dilute solutions [19].

Solubilization Study with Different Acid Mixtures

Samples weighed in 0.25 g on analytical precision balance 5 mL of HCl; HNO_3; HCl:HNO_3: H_2O_2 (1:3:2, v:v:v); HNO_3:H_2SO_4 (1:1, v:v); HNO_3:H_2SO_4 (2:1, v:v); HNO_3:H_2SO_4 (3:1, v:v); HNO_3:H_2SO_4 (3:2, v:v); HCl:HNO_3 (3:1, v:v); It was dissolved in ultrasonic bath for different times (5 min; 10 min; 20 min; 30 min; 60 min) with solvents and centrifuged at 8000 rpm for 15 min and decanted. In order to prevent small particles from remaining in the solution, filtering was carried out with a filter paper. The volume of the prepared solutions was completed to 10 mL with ultrapure water and analysed with FAAS

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RESULTS

The measurement of Ti was performed at a wavelength of 363.4 nm, a slit width of 0.5 nm, and a deuterium lamp current of 20.0 mA at 6.95/11.00 acetylene/N_2O gas flow.

Calibration charts of standard solutions are presented in Figure 1.

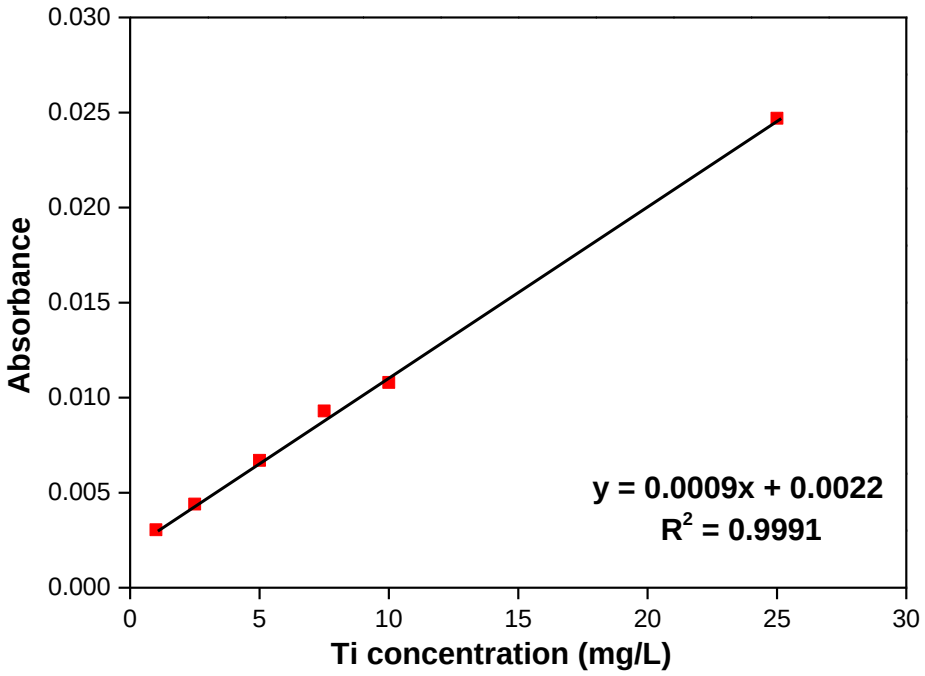

Figure 1. Ti concentration-absorbance curve determined by FAAS

The Ti concentration curve against the time obtained in the dissolution process with HCl, HNO_3, HCl:HNO_3:H_2O_2 (1:3:2, v:v:v), and HNO_3:HCl (1:3, v:v) are given in Figure 2.

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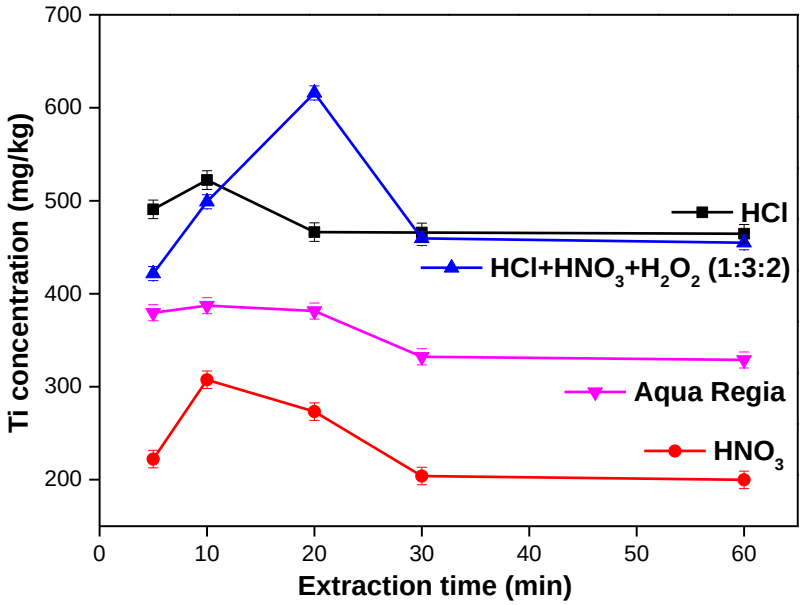

Figure 2. The best extraction time of Ti in the solubilization processes with HCl, HNO_3, HCl:HNO_3:H_2O_2 (1:3:2, v:v:v), and HNO_3:HCl (1:3, v:v)

The Ti concentration curve against the time obtained in the dissolving process with the mixture of HNO_3:H_2SO_4 (2:1, v:v), HNO_3:H_2SO_4 (3:1, v:v), HNO_3:H_2SO_4 (3:2, v:v), and HNO_3: H_2SO_4 (1:1, v:v) are given in Figure 3.

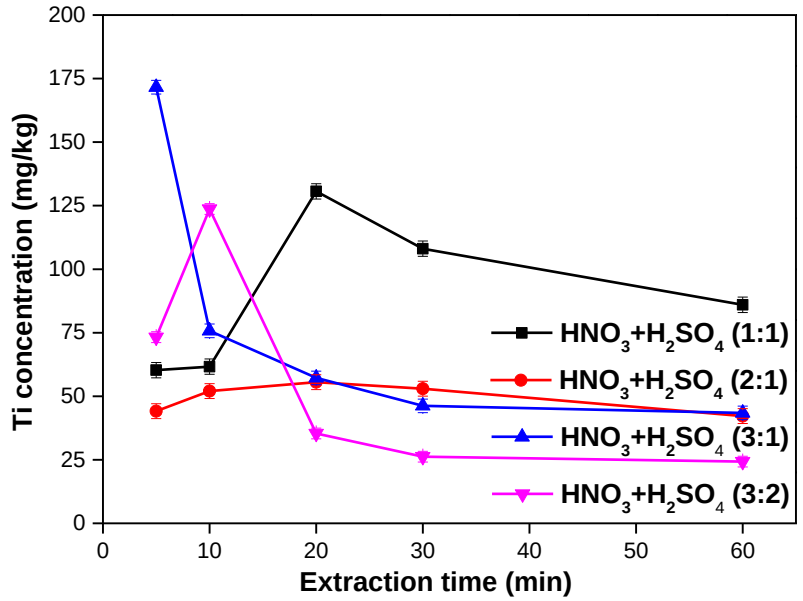

Figure 3: The best extraction time of Ti in the solubilization processes with HNO_3:H_2SO_4 (2:1, v:v), HNO_3:H_2SO_4 (3:1, v:v), HNO_3:H_2SO_4 (3:2, v:v), and HNO_3: H_2SO_4 (1:1, v:v)

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CONCLUSION

Ti; It is a material that is sought and used in many areas such as the plastic industry, electrical applications, aviation industry (in aircraft body construction, jet engines, missiles), biomedical applications, fire retardants, insulation materials, rubber, leather and textile industries, as it can be easily processed, has high corrosion resistance, and is more robust and lighter than steel.

During the preparation of the samples for analysis, the fast and effective SAE device (with time optimization) was used. Different solvent mixtures (HCl, HNO_3, HCl:HNO_3:H_2O_2 (1:3:2, v:v:v), HCl:HNO_3 (3:1, v:v)) and different solubilization times (5 min; 10 min; 20 min; 30 min; 60 min) in an ultrasonic bath were tried to prepare the ash samples for analysis. When the obtained results were examined, it was determined that the maximum dissolution process took place in 20 minutes with the mixture of HCl:HNO_3:H_2O_2 (1:3:2, v:v:v) for Ti.

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