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Investigation of Hemispherical Thermal Emittance Using the Electrodeposition Technique on Bright Chrome Plated Thin Films at Different Percentage Concentration on Stainless Steel AISI 304 BA 2B For Possible Solar Energy Application.

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Abstract

A flat plate of stainless steel AISI 304 BA 2B was cut into 6 sample plates measuring (7.8cm x 7.7cm x 1.05cm) and polished using emery paper of grit numbers 100, 150 and 320 respectively, and vim polishing powder until their mirror finishing were obtained. The polished sample plates were thoroughly washed rinsed with distilled water, drip dried and were numbered. Each of the plates was chemically deposited in each freshly prepared solutions of 79 to 99% of chromic acid at 45^{0}C for deposition time of 10 minutes. Hemispherical thermal emittance values of the polished plates were measured before and after chemical deposition process. The hemispherical thermal emittance were found to increase slightly depending on the percentage concentration of the chromic acids in the solutions. The average hemispherical thermal emittance of the polished plates was found to be 0.15 ± 0.01. The hemispherical thermal emittance values of the sample plates deposited in different percentage concentrations of the acids vary from 0.16 to 0.17 ± 0.01. The electrodeposition technique of stainless steel AISI BA 2B produced a spectrally selective surface with optimum values of hemispherical thermal emittance of 0.16 ± 0.01 obtain for 79% concentration of chromic acid with film thickness of 1.308 ± 0.001µm. This surface could be employed in fabrication of spectrally selective surface to enhance the efficiency of flat plate solar collector.

1.0 Introduction

The application of solar energy are numerous and environment friendly when it is converted to heat, electricity or biomass. The devices for the conversion of solar energy into heat and electricity are photo thermal (solar thermal) devices and photovoltaic or solar cells, respectively. An efficient photo thermal device requires the presence of a spectral selective absorber. A spectral selective absorber is a surface that was maximum absorption of solar wavelengths (0.3 to 2.5 µm) and a low emittance for thermal wavelengths (3.0 to 30.0µm) [1] .Advantages of using spectral selective absorbers in solar energy collectors were first introduced by Igbinovia and Ilenikhena [2].Several methods have been used to fabricate selective surfaces on metallic substrates such as copper, galvanized iron, steel and aluminum substrates. Most of the selective absorbers were found to suffer atmospheric degradation and corrosion at elevated temperatures [3-4]. Recently, there is a wide spread of research on the fabrication of selective absorbers on stainless steels to overcome atmospheric degradation and corrosion affecting selective absorbers at high temperatures. Choudhury et al [5] reported colouration of stainless steels by aluminium anodizers in Australia using hot chromic and sulphuric acids to produce selective absorbers. Granziera [6] produced selective coatings on stainless steel 430 by chemical and thermal oxidation processes. Sharma et al [1] produced selective surfaces on stainless steel AISI 321 by chemical oxidation process. The electroless chemical bath deposition (ECBD) technique was predominantly employed in production of silver mirrors. It is a simple, cost effective and reproducible method of producing compound semi conductor metal halide and chalcogenide thin films on both metallic and non metallic substrates [7-9]. This films produced by this technique have comparable structural and optoelectric properties to those produced using other sophisticated thin film deposition technique such as chemical vapour deposition and sputtering. The technique has also been applied in producing emerging materials for solar cells, protective coatings, solar collectors and

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Corresponding author: ***Akhabue, E.O.,*** E-mail: osehontue.akhabue@uniben.edu, Tel.: +2347060515540 (Damisa John) solar thermal controls in buildings. It is now being adopted by some industries [10-14]. Its technology is based on slow release or controlled precipitation of the desired compound from its ions in a reaction bath. A complexing agent or liquand acting as a catalyst is usually employed to control the reaction in a suitable medium as indicated by the pH to obtain crystal growth. Otherwise spontaneous reaction and sedimentation of materials will result.

The aim of this research is to fabricate a spectrally selective surface. The objectives which are to improve the efficiency of solar collectors are.

1. To provide more efficient methods of storing the excess heat energy from the collectors and extracting it when required.
2. To enhance the operating temperatures of flat plate solar collectors to provide solar heat energy for heating and cooling purposes.

2.0 Experimental Studies

2.1 Sample plate preparation

A flat plate of stainless steel AISI 304 BA 2B was cut into 6 sample plates measuring (7.8cm x 7.7cm x 1.05cm) and polished using emery paper of grit numbers 100, 150 and 320 respectively, and vim polishing powder until their mirror finishing were obtained. The polished sample plates were thoroughly washed rinsed with distilled water, drip dried and were numbered.

2.2 Preparation of chromic acid solution

The mass m of chromium oxide was calculated using the formula;

$$m = \frac{M \times W \times V}{1000} \qquad (1)$$

where M is the required molar concentration,
W is the molecular weight of the reagent,
V is the volume of the required solution.

A metra electronic precision balance of model TL-600 and a capacity of 600g was used to measure the chromium trioxide for the preparation of chromic acid by dissolving it in a 5 litre beaker containing distilled water. This solution was then used in the first stage preparation of the electrolytic bath where the different solutions required for the 6 conditions required different percentage concentration of chromic acid ranging from 79% to 99%.

2.3 Basic formulation for the bath solution

The volumes of each of the prepared molar solutions of chromic acid and sulphuric in the electrolytic bath solution for various percentage concentration of chromic acid in solution is shown in Table 1. For each bath solution, four polished stainless steel AISI 304 BA 2B were bright chrome plated for 10 minutes. This was done for 99, 95, 91, 87, 83 and 79% concentration of chromic acid in solution bath.

Table 1 Bath solution constituents for bright chrome plating.

Bath solution serial number	Volume of 1M chromic acid (cm^3)	Volume of 1M sulphuric acid (cm^3)	Total volume of bath solution (cm^3)	Percentage of chromic acid in the solution (%)
1	297	3	300	99
2	285	15	300	95
3	273	27	300	91
4	261	39	300	87
5	249	51	300	83
6	237	63	300	79

2.4 Preparation of the anodes

The anodes were made of lead stripes about 5cm thick .The lead stripes were cut and formed into the required lengths of 101.6m each. Lead is attacked by chromic acid and an insulating layer of lead chromate (yellow in colour) will be formed if the anodes are not first treated. This lead chromate interferes with the plating current flow, but it is easily avoided by transforming the working surface of the anodes into lead peroxides. The oxide prevents the formation of lead chromate, while allowing the passage of the plating current.

2.5 Peroxidizing the anodes

The lead anodes used in the electrolytic bath were peroxidized to ensure constant flow of current during the electroplating process.

The stages of peroxiding the anodes are as follows:
(i) 5% solution of sulphuric acid was made in water.
(ii)The lead anodes were scraped by cleaning.
(iii)The lead anodes were then connected to a DC power supply, one positive, and one negative.
(iv)The current was adjusted with a rheostat to 5A and this was maintained at 12V for 15 minutes.
(v)The polarity of the DC supply was reversed and 5A was maintained for 15 minutes
(vi)Finally, the polarity of the DC supply was reversed one more time and was maintained for another 20 minutes.

A dark brown coating on the anode indicated the presence of the protective layer of lead peroxide. The prepared anodes were dried and put aside ready for use. The process was repeated on any anodes which tend to develop yellow spots during use because of lack of the lead peroxide coating.

2.6 Electrodeposition process

Electrodeposition also called electrochemical deposition or cathodic deposition is the method of thin film deposition used to produce bright chrome. The electroplating process was carried out in a 500cm^3 beaker containing the required chromic acid. In the first stage of the experiment, 297cm^3 of the prepared 1M Chromic acid was added to 3cm^3 of 1M sulphuric acid. This gave a 99% concentration of Chromic acid solution of 300cm^3 of the bath solution. The lead strip was used as the anode while sample plate 1 was used as the cathode. The set up was arranged as shown in Figure1.

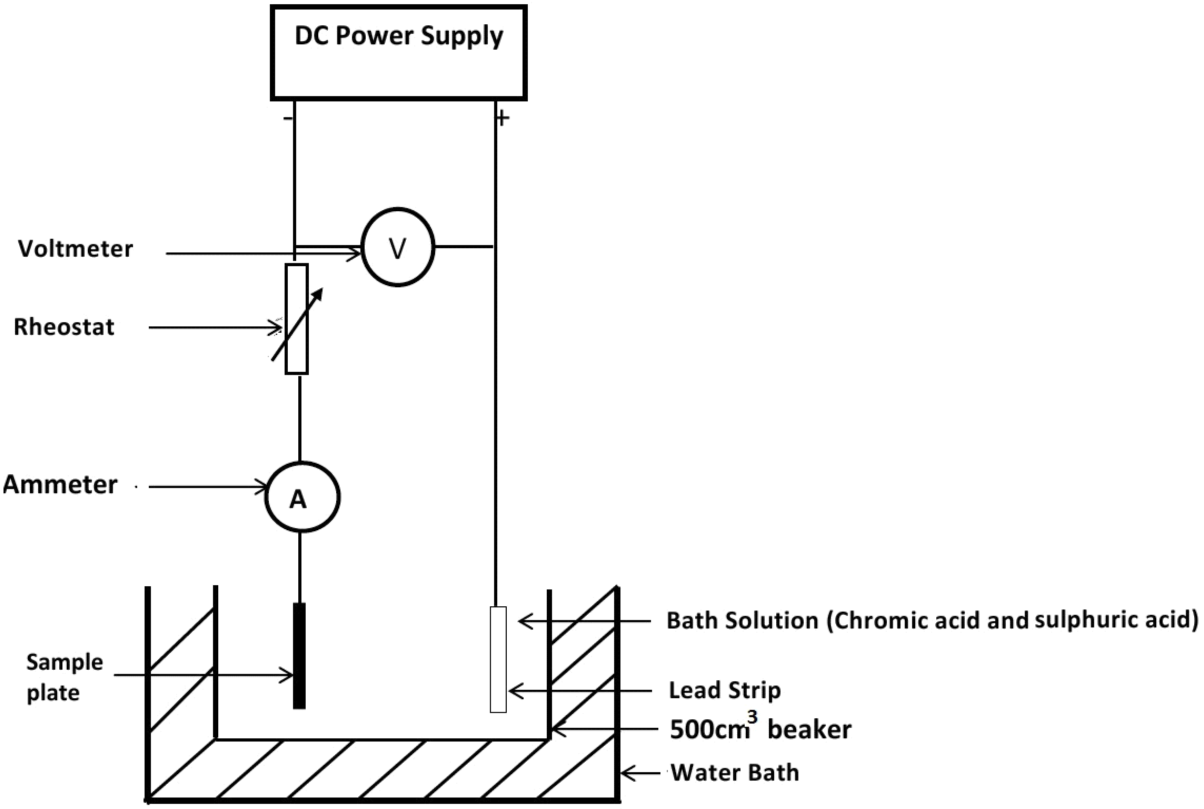

Fig. 1 Schematic representation of the electrochemical deposition process.

The beaker containing the solution of acids (chromic and sulphuric acids) was placed in a hot water bath with at 45℃. For chrome plating direct current is required the reason is that chromic acid has a strong itching action on most metals when no current is flowing and chrome is deposited on the piece only when the current is flowing. The current used for the chrome plating was 2±0.1A and the voltage was maintained at 12V for 99, 95, 91, 87, 83 and 79% sulphuric acid for 10 minutes. A voltmeter was used to monitor the voltage while an ammeter was used to measure the current and a rheostat was used to adjust the current. The voltage was maintained at 12V and the current was adjusted with the rheostat to obtain 2A. A constant temperature of 45^{0}C was maintained and this was checked continuously with a thermometer inserted in the water bath. A stop watch of sensitivity 0.2 seconds was used to note the deposition time. At the end of the deposition process which lasted for 10 minutes, the sample plate was quickly removed from the solution of acids and transferred into a bowl of distilled water at room temperature and rinsed in it. It was rinsed again in another bowl of distilled water to remove all traces of the acids and finally left to dry. A bright film of chromium oxide was found on the cathode (sample plates) as a result of electrolysis that occurred in the bath solution when current flowed in it. Similarly, 285cm^3 of 1M chromic acid was added to 15cm^3 of 1M sulphuric acid making a total volume of 300cm^3 and this contained 95% of chromic acid. The solution containing the chromic acid and sulphuric acid was used as the electrolyte. Using the lead as the anode, plate 2 was connected as the cathode and 10 minutes at 45^{0}C at a voltage of 12V when a current of 2A flowed through it. Bright chrome of chromium oxide was observed at the cathode (sample plate). The plate was again rinsed with distilled water and allowed to drip dry. The experiment was continued under the same condition using:

(a) 91% chromic acid in the total bath solution for sample plates 3

(b) 87% chromic acid in the total bath solution for sample plates 4

(c) 83% chromic acid in the total bath solution for sample plates 5

(d) 79% chromic acid in the total bath solution for sample plates 6

At the end of the experiment, the film thickness, and hemispherical thermal emittance were calculated.

2.7 Reaction equation of thin film

$$H_2Cr0_4 \rightarrow (HCr0_4)^- + H^+ \qquad (2)$$

When chromic acid is added to water there is an emergence of a purple colouration given by equation (3)

$$CrO_3 + H_2O \longrightarrow H_2CrO_4 \qquad (3)$$

3.0 MEASUREMENTS

Several measurements were carried out in the course of this research and these include the following:

3.1.1 Measurement of hemispherical thermal emittance

The hemispherical thermal emittance of the sample plate before and after deposition was measured using an emissometer with blackbody sample as standard as shown in plate 1. The output of the emissometer was connected to a digital voltmeter. Hemispherical thermal emitance of the sample plate was determined using equation (4).

$$\frac{E_p}{E_b} = \frac{V_p}{V_b} \qquad (4)$$

Where E_p is the hemispherical thermal emittance of sample plate, E_b is hemispherical thermal emittance of black plate, V_p is the voltmeter reading of the sample plate and V_b is the voltmeter reading of the black plate.

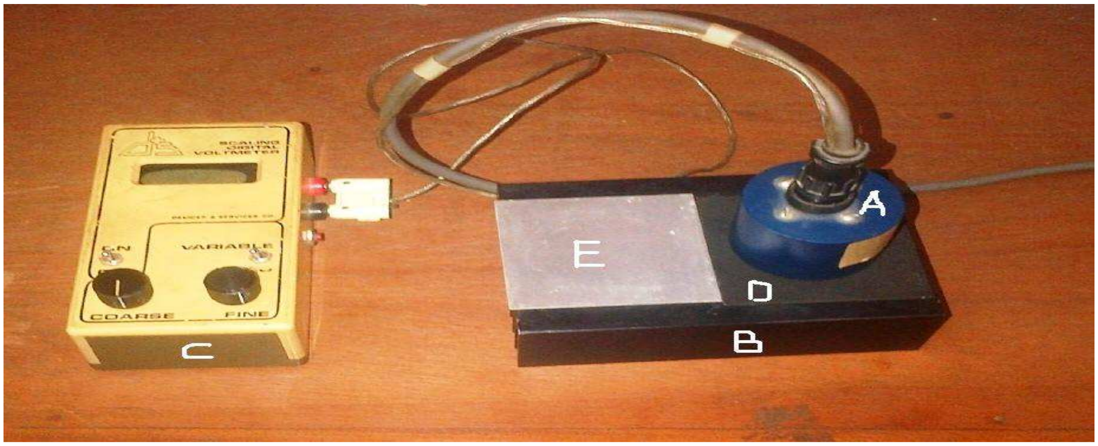

Plate 1: Emissometer and digital Voltmeter

A = Emissometer detector
B = Heat sink
C = Digital Voltmeter
D = Black plate
E = Sample plate

3.2 A measurement of film thickness

The film thickness was calculated from equation (5) [1].

$$t = \frac{m}{2dA} \tag{5}$$

Where *m* is the mass of film deposited on a sample plate, obtained from the difference in mass of each sample plate before and after film deposition, *A* is the area of film of the sample plate and *d* is the density of film on the sample plate.

4.0 Results and Discussion

Table 2 shows the hemispherical thermal emittance of polished stainless steel plates at for different percentage concentration of chromic acids for deposition time of 10 minutes, while Table 3 shows the corresponding film thickness of coated sample plates for different percentage concentration of chromic acids for deposition time of 10 minutes.

Table 2: Hemispherical thermal emittance of bright chrome plated stainless steel AISI 304 BA 2B for different percentage concentration of chromic acid in the solution of chromic acid and sulphuric acid for 10 minutes.

Plate No	Surface Treatment	Percentage concentration of chromic acid in solution (%)	Emissiometer voltmeter		Thermal emittance $E_p \pm 0.01$
			Black plate $V_b \pm 0.01$ (mV)	Sample plate $V_s \pm 0.01$ (mV)	
	Polished and uncoated	-	3.52	0.573	0.15
1	Polished and coated	99	1.580	0.275	0.16
2	Polished and coated	95	1.580	0.282	0.17
3	Polished and coated	91	1.580	0.285	0.17
4	Polished and coated	87	1.580	0.283	0.17
5	Polished and coated	83	1.580	0.285	0.17
6	Polished and coated	79	1.580	0.270	0.16

Table 3: Film thickness of bright chrome films deposited on stainless steel AISI 304 BA 2B for different percentage concentration of chromic acid in the solution of chromic acid and sulphuric acid for 10 minutes.

Plate No	Percentage concentration of chromic acid in solution (%)	Mass of deposited film m±0.01 (g)	Area of coated plate A ± 0.01 (cm^2)	Film thickness t ± 0.001 (μm)	Hemispherical thermal emittance of coated plate $E_p \pm 0.01$
1	99	0.05	35.625	0.256	0.16
2	95	0.06	37.500	0.280	0.17
3	91	0.02	37.500	0.380	0.17
4	87	0.03	38.44	0.513	0.17
5	83	0.04	30.28	0.920	0.17
6	79	0.07	37.50	1.308	0.16

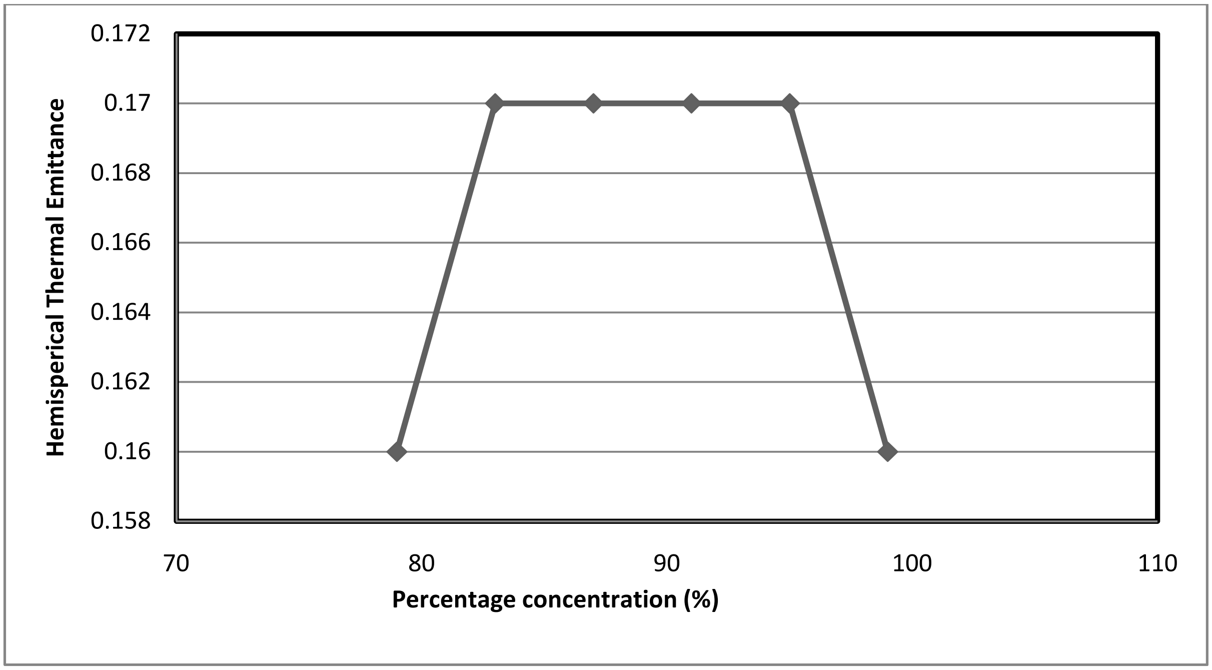

Fig.2: Variation of Hemispherical thermal emittance with percentage concentration of chromic acid at deposition time of 10minutes.

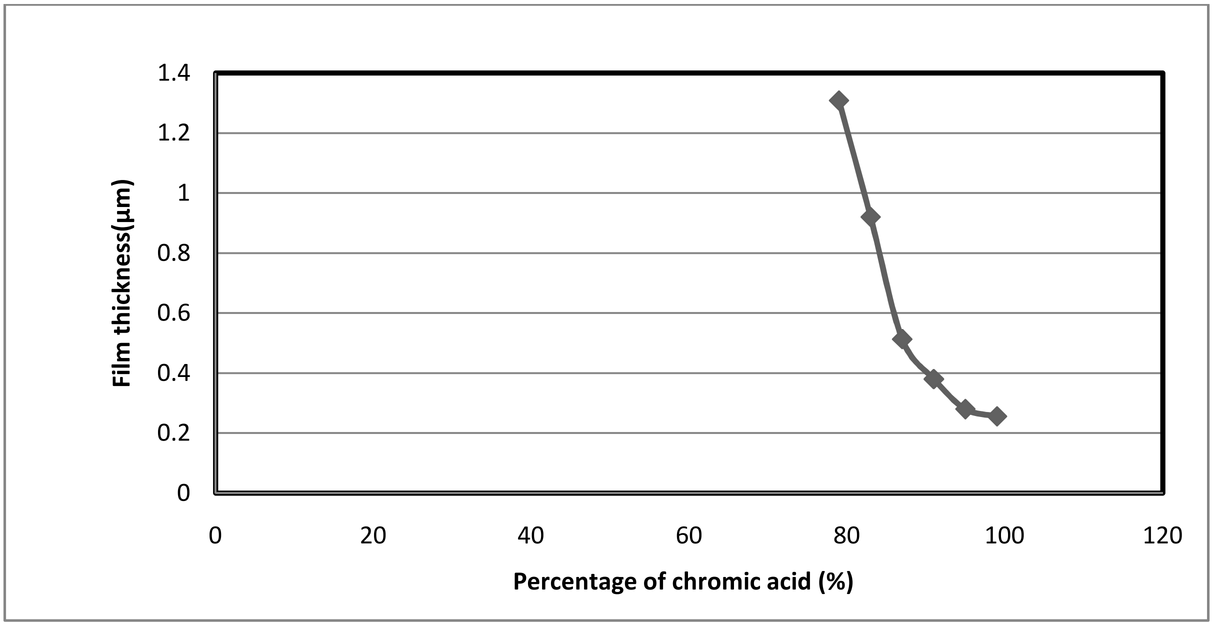

Fig.3: Variation of film thickness with percentage concentration of chromic acid at deposition time of 10minutes.

The average hemispherical thermal emittance value of polished stainless steel AISI 304 2B BA is 0.15 ± 0.01. This value compares well with thermal emittance of 0.15 ± 0.01 for polished stainless steel 430 [15-18, 2] and 0.14±0.01 for the polished stainless steel AISI 304 2B BA [19]. Table 2 and Figure 2 show that the hemispherical thermal emittance value of coated sample plates increases slowly from 0.16 to 0.17 ± 0.01 different percentage concentration of chromic acid for deposition time of 10 minutes this values compare favourably with thermal emittance value of 0.16 to 0.19 ± 0.01 for the improved electroless chemical bath deposition of stainless steel 430 [15], thermal emittance value of 0.16 to 0.18 ± 0.01 for the solution growth of stainless steel 430 [16], thermal emittance value of 0.15 to 0.19 ± 0.01 for improved chemical bath deposition (CBD). Table3 and Figure 3 show that the thickness of the film increases from 0.256 to 1.308µm at different percentage concentration of chromic acid for deposition time of 10 minutes. The thickness of the film could be hardened to withstand adverse weather condition and at the same time retaining its minimum thermal radiation losses to enhance the efficiency of the photo thermal converters. Surfaces with poor film thickness are not durable and cannot withstand adverse weather conditions while those with higher film thickness and higher thermal emittance cannot retain much heat to enhance efficiency of photo-thermal converters. Surfaces with low thermal emittance may be employed in the fabrication of spectrally selective surfaces which could be used in poultry production for the construction of solar chick broader and could provide heat, for warmth and protection for young chicks which have little or no insulating feathers from adverse weather conditions during the day [19-20, 1].

5.0 Conclusion

Thin films of bright chrome were deposited on the polished stainless steel AISI 304 BA 2B sample plates using electroplating technique for different percentage concentration of chromic acid for deposition time of 10 minutes. An emissiometer was used to determine the hemispherical thermal emittance values of the sample plates before and after film deposition. The average hemispherical thermal emittance of the polished sample plates is 0.15 ± 0.01. Values of the hemispherical thermal emittance of the coated plates vary from 0.16 to 0.17 ± 0.01. Their corresponding film thicknesses vary from 0.256 to 1.308 ± 0.001µm. The values of hemispherical thermal emittance of the coated sample plates are low and increase slightly with increase in percentage concentration of chromic acid. The surface with the most favourable hemispherical thermal emittance values of 0.16± 0.01 and film thickness 1.308µm ± 0.001 were obtained for 79% concentration of chromic acid could be employed to fabricate selective absorbers for photothermal converters.

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