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*RESEARCH ARTICLE*

# OPTIMIZATION of PLATINUM BATHS USED in JEWELRY INDUSTRY in TURKEY

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## ABSTRACT

Platinum group metals are used a lot in fields such as jewellery, electronics and health due to being precious metals and their brightness, corrosion resistance, thermal resistance and electrical conductivity. With the excessive increase in the prices of gold and rhodium, which are the most used in the jewelry sector, a tendency towards the more affordable platinum metal has begun. In this study, the optimization of baths used in the jewelry industry, which is one of the areas where platinum coating is used most, is discussed. The conditions required for the re-coating of the platinum bath, which is used and separated as waste in jewelry workshops, were investigated experimentally. A waste bath was taken from a jewelry plating workshop in the Grand Bazaar, and the content of the bath was determined by ICP-OES analysis and IR Spectroscopy. Later, on this waste platinum bath, a few substances, which are used as brighteners in the market, and which show parallel diffraction with the original bath in FTIR analysis were added and the effects of temperature, current and time parameters on the coating were investigated. L*a*b values were measured with PCE Instruments Colorimeter device by making platinum coating on brass-coated plates using the waste bath to which brighteners were added. As a result of the study, the brighteners and color properties of the coated surfaces and the reference coating properties were compared.

**Keywords:** *Platinum, Metal coating, brightness*

## 1. INTRODUCTION

Rhodium, platinum and palladium of Groups 9 and 10 in the periodic table which are considered to be precious metals are called platinum group elements (PGM). These metals have high corrosion resistance and good thermal properties in pure form, and they are mostly good conductors [1]. Thanks to the characteristics they demonstrate, PGMs have a wide range of uses in the automotive industry, chemistry industry, electronic parts, ceramics, glass industry as well as some aviation components [2], and jewelry. Since these metals are precious metals, they can demonstrate the same properties with a lower consumption amount when the coating technique is applied. Table 1 shows the platinum consumption by industry within the last three years. When the amount is analyzed, it is seen that the largest use of platinum within the last three years was in automotive and jewelry industry. They are used in converters, filters and electronic parts in the automotive industry. As for jewelry, platinum coatings are made on decorative products and jewelry [3].

**Table 1.** Platinum consumption by industry within the last three years [4].

| **SECTORAL DEMAND (tonnes)** | **2019** | **2020** | **2021** |
|---|---|---|---|
| Automotive | 89 | 71.2 | 90.5 |
| Chemical | 20.8 | 19.9 | 19.7 |
| Electronics | 7.2 | 7.5 | 8.7 |
| Glass | 13.7 | 14.1 | 16 |
| Investment | 35.1 | 31.8 | 9.6 |
| Jewelry | 64.2 | 53.1 | 55.9 |
| Medical ve biomedical | 7.5 | 6.7 | 7.2 |
| Petroleum | 8 | 9.4 | 5.4 |
| Other | 18.3 | 14.3 | 16.8 |
| **Total gross demand** | **263.8** | **228** | **229.8** |

### 1.1. Coating

Surface coatings, which are obtained by applying a material to the surface of another either by overlaying or precipitating with the purpose of protecting the material from the effects of the environment as well as improving its appearance and properties [5]. Gaseous state, solution state and molten/semi-molten state processes are used in metallic coatings.

The most common plating method in the jewelry industry is the electrolytic plating method. This method is carried out by depositing a metal on the surface with the help of an electric current [6]. In the electroplating device, the part to be coated becomes the cathode. A high conductivity metal is used as anode. The anode and cathode placed in the electrolyte containing the material to be coated are connected to a power source. When the current is applied, reduction occurs and metal ions accumulate on the coated material [7].

The success of a coating process depends on initial surface preparation which includes removing burrs and filing. Prior to coating, some other processes are applied to the surface for cleaning. They are essential since they affect the properties and quality of the coating. For this reason, in order to make the surface appropriate for the coating process, some processes such as removing impurities, improving the surface roughness and activating the surface to be coated are performed by chemical, electrochemical or mechanical means.

The brightness of the surface is a desired feature in the metal plating process. The glossy surface determines that the reflection is smooth. As for rough surfaces, it would be difficult to achieve gloss since the reflection will not be smooth. In chemical and electrochemical processes to improve the roughness of the surface, some additives called brighteners are added to the electrolyte. These additives are classified into two categories; namely, carrier brighteners and brightening additives. Carrier brighteners, also known as primary brighteners, are given below.

- Sulfonamides
- Sulfonimides

- Benzene sulfonic acids (mono-di or trisulfonic acids)
- Alkyl sulphonic acids
- Sulfinic acids
- Arylsulfonsulfanates

The use of these brighteners alone is not enough. Additional additives are used to provide a brighter appearance. These additives are given below [7];

- Thiourea
- Acylthiourea
- Mercaptoalkylsulfonic acid
- Bis(sulfoalkyl) acid amide
- Thiocarboxylic acid amine
- Thiocarbozone
- Thiosemicarbozone
- Thiohaydantoin

Figure 1 shows a laboratory-scale mechanism of a catalytic coating. The ionic solution is called the electrolyte. Although electrolytes are generally in liquid form, they may also be gases.

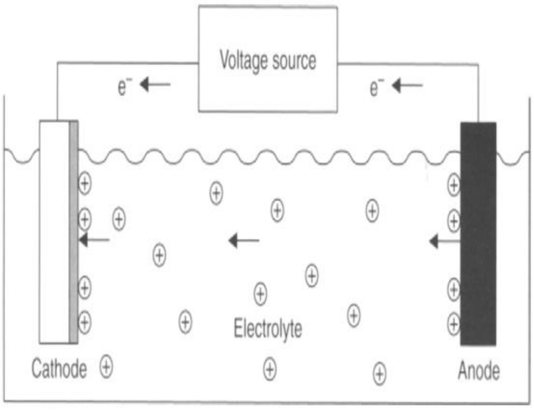

**Figure 1.** Laboratory-scale mechanism of a catalytic coating.

While the most convenient salt of the metal is mainly used in an electrolyte, impurities may also contribute to the bath processes.

The anode is the positively charged electrode used to coat the material. While there is deposition at the cathode, the anode is only for ionization reactions, so no disposition occurs.

The cathode is the negative-ended electrode on which the coating process is performed. For flow coating, the material, which has undergone the necessary pre-cleaning processes, is immersed in the solution and the coating is performed because of the required contact. Reactions 1 and 2 [7] show the fundamental reactions occurring at the cathode and the anode.

$Me^{n+} + n^{e-} \longrightarrow M$ (cathodic reaction) (1)

$Me^{0} \longrightarrow Me^{n+} + n^{e-}$ (anodic reaction) (2)

Electric current is delivered by a power supply. Furthermore, there is also a temperature sensor and stirrer available in electroplating devices.

Since current is used in electrolytic coating, high current and low current zones are formed on the metal surface. High flow zones occurring on uneven surfaces will lead to inhomogeneous coating. The material to be coated must be prepared for pre-treatment.

For the deposition thickness to be the same in every region of the surface, the current density must be the same at all points of the cathode. This is possible only if the distance between the closest point of the anode and every point of the cathode is the same. The current-related resistance between different points of the anode and cathode is different, and therefore the current density at these points varies [8].

The coating can be divided into two stages as surface preparation and surface coating processes. Burr cleaning and filing are the physical processes to be performed. Prior to preparing the surface, it must initially undergo a cleaning phase which includes hot degreasing, electrolytic degreasing, and acidic cleaning. Then the coating is performed and followed by quality control Water rinsing process is performed at each step.

The first cleaning phase is through hot degreasing. The material should be free from chemicals used during preparation, lubrication through touching as well as environmental dirt and oxides. Then the acidic cleaning is performed. Even if the parts are lubricated, they are subject to some corrosion. Baths with 20-50% acid content are used to remove the rust. During acidic cleaning, an inhibitor should be used to prevent the iron from corroding because of the acid and contaminating the bath. Examples of acid solutions used in surface cleaning and the processing conditions of these solutions are given in Table 2.

In electrolytic degreasing, although the surface of the part is flat, it should not be ignored that there are micro pits on the surface. Electrolysis with water is conducted to clean the remaining oil in these pits. As a result of hydrolysis, the released hydrogen molecules take the oil in the micro pits and bring it to the surface.

**Table 2.** Acid types and concentrations used in surface cleaning [5].

| No | Acid Solution | Concentration | Work Conditions |
|---|---|---|---|
| **1** | Hydrochloric acid (HCl) | %20-85 | Room temperature |
| **2** | Sulfuric acid ($H_2SO_4$) | %5-15 | Under inhibitor conditionss and at 50-80 °C |
| **3** | Sulfuric acid ($H_2SO_4$) | %4-6 | At 50-65 °C and at 3.2-6.5 A/dm² |
| **4** | Hydrochloric acid (HCl)<br>Nitric acid ($HNO_3$) | %20-85<br>%1-5 | Room temperature |
| **5** | Sulfuric Asid ($H_2SO_4$)<br>Potassium nitrate ($KNO_3$) | 22.5 g/l<br>22.5 g/l | At 70 °C |

**Table 3.** The most commonly used coating baths [5].

| Metal | Bath type | Anode | Cathode current density (A/dm²) | Temperature (°C) | Current efficiency (%) |
|---|---|---|---|---|---|
| **Gold** | Cyanide | Au | 0.1-0.5 | 60-80 | 70-90 |
| **Copper** | Asid-Sulfate | Cu | 1-4 | 25-50 | 97-100 |
| | Alkali Cyanide | Cu | 0.3-1.5 | 35-40 | 30-60 |
| **Zinc** | Sulfate | Zn | 1-3 | 20-30 | 99 |
| | Sulfate (Hot) | Zn | 8-10 | 50-60 | |
| | Chloride | Zn | 4-10 | 20-40 | |
| | Alkali Cyanide | Zn | 0.8-2 | 40-50 | 85-90 |
| **Iron** | Chloride | Fe | 10-18 | 90-100 | 90-98 |
| | Couple Sulfate | Fe | 2-3 | 20-30 | 95-98 |
| **Silver** | Cyanide | Ag | 0.3-0.8 | 15-25 | 98-100 |
| **Cadmium** | Alkali Cyanide | Cd | 1-5 | 20-30 | |
| **Tin** | Alkali Stannite | Sn | 1 | 50 | |
| | Alkali Stannate | Sn | 0.5-1.5 | 60 | 70-95 |
| **Cobalt** | Sulfate | Co | 3-17 | 20-30 | 95-98 |
| **Chromium** | Chromic Acid +Sulfate | Pb | 10-30 | 40-50 | 12-20 |
| **Lead** | Fluoroborate | Pb | 0.5-2 | 20-30 | 20-93 |
| | Perchlorate | Pb | 2-3 | 20-30 | 95 |
| **Nickel** | Sulfate | Ni | 0.5-2 | 20-30 | 94-98 |
| **Sulfate** | Sulfate Chloride | Ni | 1.5-5 | 50 | 94-98 |
| **Palladium** | Chloride | Pd | 1 | | 98-100 |
| **Platinium** | Phosphate | Pt | 0.1 | 70 | |
| **Alloy** | Cyanide | Cu-Zn | 0.2-0.3 | 32-45 | 80 |

Only a few of these brighteners, which provide a bright appearance, are used in the coating industry. The major reason for this is the necessity of optimizing the appropriate temperature and current density in order to make the coatings [7].

Afterwards, degreasing and acidic cleaning processes are performed. After the coating is completed, the surface is made ready for use by rinsing and drying, respectively [9], [10].

The plating bath basically contains the salts of the metal to be coated. However, conductivity enhancers, brighteners, surface wetting agents and buffer solutions can be added to increase the coating quality and facilitate the process [7], [11].

There are multiple factors for the effectiveness of a coating. Among these are current density, current efficiency, pH, temperature, concentration, dispersion power, and surface properties [8], [12], [13].

## 2. MATERIALS AND METHODS

This study was carried out in the chemical engineering laboratory of Yıldız Technical University. A jewelry workshop in the Grand Bazaar provided a bath solution which was used before and separated as waste since it was not functioning any more. The parts to be coated were pre-treated, polished, degreased and cleaned, and made ready for the experiments. As a result of the literature review and market research, 2 different brighteners were determined and obtained to be used in the experiments. For the electrolysis setup, a regulator (Yıldırım Electronic Devices Ind. Trd. Ltd. Co.), connection cables, a heater (Yellowline MSC Basic C) were used. In addition, Perkin Elmer Spectrum Two FTIR and PCE Instruments Colorimeter devices were used. ICP-OES analysis was conducted in the Metallic Analysis Laboratory in Başakşehir. The experimental setup used for the electroplating is given in Figure 2.

**Figure 2.** Electrolytic coating mechanism.

The platinum bath used by the jewelers was analyzed both before it was used and in its waste condition to be used in the experiments using the ICP-OES 4200MP-AES model device to determine the amount of platinum it contained, and the difference in their chemical structures was checked by FTIR analysis in Figure 3. In the experiments, Saccerete Clear and Triton-x 100 brighteners were added to the waste solution taken from the workshop, whose ICP results are given in Table 4.

Triton-x 100 is a nonionic surfactant with a hydrophilic polyethylene oxide chain and an aromatic hydrocarbon lipophilic or hydrophobic group. It is used in industrial (metal plating) processes [14].

By adding certain amounts of brighteners, coating was made at a temperature range of 35 °C, 45 °C and 55 °C, with a current of 2.5 A/dm$^2$ in a processing time of 1 minute. Coating was done under the same conditions as the original coating bath, and the waste bath was optimized to achieve the desired brightness and color properties of the coating.

**Table 4.** ICP-OES analyses results for the original bath and waste bath.

| Sample Name | Test Method | Test materiel | Result |
|---|---|---|---|
| **Original Pt Bath** | ICP-OES-MP-AES | Pt | 4.001 g/lt |
| **Waste Pt Bath** | ICP-OES-MP-AES | Pt | 2.065 g/lt |

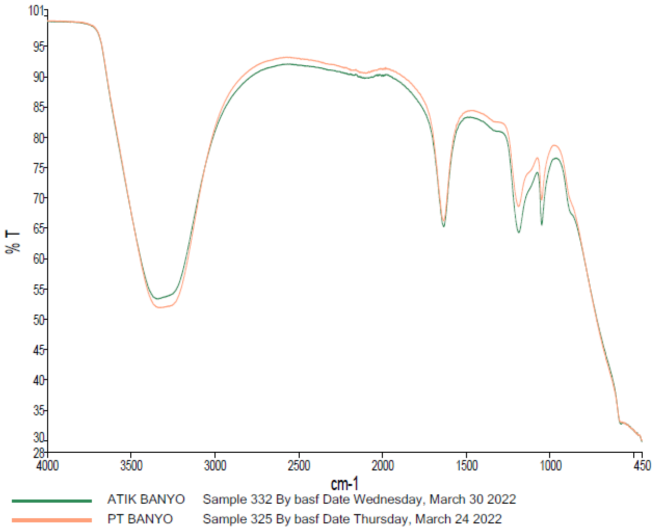

**Figure 3.** Comparative FTIR graphs of original bath and waste bath.

After coating processes, L, a, b values were measured using the PCE Instruments Colorimeter device. Measurement results represent points within a three-dimensional color system, as shown in Figure 4. The L value on the vertical axis can be defined as brightness. Regarding the L value ranging between 0-100, zero represents black while a hundred represents white. The a and b values on the horizontal axis represent the yellow, blue, red, and green tones of the color. “b” is the yellow/blue axis with b (+) indicating the yellow hue while b (-) indicating the blue hue. Likewise, in the “a” axis representing the red/green colors, a (+) indicates the red hue, while a indicates that the green hue increases in the (-) direction [15].

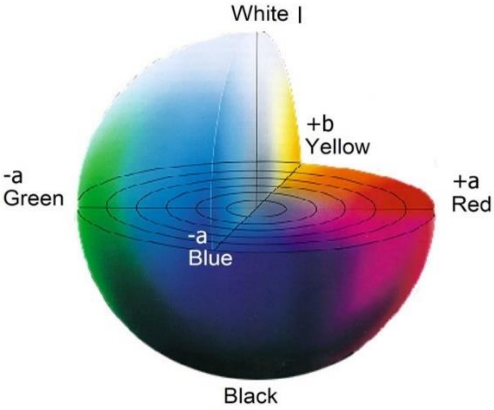

**Figure 4.** L*a*b color graph [16].

## 3. RESEARCH RESULTS

The ICP-OES analysis revealed that the amount of Pt in the waste bath, which was separated by the workshop as it did not yield sufficient brightness, was reduced to approximately half. Since it is known that it does the coating in half the amount but does not reach sufficient brightness, the coating processes were carried out by adding the brightening agents Saccerete Clear and Triton-x 100. Under the same conditions, coatings were also made with the original bath which had a Pt content of 4 g/lt. L*a*b measurements were made for three different areas for each coating with the PCE Instruments Colorimeter device and the results are given in Table 5, Table 6, Table 7 and Table 8.

**Table 5.** L*a*b results of the original bath containing 4 g/L of Pt and the coatings at 35 °C, 45 °C and 55 °C.

| | | **2.5 A (7-8 V)** | | |
|---|---|---|---|---|
| | | **L** | **a** | **b** |
| **4 g/l Original Pt bath** | 35 °C | 93.7 | 1.91 | 6.84 |
| | | 93.87 | 2.23 | 6.89 |
| | | 92.98 | 2.1 | 7.18 |
| | 45 °C | 95.04 | 1.81 | 7 |
| | | 94.85 | 2.05 | 7.07 |
| | | 93.52 | 2.26 | 6.67 |
| | 55 °C | 95.42 | 1.78 | 5.17 |
| | | 94.31 | 1.68 | 5.74 |
| | | 94.59 | 1.83 | 5.72 |

**Table 6.** L*a*b results of coatings at 35°C with the filtered waste bath containing 2.065 g/L of Pt.

| | | **35 °C** | | |
|---|---|---|---|---|
| | | **L** | **a** | **b** |
| **Waste bath** | 2.5 A (7-8 V) | 72.59 | 1.79 | 6.77 |
| | | 72.32 | 1.83 | 6.56 |
| | | 74.49 | 2.66 | 7.43 |
| **Saccerete Clear 0.5 ml additional** | 2.5 A (7-8 V) | 87.48 | 2.33 | 6.09 |
| | | 89.96 | 2.62 | 7.0 |
| | | 89.06 | 2.72 | 6.36 |
| **Triton-x 0.25 ml additional** | 2.5 A (7-8 V) | 74.6 | 3.24 | 6.89 |
| | | 77.82 | 3.32 | 6.4 |
| | | 76.22 | 4.24 | 8.02 |

**Table 7.** L*a*b results of the filtered waste bath containing 2.065 g/L of Pt and the coatings at 45 °C.

| | | **45 °C** | | |
|---|---|---|---|---|
| | | **L** | **a** | **b** |
| **Waste bath** | 2.5 A (7-8 V) | 77.21 | 2.6 | 8.85 |
| | | 78.73 | 2.99 | 8.95 |
| | | 78.4 | 2.24 | 8.27 |
| **Saccerete Clear 0.5 ml additional** | 2.5 A (7-8 V) | 84.06 | 3.58 | 8.6 |
| | | 84.45 | 3.46 | 8.75 |
| | | 86.78 | 2.5 | 7.63 |
| **Triton-x 0.25 ml additional** | 2.5 A (7-8 V) | 85.33 | 4.22 | 7.78 |
| | | 83.91 | 4.34 | 8.51 |
| | | 83.06 | 4.35 | 8.87 |

**Table 8.** L*a*b results of the filtered waste bath containing 2.065 g/L of Pt and the coatings at 45 °C.

| | | **55 °C** | | |
|---|---|---|---|---|
| | | **L** | **a** | **b** |
| **Waste bath** | 2.5 A (7-8 V) | 78.49 | 3.65 | 11.7 |
| | | 74.71 | 3.18 | 11.51 |
| | | 79.75 | 3.97 | 12.24 |
| **Saccerete Clear 0.5 ml additional** | 2.5 A (7-8 V) | 88.39 | 2.4 | 7.36 |
| | | 87.41 | 2.46 | 7.18 |

| | | | | |
|---|---|---|---|---|
| | | 86.73 | 2.6 | 7.54 |
| **Triton-x 0.25 ml additional** | 2.5 A (7-8 V) | 72.29 | 4.64 | 13.39 |
| | | 73.57 | 4.42 | 13.19 |
| | | 71.88 | 4.49 | 13.21 |

## 4. DISCUSSION

Electrolysis of platinum dates back to ancient times. In 1989, Matthey described a new bath in his patent. In this bath, based on the tetrammineplatinum (II) solution, the coating process is achieved at Pt (NH$_3$)]$^+$, low concentration, aqueous buffer at a pH range of 10.0-10.6, operating at the recommended operating temperature range between 91 and 95°C.

Skinner [3] used a new bath in his study, and as a result, it is seen that this study made a high performance platinum coating with a smooth surface at low current density.

In various coating studies, in the thesis study of Bakan [7], the effect of different pH values on the brightness of the silver coating was investigated, and when the effect of the additives on the coating color was examined, it was observed that the ethylene glycol additive alone gave the color closest to the reference silver sample.

In a study conducted by Satpathy et al., it was stated that silver coatings were made with the environmentally friendly thiosulfate-based silver electroplating method using agalvanic bath and brightness was achieved [17].

## 5. CONCLUSION

As seen in Table 6, the effect of the addition of Saccerete Clear at 35 °C on the L, a, b values of the prepared bath is seen. An L value of 89.96 stands for brightness and it is seen that a is measured as 2.62 while b is 7. According to the color scale in Figure 4, it is seen that the b value is obtained as a color close to green. Again, in Figure 4, it is seen that the a value is close to red.

According to Table 6, when Triton -x 100 is used, it has been observed that L, a, b values decrease and foam occurs in the solution. For this reason, it has been determined that it causes a negative effect on the coating. As seen in Table 7, increasing the bath temperature from 35°C to 45°C increased the L, a, b values in the filtered waste bath, while the addition of 0.5 ml of Saccerete Clear at 45°C dropped the L, a, b values of the bath. Additions of Triton- x 100 at 45°C increased the L, a, b values. In Table 8, although it is expected that there will be a positive effect on bath efficiency with an increase in temperature by 55°C, a decrease was observed in the other brightener except for Saccerete Clear. The revelation of high a, b values as a result of the brightener addition indicate a deviation from the original bath. As seen in Table 5, it was determined that the L, a, b values were not satisfactory. In addition, comparative FTIR graphs of the original bath and the waste bath are given in Figure 3. According to this graph, a big difference is not observed in the spectra.

In this study, the waste bath used in the jewelry industry was filtered as a pre-treatment to get rid of sediments and by adding 0.5 ml of Saccerete Clear and Triton-x 0.25 ml brighteners to this bath, 2.5 A ( 7-8 V) current density coatings were made.

It is known that approximately 2.5 kg of coating was made with the original 4 g/L Pt bath. In the same experimental conditions, L, a, b values on the color scale of the original bath were also examined.
It is seen that the L, a, b values were found to be 95.42, 1.78, and 5.17, respectively, at 55 °C, which gives the best brightness in the original Pt bath. In the study, in which brighteners have been added at 55°C, it is seen that the expected increase in L, a, b values could not achieve the brightness of the original bath. Only with the addition of 0.5 ml of Saccerete Clear, L, a, b values of 89.96, 2.62, 7.0 indicates an achievement of a bit of brightness at 35°C which is still not the desired brightness of the original bath.

Both of the brighteners used could not provide the desired brightness in the bath. It was concluded that it is not suitable for use as a commercial product.

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