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Copper Black Coatings for the Absorption of Solar Concentration With an APPJ SiO2 Super-Hydrophobic Protection:

Selective Copper Black Coatings for Solar Power

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ABSTRACT

Solar thermal energy can be captured on absorbent surfaces, but coatings tend to deteriorate, due to changes in hue, thermal shocks, or detachment of all layers. There is a great challenge in reducing the deterioration because of environmental factors such as corrosion, impact, and dust control, among others. The absorbent coatings interact with the incident solar radiation transforming it as heat energy, and selectivity allows a low emissivity. In this work, a three-layer system on copper is proposed. An anodized CuO or black copper layer as an absorbent with high absorptance is proposed. A protective layer was added to extend the lifetime of use while preserving the functional characteristics of the absorbent black layer by using SiO2 deposited by atmospheric pressure plasma jet (APPJ) using hexamethyldisiloxane. A selective layer of aluminum was deposited by physical vapor deposition (PVD). Thermal shocks were applied by concentrated solar power with a Fresnel lens to represent sudden temperature changes in the radiation absorbent when the weather changes.

KEYWORDS

Aluminum, Anodization, Black Copper, Central Tower, Concentrated Solar Power, CuO, Parabolic Trough, PVD, Selective Layer

INTRODUCTION

For many years, our consumer society globalization has contributed to intensifying, has demanded products in quantity, quality, and variety, accelerating both a state of dissatisfaction, health concerns, and environmental pollution with no thought given to improving living conditions. There has been

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a formidable industrial development, which has been growing in installed facilities for the synthesis and manufacture of products that meet various needs. Today, new processes and new products are in the markets to satisfy new needs developed every day, which will test the creativity, innovation, science, and technology, but going to endanger ecosystems, human health, and doubtless going to stress every single person now and in each generation to come.

Given this perspective, in 1997 the governments agreed on the Kyoto Protocol to the UN Framework Convention on Climate Change (UNFCCC), which sets legally binding targets so that, during the 2008-2012 period, industrialized countries reduce 5.2%, on the 1990 levels, the emissions of the main greenhouse gases. There are alternative energy sources, such as sun, wind, wastes, etc., which are renewable every year, do not run out, and also do not pollute the environment, which means a double advantage.

Solar energy is very attractive because it is clean, renewable, affordable, and with lower impact on the environment (Liu, 2016; Carrillo, 2019). The reduction of gases is an advantage of using solar energy. Solar thermal collectors are environmentally friendly and do not pollute as a way to collect energy. The efficiencies of solar thermal collectors have been improved and its absorbent systems or coatings increased the operating and inactivation temperatures (Bagmanci, 2019; Wu, 2019).

Further development of collectors and new applications of solar energy, such as refrigeration and industrial solar heating, are raising the operating temperatures of absorbent materials even further. Since the first collectors, absorbing solar coatings actively have been developing, using advanced designs with improved efficiencies (Bagmanci, 2019).

The degradation processes in solar absorbers are caused by high temperatures, high humidity, water condensation, chlorine, and sulfide. In the case of moisture, corrosion mechanisms have been reported in absorbent coatings on aluminum substrates (Kotilainen, 2014).

The selective coatings are used on black absorbent surfaces to reduce radiative energy losses (Lizama-Tzec, 2019; Thappa, 2020). Such selective coatings require to have a low emittance ε (<0.2) of infrared wavelengths that, combined with the black surfaces that have high solar absorbance α (> 0.9) together, reach higher solar energy harvesting efficiencies. Super absorbent black coatings help us to have better absorption and concentration of solar energy for the different areas where it is intended to be used. The parabolic trough collectors are systems that require selective black absorbing surfaces that receive and transfer the energy (Tzivanidis, 2015).

BACKGROUND

A solar absorbent surface fulfills with requirements such as a high solar absorptance (α) and low thermal emittance (ε) to absorb the highest and re-radiate the lower amount of energy from the sun, respectively. Frequently, for materials such as metals, a high thermal emittance is directly related to high electrical conductivity, such as metals or graphene, but this characteristic is not necessary for this application.

Prasanth *et al.* (2019) reported CuO thin films deposited by Reactive Direct Current Magnetron Sputtering of a Cu target. Their optimized films were CuO with α and ε of 0.955 and 0.52, respectively. An additional useful finding was that the emissivity was increased to 0.78 after sandblasting of the substrate, which implies that a high roughness is related to both a higher diffuse reflectance and higher emittance that is undesirable. This is related to an increment in the area and an uneven reflection.

The short lifetime of absorbent surfaces is one of the difficulties in capturing solar energy for various applications. The deterioration of the multi-layer absorbent surfaces is observed as changes in hue or detachment of one or all layers. Among the causes of this and other difficulties are the environmental ones, such as corrosion, hail and rain impacts, dust, stress-strain caused by wind, etc. Another factor is the temperature since concentrated solar power (CSP) implies for parabolic trough about 250 °C and central tower above 500 °C. This causes thermal shock failures since, even for a single day, there are changes in weather conditions, but in the year term, sudden cloudiness or cloudburst occur.

This work demonstrates the preparation of black copper coatings for absorption in solar concentration with the selectivity of reflection in the medium infrared and protection against environmental factors and use.

OBJECTIVES

General Objective

Obtaining super-absorptive black coatings for solar concentration with mid-infrared reflection selectivity and protection against environmental and usage factors.

Specific Objectives

1. Obtaining CuO black copper layer as an absorbent material for concentrated solar energy by anodizing the copper substrate;
2. Obtaining a silica layer on the black copper by the atmospheric pressure plasma jet (APPJ) technique as a protective layer;
3. Obtaining an aluminum top layer for providing spectral selectivity by reflecting the infrared radiation;
4. Test the three-layered surface under thermal shocks induced by concentrated solar energy using a Fresnel lens.

METHODOLOGY

Hypothesis

A three-layer coating consisting of a CuO black copper, SiO_2 by APPJ, and an Aluminum layer by PVD could confer both, the properties of absorptance avoiding the re-emission of mid-infrared radiation, and reducing the deterioration of the absorbent surface.

The sample preparation was according to the process shown in Figure 1. As a first step, the preparation of solutions and passivation of copper plates and tubes was carried out. The plates were coated with silica for protection using the Atmospheric Pressure Plasma Jet (APPJ) technique, a layer of aluminum as a selective layer by Physical Vapor Deposition (PVD). Then, a thermal shock was applied with a Fresnel lens by Concentrated Solar Power (CSP) under ambient conditions. The characterization includes Scanning Electron Microscopy (SEM) with a chemical composition image by Energy Dispersive X-ray Spectroscopy (EDS). Surface images were taken with a Digital Optical Microscope. A spectrometer was used to measure reflectance.

Figure 2a shows the material tubes that were used to make the black surfaces by electro-passivation, which consisted of rough and polished copper tubes of 15x2x0.1 cm. These electrodes were polishing with 0.5 μm alumina powder. The anodization was carried out in an alkaline aqueous admixture of 15 g/L of NaOH and 50 g/L of Na_2CO_3 (Montero, 1989), Figure 2b. A current of 1.2 A/cm^2 was applied to the electrodes for 2 min (Figure 2c).

The copper pieces were anodized in a solution of 1 L of deionized water with 1M sodium hydroxide. The temperature was varied between 57 and 60 °C, the current density was about 0.06 A/cm^2 and the time was 20 to 25 min. These conditions lead to an electrochemical oxidation efficiency of about 90%. Black surfaces were cleaned with deionized water.

The alkaline solution (NaOH and Na_2CO_3) for anodization was used three to four times before its replacement or re-conditioning because there were no noticeable changes between each anodization. On the other hand, the solution only with sodium hydroxide could be used for a maximum of three

Figure 1. Process steps used to prepare black copper tubes/plates

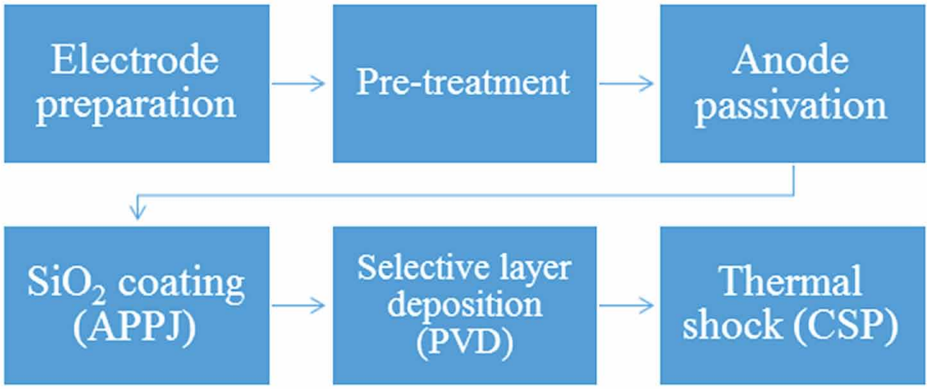
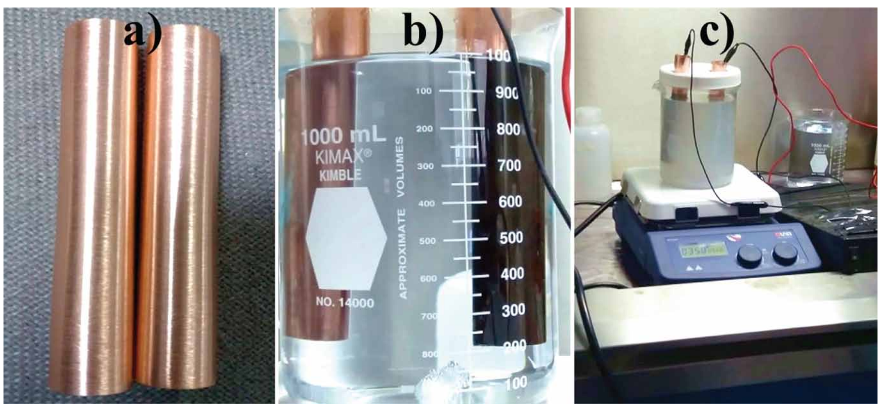


Figure 2. a) A pair of copper tubes polished with alumina; b) Cathode on the left and anode on the right, with a black surface, inside an alkaline solution; c) Heating and stirring during the anodizing of copper tubes



times, which was mainly because of water evaporation, consequently, reducing the height of the anodized black surfaces (Figure 3a,b,c).

A selective layer of aluminum was applied to the black surfaces by the physical vapor deposition technique, PVD (Selvakumar, 2012; Valleti, 2016; Gensowski, 2019). Two deposition times were used, 2 and 5 min, providing metallic layers that reflect infrared and transmit visible and UV, with thicknesses between 20 and 30 µm (Figure 4a). A silica film was applied to some samples by the atmospheric pressure plasma jet technique, APPJ (Hilt, 2016; Darny, 2017; Hossain, 2019; Teske, 2019), seeking a layer that preserves the characteristics of the black selective surfaces (Figure 4b). Hexamethyldisiloxane reagent was used as a silica precursor (Boutamine, 2014; Gilliam, 2019; Saloum, 2019; Sifuentes-Nieves, 2019; Trinh, 2019). The APPJ nozzle was on a robotic arm and the coatings were homogeneous on small and big areas. Also, more than a single layer may be required to obtain the desired properties. The elapsed time may take about tens of minutes depending in the arm speed, the width of the deposition line, and the overlap of consecutive lines to cover an area.

The above plates were heat treated with a thermal shock by the Fresnel lens at a maximum recorded temperature of 250 °C (Figure 4a). The experimental assembly with a Fresnel lens of poly (methyl methacrylate) PMMA, with a diameter of 1 m and a focal length of 1.3 m, registered a maximum of about 1200 °C, of which only a temperature of 250 °C was used or lower for heat

Figure 3. a) Copper tubes with different surface finishing caused by a less or longer anodizing time; b) Blackened surfaces after rinsing with deionized water; and c) Final finishing of black copper tubes. The decrease in the water level caused the difference in height between the pipes of the black section.

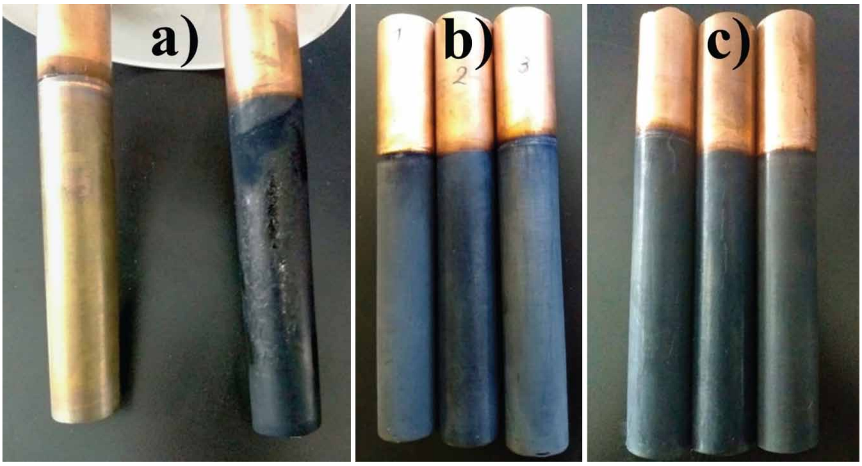
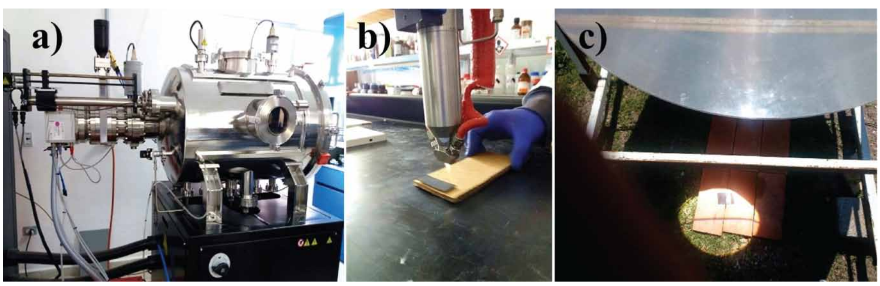


Figure 4. a) PVD setup for the selective coating using aluminum; b) APPJ coating with a protective silica layer; and c) Thermal shocks as heat treatment to the black surfaces using a PMMA Fresnel lens



shock treatment of copper samples. The heating, annealing, and cooling times were about 2 min, 5 min, and 8 min, respectively.

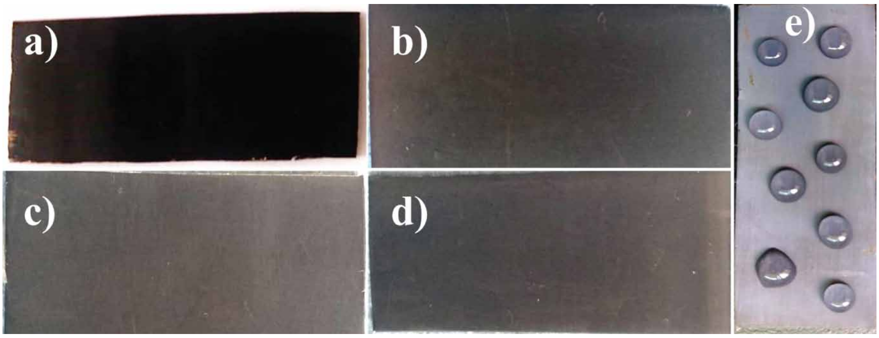
RESULTS AND DISCUSSION

Figure 5 shows a set of plates bearing different finishing. Figure 5a represents this kind of samples with a black anodizing. Figure 5b shows a copper plate, anodized and coated with silica, which was heat-treated under a concentrated beam of sunlight. Figures 5c and 5d show copper plates, anodized and coated with silica, having a thin aluminum layer (10-30 µm) with a deposition time of 2 and 5 min, respectively.

The aluminum top layer was intended for reducing the loss of heat by radiative energy. The thermal shock was proposed to represent a test, in a short period, of the behavior of the black surfaces in environmental conditions once placed on service at the field.

Complementarily to the silica by APPJ layer, a hydrophobic SiO_2-TiO_2 layer was deposited by the same technique using tetraethyl orthosilicate (TEOS) and titanium(IV) isopropoxide as silica and titania precursors, respectively. The purpose was to reduce the wettability to avoid both irregular changes in the surface caused by corrosion, dirt stains, or dust particles. Nonetheless, the

Figure 5. a) A copper plate with a black anodized finishing; b) Copper plate after a thermal shock treatment (<250 °C) having a black anodized layer and a silica coating; c) Plate with a top aluminum selective layer with 2 min of PVD; d) Plate with a top aluminum selective layer with 5 min of PVD; and e) Hydrophobic SiO_2-TiO_2 layer deposited by APPJ on a black anodizing layer above a copper plate



samples required fifteen layers to show the effect. The effect was obtained when particles, hundreds of nanometer in size, were on the surface reducing the contact of the water drops with the surface (Figure 5e). Even when this characteristic on the surface reduces such factors, the black finishing or solar radiation absorption decreases because the surface has a white finishing or optically dispersive reflection instead of high absorptance.

Figure 6 shows the micrographs of the coatings and its different layers using a Digital Optical Microscope, Keyence VHX-5000.

Figure 6a shows a micrograph of the copper oxide layer on a copper substrate. The darker parts were attributed to the deposited of a thicker layer. On the other hand, the more grayish color was attributed to a uniform coating. The iridescence was caused by light interference from reflections on the frontal and inner surfaces of the particles.

Figure 6. Micrographs of the plates shown in Figure 5a,b,c,d took using a Digital Optical Microscope. The blue scale bar above the level represents 1 μm.

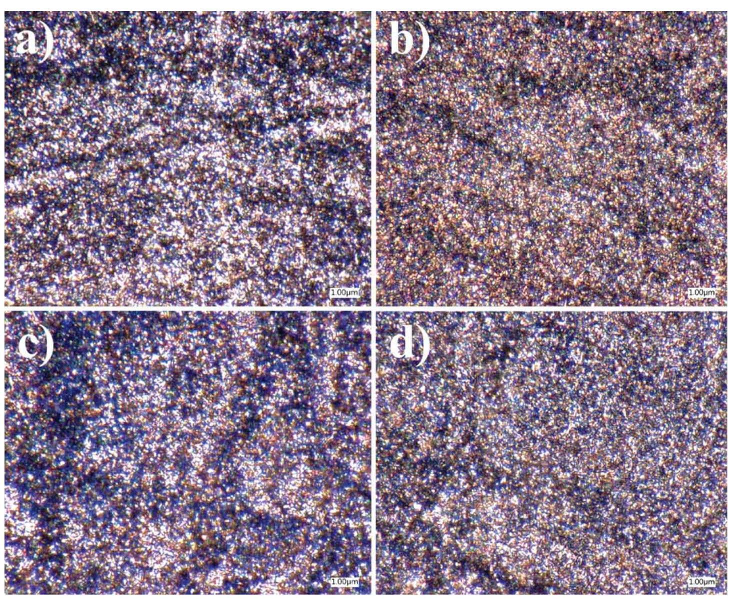


Figure 6b shows a micrograph with a CuO layer and a silica film. There was a light yellowish tone caused by the silica layer. Better uniformity of the top coating was achieved with the film applied on the substrate.

Figure 6c and Figure 6d show the last two aluminum covered plates, which was deposited as a selective layer, varying the time c) 2 min and d) 5 min. There was a difference that optically can be seen as a whitish appearance of the thicker layer using a deposition time of 5 min. There was a low content of aluminum on top of the samples in the case of the deposition time of 2 min.

Figure 7 shows the optical profilometry of Figure 5a,b,c,d samples. The reddish sections are the picks and the bluish are the valleys. a) Copper plate with a black anodized finishing, b) copper plate after a thermal shock treatment (<250 °C) having a black anodized layer and a silica coating, c) plate with a top aluminum selective layer with 2 min of PVD, and d) plate with a top aluminum selective layer with 5 min of PVD.

Figures 8, 9, and 10 show the scanning electron microscopy (SEM) micrographs of CuO oxide layer, silica on top or aluminum PVD deposition, respectively.

Figure 8 shows the morphology of a copper plate with black anodized finishing. The EDS graph shows the amount of copper relative to other elements. Carbon and oxygen are attributed to the substances that were used for anodizing. The silica was attributed to possible contamination of the plate. In the mapping, the amount of copper appears on the plate with a reddish hue. The most prominent particles are proposed to be due to the contamination of silica on the plate.

Figure 9 shows the same morphology as in Figure 8 but with a silica film. The mapping shows that the reddish hue is due to the copper coating and the dark particles are attributed to the silica film applied to it. This silica deposition was as separate particles instead of a continuous film, which depends on the deposition parameters such as nozzle scan speed, the distance between the substrate and the nozzle, air pressure in the nozzle, nozzle aperture size, and silica precursor.

Figure 10 shows the same morphology as in Figures 8 and 9 but with a 2 min deposition of aluminum using PVD. The thickness was not enough to see the aluminum layer using SEM. PVD deposition time is a key parameter that requires to be in a narrow range because a thickness of about 35 nm starts to provide a mirror-like finishing, reflecting visible wavelengths. On the other hand,

Figure 7. Digital optical microscope images showing the profilometry of the four samples: a) Copper plate with a black anodized finishing; b) Copper plate after a thermal shock treatment (<250 °C) having a black anodized layer and a silica coating; c) Plate with a top aluminum selective layer with 2 min of PVD; and d) Plate with a top aluminum selective layer with 5 min of PVD

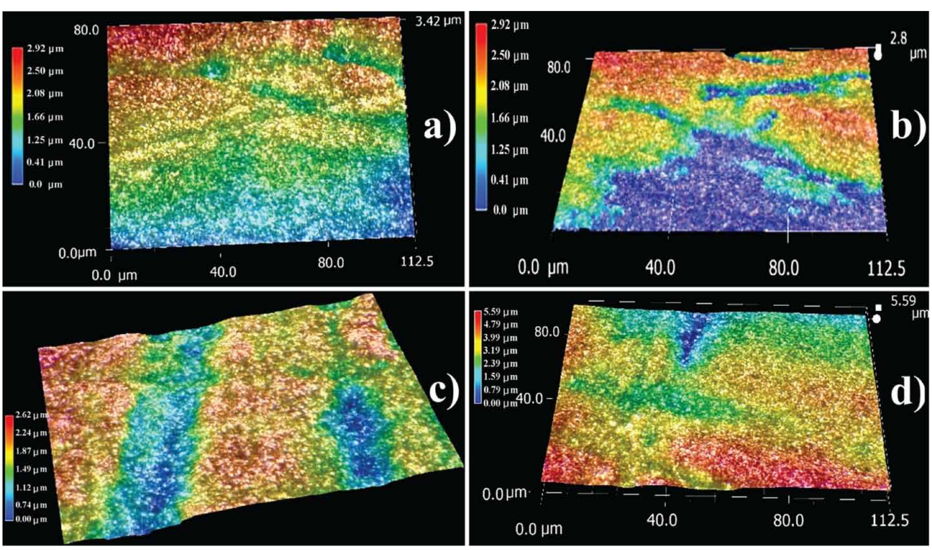


Figure 8. SEM images and EDS chemical composition mapping of a copper plate with black anodized finishing. a) Main micrograph (scale bar 10 μm); b) Zoomed in image (scale bar 5 μm); c) Spectrum of the chemical analysis showing mainly copper; d) Main micrograph for the chemical mapping (scale bar 10 μm); e) Silicon distribution on the surface; and f) Overlapping of the pair of previous images

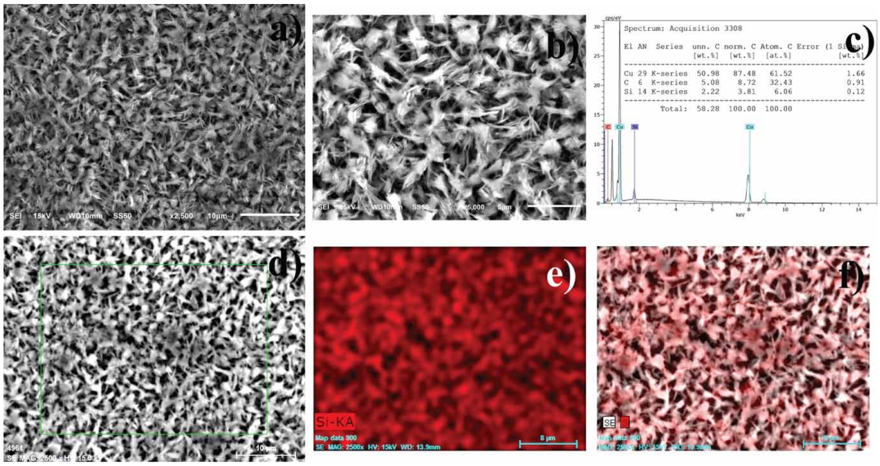
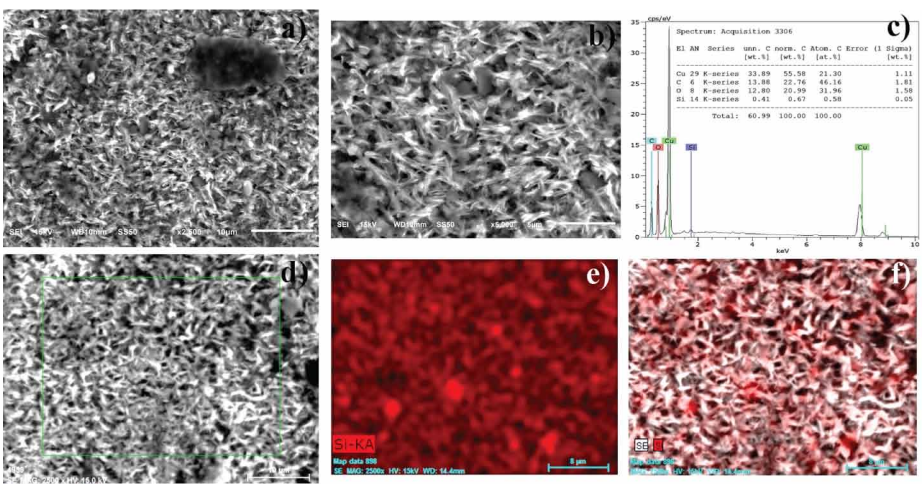


Figure 9. SEM images and EDS chemical composition mapping of a copper plate with a black anodizing and a silica layer by APPJ. a) Main micrograph showing silica particles (scale bar 10 μm); b) Zoomed in image showing smaller silica particles (scale bar 5 μm); c) Spectrum of the chemical analysis showing mainly copper-oxygen-silicon; d) Main micrograph for the chemical mapping (scale bar 10 μm); e) Silicon distribution on the surface; and f) Overlapping of both previous images.



short time depositions are not enough to reflect infrared wavelength, which was desirable for this case allowing the loss of energy by irradiation from the absorbing black surface. Then, the results show homogeneous PVD coatings but without a mirror-like finishing with a thickness of about 30 nm.

The Fourier transform infrared spectroscopy FT-IR was used to measure the reflectance as an approach to study whether the selectivity layer was reflecting infrared wavelengths. The coating on top of the black CuO layers was not enough to show a significant infrared reflection. Thicker layers were not testes because they gave a mirror finishing.

Figure 10. SEM images and EDS chemical composition mapping of a copper plate with black anodized finishing and an aluminum deposition by PVD of 2 min. a) Main micrograph (scale bar 10 μm); b) Zoomed in image (scale bar 5 μm); c) Spectrum of the chemical analysis showing mainly copper without aluminum; d) Main micrograph for the chemical mapping (scale bar 10 μm); e) Silicon distribution on the surface; and f) Overlapping of the pair of previous images.

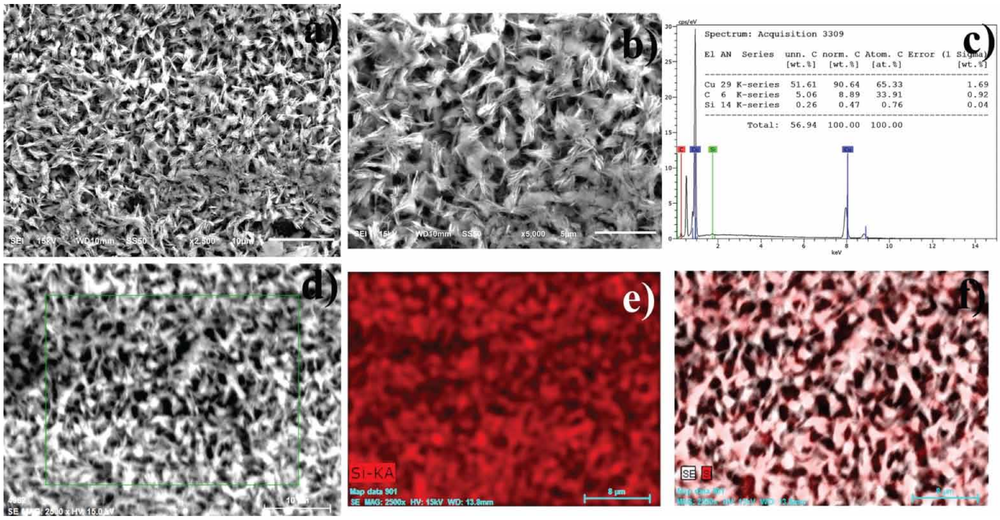
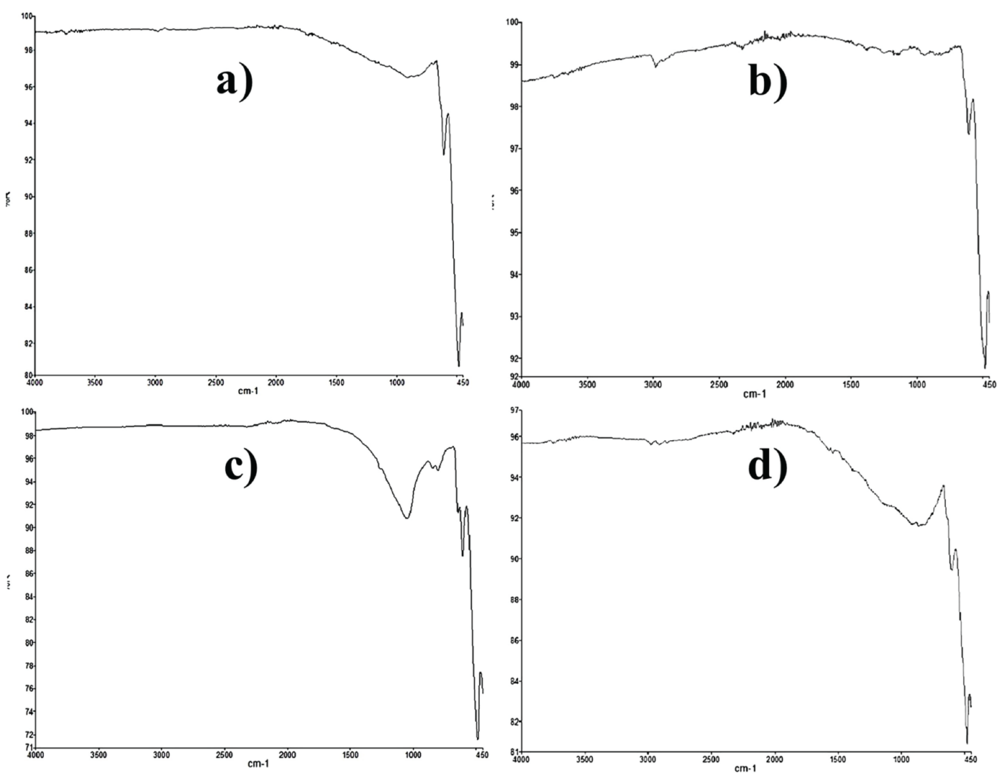


Figure 11. The Fourier transform infrared spectroscopy FT-IR of the four types of analyzed samples: a) Copper plate with a black anodized finishing; b) Copper plate after a thermal shock treatment (<250 °C) having a black anodized layer and a silica coating; c) Plate with a top aluminum selective layer with 2 min of PVD; d) Plate with a top aluminum selective layer with 5 min of PVD; and e) Hydrophobic SiO_2-TiO_2 layer deposited by APPJ on a black anodizing layer above a copper plate



CONCLUSION

A methodology was developed for the study and preparation of absorbent black coatings by anodizing copper in aqueous solutions. A silica protective coating was applied using APPJ. A top coating made of SiO_2-TiO_2 was applied to give a super-hydrophobic property. This characteristic was obtained on the surfaces. Nonetheless, this one requires multiple depositions until achieving the water repellent property, which gave a dispersive white finishing. Such characteristic was not compatible with the solar radiation absorption requirement.

The application of selective layers of aluminum was carried out for the best absorption of radiation. Nonetheless, the PVD deposition with 2 min was not enough to cover the surface with aluminum. The PVD deposition with 5 min covers the surface with a thin layer of aluminum. The obtained thickness was the best for allow the absorption of sunlight and avoid the re-irradiation of infrared wavelength due to the heated surface. These coatings were about 30 nm thick. Nonetheless, the coating on top of the black CuO layers was not enough to show a significant infrared reflection. Thicker layers were not testes because they gave a mirror finishing.

The proposal of concentrating solar power by using a Fresnel lens shows effectiveness as a test for absorbent black coatings. The sudden heating and cooling represented the samples exposed to weather changes. Some samples were heat treated with a thermal shock by the Fresnel lens at a maximum recorded temperature of 250 °C. The heating, annealing, and cooling times were about 2 min, 5 min, and 8 min, respectively.

The three-layered system on the copper substrate improve the performance of the material for absorption and transferring the thermal energy. Optimization of the process parameters is mandatory to be applied to surfaces on service at the solar fields. Other practical uses include the utilization of such layers on absorbing surfaces for water heating and drying edible fruits.

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