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Research paper

Low-Pressure Plasma; An Alternative Method of Cleaning Historical Silver Coins

***M. Aminabadi**[1*], **S. Khadir** [2*], **M. Akbari**[1], **S. Javadi Anaghizi**[1], **M Khani**[1**], **B. Shokri** [1]*

*[1]**Laser and Plasma Research Institute, Shahid Beheshti University, G.C., Evin, Tehran, Iran***

*[2] **the Research Institute of Cultural heritage and Tourism, Tehran, Tehran, Iran***

****Mohsen Aminabadi and Shiba Khadir are both first authors***

[**] *m_khani@sbu.ac.ir*

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Abstract

Cold plasma treatment is a green technology for cleaning and surface decontamination in various applications. The work in the present paper discusses the use of high-frequency cold plasma for the removal of corrosion products and surface deposits from historical silver coins. The coins, conserved by the Cultural Heritage Organization of Iran, are of the Parthian period and are of significant historical and cultural value. Cold plasma is non-invasive in nature and efficient, with various advantages over traditional cleaning methods. In an attempt to quantify the elemental composition of the coin surfaces, X-ray fluorescence (XRF), scanning electron microscopy (SEM), and energy-dispersive X-ray spectroscopy (EDAX) were performed. The success of the treatment using the plasma was later analyzed by gravimetry, optical microscopy, and comparative imagery. The results indicated a significant reduction in the surface deposits, as confirmed by the measurements of the weights as well as by the microscope. The before-and-after imagery also indicated the improvement in the clarity of the coin designs, which were originally obscured by the contamination. These findings indicate that the treatment with cold plasma is a promising, non-invasive technique for the restoration of historical metal artifacts, which restores the surface features of the artifacts with minimal risk of damage.

1. Introduction

The corrosion of ancient metals, particularly coins, is caused by various physical and chemical processes, with improper preservation conditions being a leading factor. These processes gradually alter the appearance, shape, composition, and mechanical properties of metals. In severe cases, they can degrade artifacts to such an extent that their historical significance as records of past civilizations is lost, potentially leading to their complete destruction[1]. Exposure to adverse environmental conditions, such as humidity and oxidizing agents, further accelerates surface damage. Many of these coins are discovered during construction in historical sites or through unauthorized excavations. Once uncovered, cleaning becomes essential to remove surface deposits and oxide compounds using traditional restoration methods. However, conventional cleaning techniques not only eliminate contaminants but may also damage the underlying metal surface.

In comparison, cold plasma treatment offers a significant advantage by effectively removing surface deposits while preserving the artifact's original structure. Unlike conventional methods, which may leave harmful residues or cause mechanical damage, cold plasma is a non-invasive technique that minimizes the risk of further deterioration [2]. It is important to note that certain metals, such as copper, can undergo corrosion without necessarily compromising their structural integrity. For instance, copper oxides form a stable, protective layer on the surface that does not require removal. In contrast, compounds like silver or copper chlorides and sulfides have destructive effects on metals, leading to severe degradation of artifacts. Therefore, their removal is essential to ensure the preservation of historical metal objects.

Plasma cleaning utilizes ionized gas to remove contaminants and oxides from the surface of various materials. This process is particularly valuable in the conservation of silver artifacts, as it effectively eliminates surface impurities while preserving the material's original structure. The technique involves exposing the sample to a low-pressure gaseous plasma, where reactive species—such as ions, radicals, and photons—interact with the surface, leading to the breakdown and removal of oxides and deposits [2].

A radio frequency (RF) power source is essential for generating and maintaining the plasma. In this process, RF power is applied to the gas inside a vacuum chamber, ionizing it and creating a plasma field. The frequency and power of the RF source can be adjusted to regulate the plasma's density and energy, ensuring optimal cleaning conditions. Additionally, the plasma generated by the RF source is rich in reactive species, such as hydrogen radicals, which efficiently remove metal oxides and help restore the artifact's original surface [3, 4].

Over the past two decades, researchers in various scientific fields have been exploring new and unconventional techniques for cleaning metal artifacts. Among these, high-frequency cold plasma has been recognized as an effective, non-damaging, and environmentally friendly method [5]. Extensive studies have examined the effects of Ar/O_2 and Ar/H_2 plasma on metals and their corrosion products [6, 7]. Additionally, research on H_2 plasma has demonstrated its high efficiency in removing silver chlorides and sulfides, which are particularly significant in artifact restoration [8-10]

Argon, a noble gas with a high atomic mass and well-known for its chemical inertness, plays a crucial role in plasma cleaning. When ionized, argon ions acquire high kinetic energy, allowing them to remove contaminants through sputtering. In this process, energetic plasma ions bombard the artifact's surface, dislodging oxide deposits and unwanted particles. To ensure optimal cleaning efficiency while preventing damage to the original surface, key parameters such as RF power and gas pressure must be carefully regulated [3, 11].

Oxygen plasma, which is naturally oxidizing, becomes even more effective as a cleaning agent when ionized. In the treatment of silver artifacts, it efficiently removes organic contaminants and reduces surface turbidity by oxidizing impurities into volatile compounds that can be easily eliminated [12]. During this process, atomic oxygen and ozone (O_3) react with surface contaminants, converting them into carbon dioxide (CO_2) and water vapor (H_2O), which are then removed [12, 13].

Hydrogen plasma, on the other hand, exhibits strong reducing properties, making it particularly effective in eliminating oxide layers. Hydrogen radicals facilitate reduction reactions, breaking down oxides without affecting the underlying metal. For example, in hydrogen plasma treatment, these radicals react with silver oxide (Ag_2O), converting it into metallic silver and water vapor, which is then removed from the surface. Due to its selective and non-invasive nature, hydrogen plasma is especially well-suited for cleaning delicate historical [4, 14].

Figure 1 is a schematic diagram illustrating the effects of plasma treatment on an ancient silver coin. The left panel depicts the corroded surface before treatment, while the right panel shows the restored surface after plasma exposure. Argon (

Ar^{+}) facilitates the removal of surface contaminants through sputtering, oxygen (O_2^{+}) breaks down organic matter and corrosion layers, and hydrogen (H_2^{+}) reduces oxides and chlorides. This combination ensures an effective yet non-damaging restoration process.

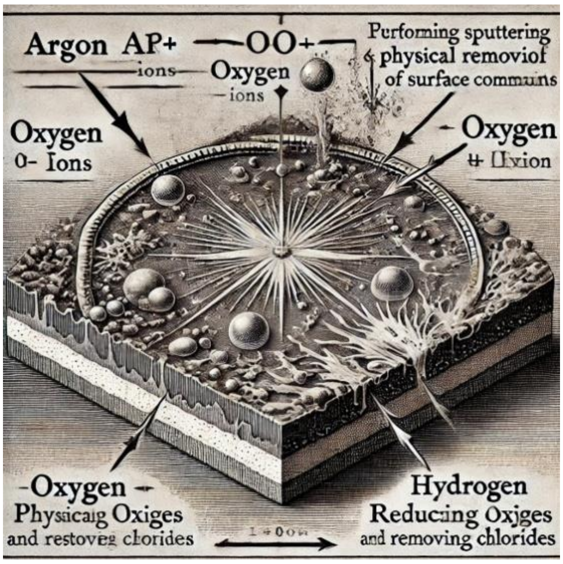

Fig.1. Schematic of the effects of argon, oxygen, and hydrogen gases on the surface.

Another advantage of RF plasma is its low operating temperature, which prevents metallographic alterations and thermal damage to artifacts [14]. Additionally, the RF power supply ensures uniform plasma distribution, allowing for consistent cleaning across the entire surface of the artifact.This study aims to assess the effects of a high-frequency, non-thermal plasma cleaning process on the surface of silver coins from the Parthian period, focusing on their chemical, physical, and visual properties.

Fig.2. The PECVD device used for conducting the experiments.

2. Material and Methods

2.1. Pre-Processing Analyses

2.1.1. X-ray Fluorescence (XRF) Analysis

In this study, X-ray fluorescence (XRF) spectrometry was conducted using a Niton XL3t GOLDD+950 (Thermo Scientific, US) to determine the elemental composition of the coins. XRF is a widely used non-destructive analytical technique, particularly valuable in cultural heritage and metallurgical research, for identifying the elemental makeup of materials. The process involves exposing the sample to a high-energy X-ray beam, which excites the atoms, causing them to emit secondary (fluorescent) X-rays unique to each element. These emitted X-rays are then detected and analyzed to determine the presence and relative abundance of elements within the sample.

XRF is particularly advantageous for examining historical metal artifacts, such as coins, as it allows for the identification of both major and trace elements without causing any damage to the artifact [6, 15].

2.1.2. SEM and EDAX Analysis

While improved spacial resolution is a major and clear advantage, one of the drawbacks of the energy dispersive x-ray spectroscopy (EDAX) technique is its failure in detecting light elements like hydrogen and lithium as well as its shallow penetration depth when compared with x-ray fluorescence (XRF) which on the other hand has a larger penetration depth. That is why the joint application of EDAX and XRF can provide a much more detailed analysis of chemical composition.

An electron microscope of the FE-SEM type was used for the examination of the elemental composition and the surface metals with the EDAX. The EDAX was a unique device by FEI ESEM QUANTA 200 and was equipped with an EDAX SILICON DRIFT 2017 detector. Besides the SEM microscopes and EDAX analyzer, the key equipment involved includes the ESEM and X-ray detector. Besides these, the FE-SEM images of metallic coins were chemical analyzed by the SEM-EDAX combo system. Various factors including its main characteristics denote SEM as one of the competence tools for the determination of microstructural status and elemental composition in historic coins.

Under an electron microscope, the reproduced image is a photomicrograph that displays a magnified and visible structure of a metal surface as well as the morphology of the particles. This instrument is the instrument of choice for the study of corrosion, microcracks, and surface modifications of aged metallic artifacts by SEM.

Besides the scanning microscopical examination, the powerful tool of energy dispersive X-ray analysis (EDAX) which is a modified system is used to analyze wave-length spectra of the corrosion and to confirm the presence of specific elements. SEM makes possible the high-resolution imaging of a focused electron beam over the sample surface, which is a method for the topographic and morphological study of samples.

This analytical method allows for in-depth study of corrosion, doping, and other surface modifications in metallic artifacts. Within this context, EDX facilitates the qualitative and quantitative identification of the metal content of a sample through atomic analysis.

While EDAX has superior spatial resolution advantages, light elements (specifically hydrogen and lithium) detection ability is restricted in comparison to XRF and its penetration depth is not that deep either. Because of this, the application of both EDAX and XRF can supply a more globally rich set of information on the composition of samples.

Subsequently, during the study of the elemental composition and surface metals in the field and the research of the artifacts involved, field-emission scanning electron microscopy (FE-SEM) imaging was accompanied by energy-dispersive X-ray spectroscopy (EDAX) using an FEI ESEM Quanta 200 scanner equipped with an EDAX Silicon Drift 2017 detector.

SEM and EDAX are the fundamental methods to examine the microstructure and elemental composition of metallic artifacts, including historical coins.

One of the merits of EDAX over XRF is its capability of localized surface study, which also makes it more apt for corrosion products, contaminants, and heterogeneous structures in historical coins and other metal artifacts [6, 7]. Also, it should be noted that, in opposition to XRF which can provide elemental information in the bulk and the volume, EDAX only performs surface analyses at particular locations and, therefore, it is suitable for the study of corrosion products, contaminants, and the development of multiple phases in the case of historical coins and other metal artifacts. However, for all of this, although the resolution is usually higher, the limit is a shallow penetration depth and a less efficient response to light elements (e.g., hydrogen, lithium) comparing with XRF. Both EDAX and XRF together thus help us to have a better and complete understanding of the chemical composition of the samples.

2.2. Plasma configuration

Because the samples were highly sensitive to the temperature, in this study cold plasma under vacuum with RF power supply was employed. The device was configured to run at a frequency of 13.56 MHz with process parameters of 100 W applied power at 20 mTorr, and a total gas flow rate of 30 SCCM. Plasma processing device image shown in figure 2. Based on the type of gas used, the duration of treatment was modified (90 min Ar/O_2, 60 min Ar/H_2)

2.3. Coin Weight Measurement

Weight of the coins was assessed on a digital balance of 0.0001 g accuracy, and each measurement was repeated three times for verification.

2.4. Imaging of the Samples

All the images of the coins were taken under regular light with a Canon EOS 2000D camera. Further, fine surface details were examined by means of one of the ZEISS Stemi 305 microscopes recording images at up to ×70 magnification.

3. Discussion

3.1. EDAX Analysis and FE-SEM Images

A considerable variation in relative surface morphology was exhibited in the analyzed coin samples as observed from the FE-SEM imaging shown in Fig. 2.

As shown in Sample C8, its surface has a rough and uneven texture while the corrosion layer can also be clearly seen. The corrosion mechanism that occurred came from oxidation as well as chloride corrosion, which usually happens on buried copper-silver coins in soil. As mentioned earlier, the bright areas in SEM images illustrate silver (Ag), lead (Pb), and gold (Au), which were approved by EDAX examination

On the other hand, sample C24 shows a more sophisticated surface structure with a higher load of deposits than C8. Cracks and pits can also be observed on the patent surface of this sample, indicating a higher level of localized corrosion. The bright areas of the SEM image represent a higher concentration of heavy elements, and this coin had more silver (9.9%) and lead (1.73%); in fact, elements more massive than gold (and lead).

Elements analysis for samples was performed to study the elemental composition, as presented in Figure 2, which showed the presence of 23.28% O in C8 and 25.74% O in C24, indicating a high oxidation process occurring in both samples. Cu_2O was found, in addition to various unknowns, and chlorine (Cl) at 4.31% in sample C8, which likely indicates the evolution of copper chloride (CuCl) compounds. This compound can quickly lead to what is known as "bronze disease," a weak copper-chloride complex that is not only unstable but is also a form of corrosion capable of doing considerable damage. Under the presence of oxygen and moisture, these compounds will be oxidized to form cuprous oxide (Cu_2O) and hydrochloric acid (HCl), which results in the continuous corrosion of the metal surface [16].

In contrast, sample C24 contains less chlorine (2.23%) but more silver (9.9%) and lead (1.73%), which could be indicative of a different alloy composition or limited impact by chloride corrosion.

Corrosion of these ancient coins mainly involves oxidation, chloride corrosion, and environmental deposits. The high Pb content found in ancient coins may be the result of traditional Pb smelting techniques used for extracting silver from its ore, as indicated by past studies such as [15].

Environmental aspects on these coins have been clearly demonstrated through SEM images and EDAX data. The high concentrations of silica (Si) and calcium (Ca) in both samples indicate prolonged exposure to soil mineral deposits over long periods of burial. Sample C24 has higher levels of silica (11.27%) and oxygen (25.74%), which would point to it having been buried in a silicate-rich environment, where a natural protective layer may have formed. In contrast, the chloride content of sample C8 (4.31%) indicates a more corrosive environment, resulting in severe chloride-induced corrosion.

The presence of minor groups like gold (Au: 1.64% in C24, 0.51% in C8) and bismuth (Bi: 1.95% in C24, 0.56% in C8) may be the result of incidental amalgamation during the alloy preparation procedure or remnants in the silver and copper lodes employed when minting coins [17]. Over long periods, mineral compounds like silicates and carbonates can deposit on the surface of the metal, sometimes creating protective coatings. The high percentage of Si (11.27%) and Ca (4.3%) in sample C24 further supports this hypothesis since it implies that a mineral layer forming over the surface of the coin could have acted as a barrier and protected the coin from additional corrosion [18].

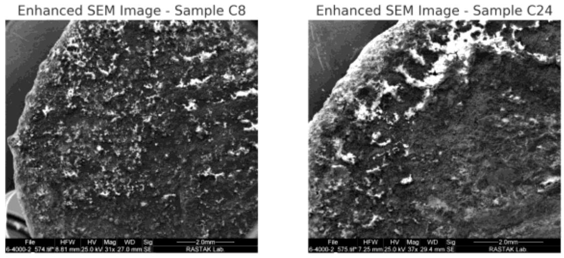

Fig.3. Scanning electron microscope images before plasma treatment

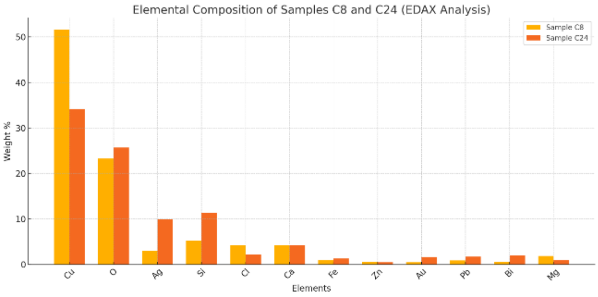

Fig.4. EDAX results

3.2. XRF Analysis

As shown in Table 1, copper (Cu) constitutes the highest percentage of the surface composition in both analysis iterations, suggesting the presence of copper oxides. The greenish coloration observed on the samples further supports this assumption. In sample C24, XRF results indicate that approximately 53% of the surface is composed of copper, while EDAX reports a lower percentage. Given the high oxygen (O) content detected in EDAX, it can be inferred that metal oxides and external deposits contributed to this discrepancy.

Table.1. XRF results in terms of weight percentage of constituent elements

Sample	Si	Si	Ti	V	Fe	Cu	Ag	Sn	Sb	Au	Pb	Bi
C8	3.575 ±0.415	3.575 ±0.415	0.105 ±0.011	0.0515 ±0.002	0.377 ±0.002	49.125 ±0.835	44.51 ±0.6	0.4195 ±0.045	0.0475 ±0.003	0.152 ±0.001	0.9305 ±0.129	0.0215 ±0.004
C24	8.5 ±0.8	8.5 ±0.8	0.116 ±0.005	0.0615 ±0.002	–	52.755 ±0.525	34.41 ±0.73	0.7615 ±0.002	0.02 ±0.005	0.114 ±0.001	0.213 ±0.014	0.0735 ±0.011

The higher concentration of silver (Ag) in XRF compared to EDAX suggests that silver is located in the deeper layers of the coin, with surface oxides and accumulated deposits obscuring its detection in EDAX. Similarly, in sample C8, XRF analysis reported a higher silver content than EDAX, which can be attributed to the greater penetration depth of XRF beams. Since XRF penetrates deeper into the sample structure, it is able to detect silver beneath oxide and corrosion layers, whereas EDAX primarily analyzes surface compounds. Furthermore, the higher chlorine (Cl) content in EDAX than in XRF for sample C8 indicates that copper chlorides are concentrated on the surface. This suggests that chloride corrosion, specifically Bronze Disease, may have developed as a result of prolonged exposure to chloride-containing environments. In sample C24, a similar discrepancy between XRF and EDAX results was observed, but with a different silver-to-copper ratio. The lower silver content detected in EDAX compared to XRF suggests the presence of a thicker layer of surface oxides and deposits, which likely reduced silver detection in surface-level EDAX analysis. Additionally, the detection of silica (Si) and calcium (Ca) in EDAX indicates that sample C24 was buried in a silicate-rich environment, leading to the deposition of soil mineral compounds on its surface. The lower chlorine (Cl) content in C24 compared to C8 suggests less chloride-induced corrosion and the formation of protective oxide layers, which likely helped slow the rate of surface degradation over time.

3.3. Imaging of the Samples

As mentioned earlier, the coin samples were imaged using a Canon EOS 2000D camera under standard lighting conditions. Due to the low purity of ancient coins, their composition includes a mixture of silver (Ag), zinc (Zn), copper (Cu), and lead (Pb), forming a significant part of their structure.

The green coloration observed in the images (Figures 5A and 6A) likely indicates the presence of malachite ($Cu_2(OH)_2CO_3$) and copper hydroxide chloride ($Cu_2(OH)_3Cl$) on the surface. Additionally, the reddish-brown hue is most likely due to cuprite (Cu_2O), suggesting oxidation and corrosion processes affecting the coins.

In Figure 5B, a comparison with image A shows that malachite, copper hydroxide chloride, and reddish-brown corrosion layers have been largely removed. This cleaning process has enhanced the clarity of engraved patterns on the coin. For instance, finer details, such as the helmet and the king's eye, have become more distinguishable, demonstrating the effectiveness of plasma treatment in removing deposits and improving the coin’s readability.

Figure 6 presents images of sample C24 before (A) and after (B) plasma treatment. In image A, the presence of copper oxides (Cu_2O, CuO), copper chlorides ($CuCl$, $CuCl_2$), and copper carbonates is evident, corroborating the EDAX and XRF data, which detected high levels of oxygen and foreign elements in the surface composition.

The green and blue coloration observed in Figures 5 and 6 most likely indicates the presence of malachite ($Cu_2(OH)_2CO_3$) or azurite ($Cu_3(CO_3)_2(OH)_2$). These compounds are common corrosion products in copper-silver coins, forming in humid or salt-rich environments, which accelerate the corrosion process.

Following cold plasma treatment under vacuum using Ar/O_2 or Ar/H_2 gases, portions of the corrosion layers were successfully removed, revealing finer details of the engravings. However, as shown in images B of Figures 5 and 6, while the plasma treatment effectively reduced the oxide and corrosion layers, some residues remain on the coin's surface

Fig.5. Figures a and b before and after plasma treatment for sample C8

Fig.6. Figures a and b before and after plasma treatment for sample C8

3.4. Microscopic Image Analysis of C8 and C24 Coins

The analysis of microscopic images of C8 and C24 coins before (A) and after (B) plasma treatment reveals significant differences in the degree of corrosion and type of surface deposits. Before treatment, sample C8 (A) exhibited non-uniform deposits, significant surface darkening, and faint, indistinct engravings, suggesting the presence of silver sulfide (Ag_2S) and copper chloride (CuCl) on its surface. In contrast, sample C24 (A) had thicker and more uniform deposits, with white mineral and oxide residues visible in certain areas. These residues indicate the presence of silver oxides (Ag_2O), copper oxides (Cu_2O), and other mineral compounds. The differences between the two samples are likely due to the different environmental conditions in which they were stored.

After plasma treatment (B), both coins demonstrated significant improvements in surface clarity and deposit removal, though the extent of these changes varied between the samples. In sample C8 (B), the engraved patterns became more distinct, and the darker surface areas were reduced, indicating that plasma processing effectively removed surface deposits. These changes highlight the high efficiency of hydrogen (H_2) plasma in eliminating chloride and sulfide compounds.

In contrast, sample C24 (B) exhibited noticeable improvements in some regions, but thicker deposits remained in certain areas. This is likely due to the presence of more substantial oxide and mineral layers, which may require longer processing times or an alternative gas mixture for complete removal. A comparison between the two samples suggests that H_2 plasma was highly effective in eliminating copper and silver chlorides (AgCl, CuCl), as evidenced by the greater improvement in C8 after treatment. Meanwhile, argon-oxygen (Ar/O_2) plasma appeared to be more effective in removing oxides and mineral deposits, as observed in sample C24. Overall, C8 exhibited a higher rate of deposit removal, whereas C24 retained some surface compounds, indicating that it may require further processing. These findings confirm the effectiveness of low-pressure plasma treatment in enhancing surface clarity and selectively removing unwanted compounds from historical coins without damaging their original metal structure.

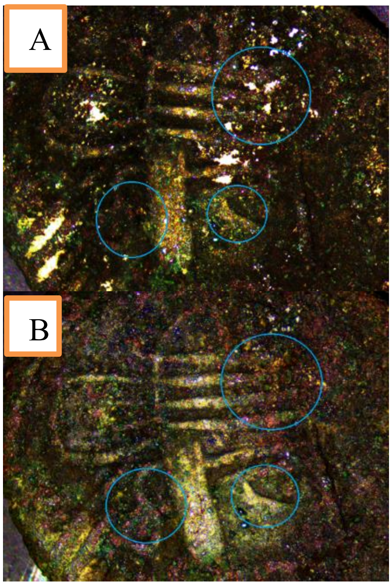

Fig.7. Microscopic images of the coin surface: images a and b before and after plasma, respectively, for sample C8.

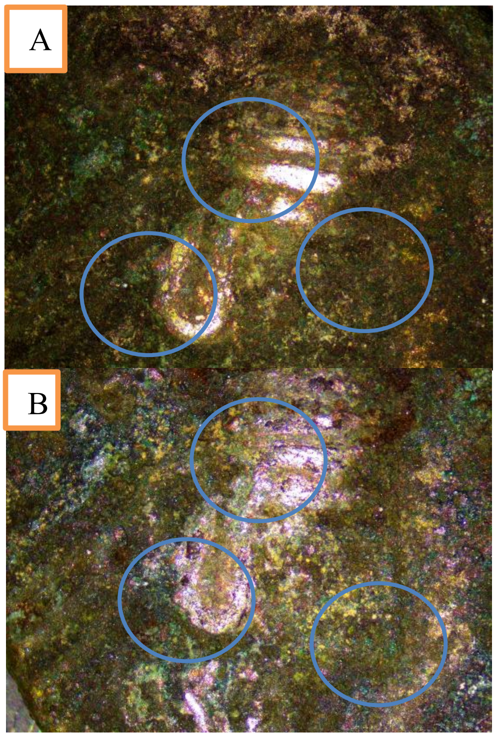

Fig.8. Microscopic images of the coin surface: images a and b before and after plasma, respectively, for sample C24.

3.5. Coin Weight Measurement

A comparison of the weights of C8 and C24 coins before and after plasma processing indicates that this method effectively reduces mass by eliminating surface deposits and corrosion compounds. Sample C8 initially weighed 3.721 g before treatment, and after plasma processing, its weight decreased to 3.7012 g, reflecting a mass reduction of 0.0198 g. This loss confirms the significant removal of surface deposits, particularly chloride (CuCl, AgCl) and sulfide (Ag_2S) compounds, as previously confirmed by SEM/EDAX and XRF analyses.

Conversely, sample C24 exhibited a lower weight loss of 0.012 g, suggesting that its corrosion layers were denser and more strongly bound to the surface. This difference implies that C24's oxide layers were thicker and more resistant, requiring a longer plasma exposure time or a modified gas composition for complete removal.

The mass reduction observed after plasma treatment, without any damage to the base metal, highlights the selective removal of corrosion compounds while preserving the artifact's original structure. Compared to chemical cleaning methods, which may dissolve beneficial metal compounds and alter surface integrity, plasma cleaning provides a controlled and non-invasive approach that removes only the destructive layers while maintaining the original texture of the metal.

The greater weight reduction in C8 suggests that hydrogen (H_2) plasma was more effective in eliminating surface chlorides and sulfides, whereas C24, which likely contained thicker oxide compounds (such as Cu_2O and Ag_2O), responded better to Ar/O_2 plasma treatment. These findings confirm that low-pressure RF plasma is a safe and efficient method for removing surface corrosion from historical metal artifacts, preventing unnecessary mass loss while ensuring artifact preservation.

Sample	Initial weight (g)	After 90 min Ar/O_2 (g)	After 90 min Ar/O_2 (g)	After 60 min Ar/H_2 (g
C8	0.0008 ±3.701	0.0125 ±3.715	0.0105 ±3.721	0.0008 ±3.701
C24	0.0031 ±3.813	0.0016 ±3.820	0.0085 ±3.825	0.0031 ±3.813

Table.2. Average weight of coins in grams

4. Conclusion

This study highlights the effectiveness of low-pressure RF plasma with Ar/O2 and Ar/H2 gas mixtures as a non-invasive method for cleaning historical silver coins. By removing surface deposits, oxides and chlorides, this method offers a reliable alternative to traditional cleaning methods. Prior to plasma treatment, SEM/EDAX and XRF analyses provided valuable insights into the surface composition of the coins and helped to assess the impact of corrosion. The results showed high levels of oxygen and chloride, indicating significant corrosion effects. Interestingly, XRF analysis detected a higher concentration of silver compared to EDAX, likely due to its greater penetration depth, which allows it to detect silver beneath oxide and chloride layers. This suggests that surface corrosion can mask the true composition of the metal in EDAX analysis. Additionally, the presence of lead (Pb) and bismuth (Bi) in the samples supports the idea that the coins were refined using traditional lead-based metallurgical methods.

Gravimetric analysis after plasma treatment confirmed its effectiveness, showing partial weight losses of 0.0198 g in sample C8 and 0.012 g in sample C24. The minimal material loss indicates that the method is non-destructive and preserves the underlying metal structure.

Microscopic imaging before treatment revealed rough surfaces with corrosion deposits, including copper and silver sulfide (Ag2S) compounds, which is consistent with previous research.

The results further showed that the Ar/O2 plasma is particularly effective in removing copper oxides and organic contaminants, while the H2 plasma is superior in reducing chloride compounds.

Overall, this study confirms that low-pressure RF plasma is a safe and efficient cleaning method for historical silver coins, which has a major advantage over conventional chemical and mechanical techniques. It selectively removes corrosion without damaging the original metal, making it an excellent option for the restoration of historical artifacts. Future research should focus on optimizing plasma parameters to improve cleaning efficiency and investigating the long-term effects of plasma treatment on the stability and preservation of historical silver coins.

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