



DESIGN OF SOL-GEL COPPER OXIDE BASED COATING FOR SOLAR ENERGY APPLICATIONS

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ABSTRACT:

Solar energy harvesting is of a great interest lately as it's a clean and environmentally friendly energy source. Therefore, Solar selective coatings development became one of the pioneering research routes nowadays. Copper oxide based films were prepared by a sol-gel route and deposited on a stainless steel substrate via dip coating. The solution was prepared with several concentrations of polyethylene glycol as a surfactant and agglomeration controlling agent. The effect of several concentrations on the optical properties was studied. The microstructure of the coats was discussed in details reflecting on the optical properties as well. In general, the copper oxide based coats have shown uniform and dark visual appearance with acceptable adherence. The coats have been successfully prepared and analysed with a promising absorptivity up to 96.3% with an optical band gap that varies between 2.5eV and 3.03eV.

Keywords: Solar selective coating, copper oxide, PEG, sol-gel, bandgap, characterization.

1. INTRODUCTION

Among all transition metal oxides, copper oxide (CuO) has gained a great interest in the solar energy harvesting field due to its unique dielectric and optical properties [1]. CuO is widely known as a promising solar selective material with a low cost preparation procedure. Cu₂O, another oxide of copper, shows good solar absorption but lower than that of CuO [2]. CuO has a band gap reported between 1.3 and 1.7eV with a black color and partial transparency in the visible range [3].

Several copper oxide preparation techniques are available and have shown reliable results in thin film formation. However, due to the cost-viability of the sol-gel technique as one of the easiest ways to produce metal oxide layers over different substrates, it has gained a wide range of research interest through the past couple of years. As stated clearly in [4], sol-gel preparation can be controlled through adjusting several parameters as the precursor's concentration, the solvent's type, the solution's pH value, types of additives, heat treatment, solution aging, ... etc. Moreover, the particles formed in gel matrix possess uniform shape and size that enhances the optical, electrical, magnetic and other intrinsic nature of the materials.

Copper oxide was successfully prepared in several previous literatures with different applications and requirements. Halin et. al [5] used sol-gel method to prepare cuprous oxide thin films to coat solar cells. They have proven that adding a polymeric additive to the solution steps like polyethylene glycol (PEG) can enhance the properties of thin films relatively. Hashim et. al [6] discussed the effect of molarity concentration on the properties of the synthesized films. They

found that the transmittances of CuO thin films decrease as the thickness increase with increase of molar concentrations. They have also deduced that electrical properties show current increase when the molar concentration increases. Mallick et. al [7] studied the effect of changing the solvent through the sol-gel preparation on the microstructure, properties and behavior of thin films. They compared ethanol and propanol and their effect on optical absorption. Bibi et. al [8] discussed the results of changing the annealing temperature over the different properties of films. They observed that a structural change in the prepared copper oxide was observed from cluster formation to Nano-fibrils by increasing the annealing temperature.

In the present work, a route for preparing solar selective coat based on copper oxide using sol-gel technique is used. The authors studied the effect of the variation of the weight percentage of the addition of a polymer, mainly PEG, on the optical properties of the targeted coat.

2. MATERIALS AND METHODS

Different applications monitor the sol-gel synthesis route by using a certain precursor, adding a certain agent, using a specific solvent or using a unique substrate. The three basic precursors used previously in copper oxide film preparation were copper nitrate, copper acetate and copper chloride. As stated in [9], these three metal salts can give successful gelled structure and consequently, produce promising thin films.

In this study, the percentage of added PEG is increased and studied in an attempt to increase the optical properties of the coat. This can be done by increasing the heat treatment temperature up to 600oC. The polymeric chains in that case, start to break down leaving a porous structured film behind in addition to some carbon content that can both contribute to the increase in absorption of solar energy.

3. EXPERIMENTAL WORK

As previously mentioned, the preparation of metal oxide from an acetate precursor is the widely used procedure. Consequently, the procedure of sol-gel preparation was based on that mentioned in other literature [3], [6], [15], [18] with some modifications based on the target of this experimental work. Chemicals used with their predefined masses are shown in details in Table 2 . In [6], it was stated that a molar concentration of 0.5 M gave the best stable results. Therefore, it was used as a reference molar concentration for the rest of procedure. The detailed steps of sol-gel preparation of copper oxide thin film were as follows:

To prepare 25 ml of the solution:

- 1- Add the PEG to 5 mL of distilled water and leave it under stirring for 5 minutes.
- 2- Add the previous solution dropwise to the IPA in addition to the DEA and leave stirring for 1 hour.
- 3- Add the CuAc to the resultant solution and leave it stirring overnight for 50% evaporation of the total volume.
- 4- By the next day, 25ml of the solution are left.

Leave the solution in a sealed beaker without stirring for stabilization one whole day before dip coating.

Table 1 shows a cross mapping between different previous literature and the type of substrate, precursor and solvent used. It can be observed that the most frequently used precursor was copper acetate and the commonly used solvent was isopropanol.

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To prepare 25 ml of the solution:

- 5- Add the PEG to 5 mL of distilled water and leave it under stirring for 5 minutes.
- 6- Add the previous solution dropwise to the IPA in addition to the DEA and leave stirring for 1 hour.
- 7- Add the CuAc to the resultant solution and leave it stirring overnight for 50% evaporation of the total volume.
- 8- By the next day, 25ml of the solution are left.

Leave the solution in a sealed beaker without stirring for stabilization one whole day before dip coating.

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Table 1 - Cross mapping between previous literature and the used precursors, substrates and solvents

Research paper	Substrate	Precursor of copper			Solvent	Target application
		Nitrate	Acetate	Chloride		
[3]	Glass		①		Isopropanol	Thin film
[6]	Quartz		①		Isopropanol	Thin film
[8]	N/A		①		Propanol	Thin film
[10]	Glass		①		Ethanol	Thin film
[11]	Glass Quartz			①	Methanol	Thin film
[12]	conducting glass support		①		Ethanol	Thin film
[13]	various glasses			①	Ethanol	Thin film
[14]	Glass		①		Methanol	Thin film
[15]	N/A		①		Isopropanol	Thin film
[16]	N/A	①			Acetic acid	Powder production
[17]	N/A		①		Urea	Powder production
[18]	Glass		①		Isopropanol	Thin film
[19]	N/A			①	KOH/NaOH	Powder production

Table 2 - Chemicals used in sol-gel preparation of 25ml of the solution

Chemical	Formula	Mass or Volume used in preparation	Abbreviation in literature
Copper II acetate monohydrate	$C_4H_8CuO_5$	4.99 [gm]	CuAc
Isopropyl alcohol	C_3H_8O	45 [mL]	IPA
Polyethylene glycol [MW=4000]	$C_{2n}H_{4n+2}O_{n+1}$	3.15 [gm]	PEG
Di-ethanol amine	$C_4H_{11}NO_2$	2.5 [mL]	DEA

The effect of PEG was investigated by increasing the wt% of PEG with respect to the solvent. wt% of PEG with respect to the solvent was varied to be 5,10,15 and 20 wt% respectively. The resulting solution was homogeneous, viscous and deep dark blue in color without any suspensions or precipitates in all cases.

The substrate used in this research was Stainless Steel 304 (SS) cut at a size of 3cm X 10cm with 1 mm thickness. To prepare the substrate samples for coating, all samples were sandblast and then cleaned by distilled water. Further cleaning and degreasing was achieved by having them immersed in acetone and submitted in an ultrasonic cleaner for 10 minutes. Samples were then carefully dehydrated by a flow of hot air and then dipped into the sol-gel with a withdrawal speed of 1cm/min. Coat drying was carried out at 250°C for 15 minutes before heat treatment. According to [18] and [3], Sintering temperature must exceed 350°C in order to oxidize Cu into stable CuO. In addition, in order to obtain a porous film structure to harvest more solar energy, PEG needed to be heated up to 550°C to vaporize. Thus, heat treatment temperature was chosen to be 600°C for 1 hour. The results section will discuss the effect of PEG wt% variation on the optical properties in details.

Different measurements and characterization procedures were held in order to characterize the coating and test its functionality. The experimentally measured absorptivity and emissivity were measured using a UV spectrophotometer PG Instruments 90+ with integrating sphere and the infrared (IR) spectrometer Nicolet 380 FTIR respectively. Surface morphology and existing phases and compounds were investigated through SEM Camscan 44 and Philips PW 2404 X-RAY Spectrometer respectively.

5. RESULTS AND DISCUSSION

Table 3 shows the nomenclature that will be used throughout this section corresponding to each sample referring to its properties with respect to the variation of PEG wt%.

Table 3 - Nomenclature of prepared samples with properties variation

Sample Name	PEG wt%
P5	5%
P10	10%
P15	15%
P20	20%

4.1 UV-Visible absorptivity analysis:

For successful solar energy harvesting, absorption in the UV range is desirable to be maximized. Reflectance spectra in Uv-Vis range was collected through the wavelength range between 300-

700 nm. Absorption was calculated based on the fact that $A + R + T = 1$, and since stainless steel is opaque, $T = 0$. Therefore, it can be assumed that $A+R = 1$ and average absorptivity can be calculated through the integration involving the spectral solar irradiance according to [20] as follows

$$\alpha = \frac{\int_{0.3}^{0.7} I_{sol}(\lambda)(1-R(\lambda))d(\lambda)}{\int_{0.3}^{0.7} I_{sol}(\lambda)d(\lambda)} \quad (1)$$

Where

α Average absorptivity

I_{sol} Solar irradiance

λ Wavelength

R Measured reflectance

The wt% of the PEG was increased gradually with respect to the solvent and its effect on the optical properties was determined.

Figure 1 shows the absorptivity trends of different PEG wt%. Increasing the percentage of the PEG from 5% to 20% increased the absorptivity as a trend and as an average value as shown in Figure 3.

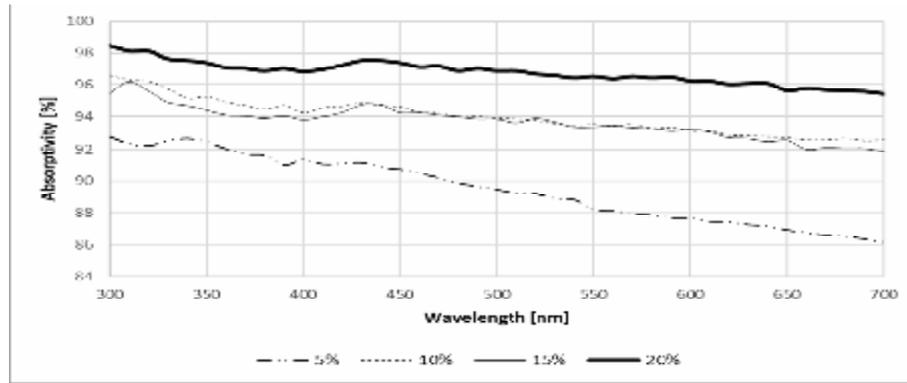


Figure 1 - Absorptivity spectra of CuO films with different PEG wt%

The optical absorptivity can also contribute to the determination of the optical bandgap of the coat. As explained in [21], a small bandgap allows a large range of photon absorption. However, in most cases, most of the photon energy is lost as heat. A large bandgap coat has low absorbance compared to others. Tauc plot was drawn for all samples as stated in [10] based on the following relation

$$\alpha h\nu = A(h\nu - E_g)^n \quad (2)$$

Where

α Measured Absorptivity

$h\nu$ Energy [eV]

A Constant

E_g Bandgap [eV]

n depends on the nature of the transition

, all reported similar bandgap values to [5], [7], [8], [10], [17], [22]previous literature, such as reported that a better solar energy harnessing is [8]those obtained in this study. Maryam Bibi in

, it [23]improved by widening the bandgap of copper oxide structures in particular. Moreover, in [22]was reported that porous structures always behold wide bandgaps. N. Zayyoun et. al in against $h\nu$ simply stated that obtaining the bandgap value corresponds to plotting the energy where their intersection obtains the value for the corresponding bandgap. $(\alpha h\nu)^2$ Figure 2 shows a sample of the tauc plots used to obtain the bandgap for several samples. This tauc plot corresponds to the sample P20 with the highest PEG content. Figure 3 shows the mapping between average absorptivity and calculated average bandgap of each sample type. It can be observed that there's a remarkable increase in the average bandgap with the increase of the PEG %. This verifies the increase in porosity with the increase of the PEG %. Increased absorptivity, however, can be explained by the existence of carbonic residuals from the PEG that were able to absorb more energy.

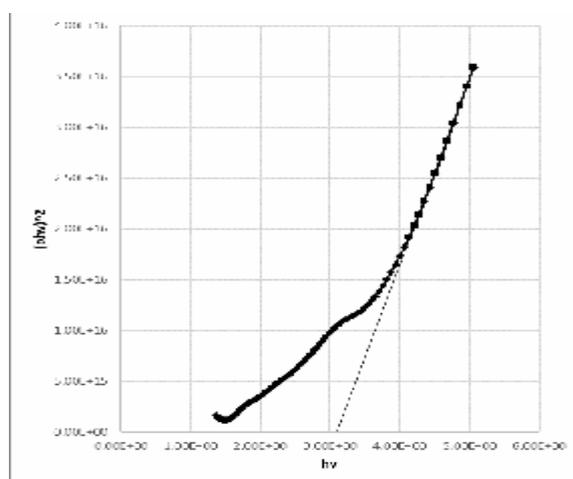


Figure 2 Sample Tauc plot corresponding to P20

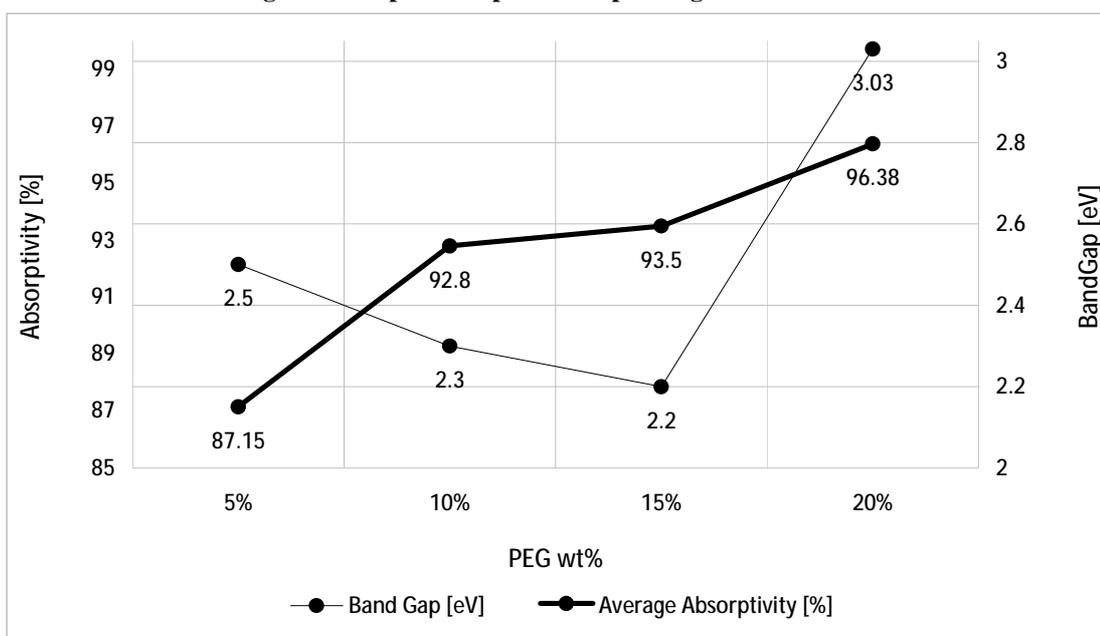


Figure 3 – PEG wt% Vs the Absorptivity and Band gap

4.2 IR emissivity measurement:

In order to obtain a coat that has effective absorption in both the UV and the IR wavelength range, thermal emittance of the coat in the IR range should be minimized. To measure the thermal emittance, the following equation [20] was used from the data collected from the FTIR analysis represented in $R(\lambda)$ such that

$$\varepsilon_{th} = \frac{\int_{\lambda=2.5}^{20} I_p(\lambda)(1-R(\lambda)) d(\lambda)}{\int_{\lambda=2.5}^{20} I_p(\lambda) d(\lambda)} \quad (3)$$

Where

ε_{th} Thermal emittance [%]

$I_p(\lambda)$ Plank's blackbody at $100^\circ C$

$R(\lambda)$ Reflectance value [%]

It was observed that all coats have similar emissivity behavior as the sample shown in Figure 4. It was observed that the emissivity measurements of the copper oxide coats was very high with respect to other coats mentioned in previous literature that are used for solar energy capture .

shows the detailed values of each sample type. All coats with the variation of PEG wt% were observed to have an emissivity that exceeds the 50% which is regarded as a very high emissivity in solar selective coating applications. This initiates the need of either a multi-layered structure coat or a mixture based coat that can reduce the emissivity of the copper oxide layer. This can be achieved by adding a thin reflective layer underneath the copper oxide layer and adding an anti-reflecting layer over it in order to entrap the collected solar radiations in between.

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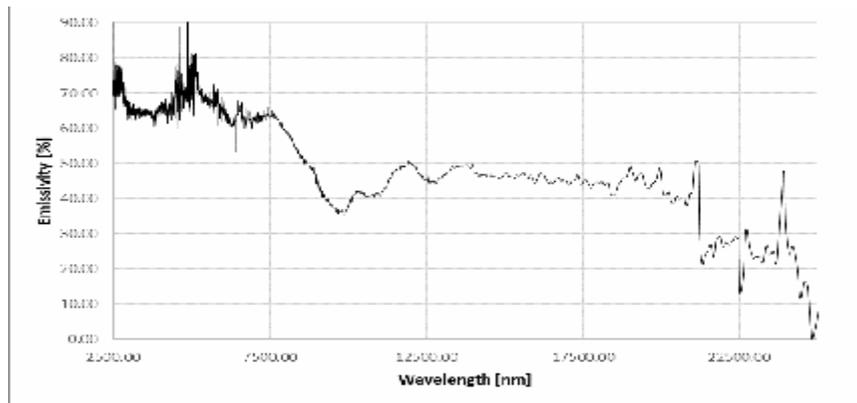


Figure 4 – Sample FTIR spectra of coats of sample P20

4.3 Selectivity of Coat Analysis:

The selectivity of a solar coat is measured by the ratio α/ε_{th} . However, this measurement can be misleading when the absorptivity is low and emissivity is relatively very low. Therefore, some other research, like [20], has adopted another definition of selectivity to be equal to $\alpha - \frac{1}{2}\varepsilon_{th}$. They have stated that this formula defines an acceptable weight factor to emissivity other than the ratio used. Compares the selectivity of samples using both stated formulas.

Table 4 - Optical properties of different coats and selectivity measurements in the two proposed formulae

Sample	α	ϵ	$\frac{\alpha}{\epsilon_{rh}} S =$	$\alpha - \frac{1}{2} \epsilon_{rh} S' =$
P5	87.15	59.4	1.46	57.45
P10	92.8	64.3	1.44	60.66
P15	93.5	69.4	1.34	58.83
P20	96.38	68.9	1.39	61.93

Based on the selectivity measure of S, p20 is not the best candidate in selectivity though it has highest value of α and ϵ which has a similar value to others. On the contrary, S' has awarded the same sample, p20, the highest value of selectivity which contributes to the enhanced absorptivity.

4.4 Surface Morphology Through Scanning Electron Microscopy(SEM):

The morphology of the coat was analyzed using SEM images and compared to agreement with EDX stated in the upcoming sections. Table 5 shows the samples that were investigated through SEM analysis. Film thickness was measured through optical microscopic cross section investigation. It can be observed that a higher PEG content gave a more thick layer which is agreed with the emphasis previously introduced in [5].

Table 5- Characteristics of samples for SEM

Sample	PEG%	α [%]	ϵ [%]	Thickness [μ m]
P5	5%	86.3%	59.5%	2.96
P20	20%	95%	69.4%	6.77

Investigating the sample P5, sol-gel was prepared with the same procedure mentioned in the experimental section. By visual inspection, the resulting coat was homogeneous, adherent and uniform. Further investigation through the SEM images is shown in

Figure 5. Surface morphology is consistent with uniformly distributed closely packed islands of well-defined particles. With the 4000X magnification, semi-spherical grains of CuO can be well defined. Similar morphologies were reported in previous research such as in [24] where CuO particles were prepared via chemical precipitation method using Copper (II) chloride dehydrate as a precursor and annealed at 600°C.

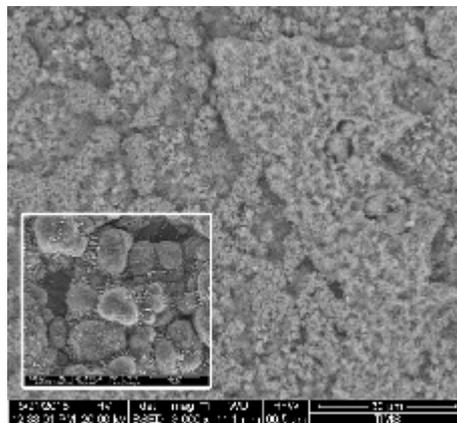


Figure 5 - SEM image at 3000X for sample P5

Sample P20 corresponds to a PEG wt% = 20% of the total solvent weight percentage. According to [25], a porous structure can be obtained after the coat heat treatment where full vaporization of PEG chain structure takes place leaving cavities behind. The early visual inspection of the coat showed a fully adherent structure but there was observed fine ashes on the surface which can be defined as the residuals of extra carbon content resulting from the PEG vaporization. Figure 6 shows the SEM surface morphology of sample P20. Two different textures are observed in the image referred to as areas 'a' and 'b'. In addition, some agglomerated grains are observed at other areas. Tran et. al in [26] related the presence of agglomerated clusters to the presence of water added to the alcohol as a solvent. According to their theory, the water viscosity is higher than that of the alcohol. Consequently, the coarsening process of the nanoparticles that dissolved in water takes less time than that dissolved in alcohol resulting in agglomerated clusters.

At higher magnification area a as shown in Figure 6 to the right, there exists well defined loosely packed spherical arrays. Same structure was observed in [26] and [27] where they defined this structure as a function of the increase of the PEG content as a surfactant. It has a great role in obtaining a smooth surface with an increased sphericity in the existing particles. On the other hand, area b shows well defined pores or voids in the structure. These pores are a result of the vaporization of the PEG chains through the heat treatment stage. They are responsible for entrapping more solar energy thus increasing the absorptivity as discussed earlier.

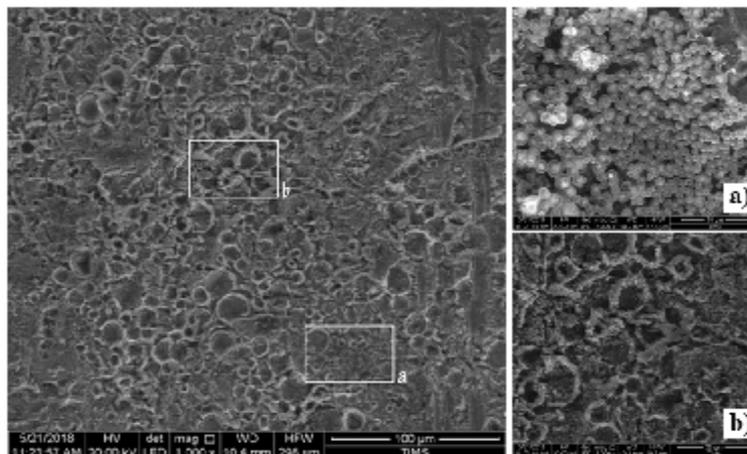


Figure 6- SEM image for sample P20 with magnified areas a and b for investigation

4.5 Elemental Analysis Through EDX:

To have a closer look at the existing phases and analyze the presence of all elements seen in the SEM analysis. EDX analysis was held at several structures for validation. Figure 7 shows the EDX analysis for area a that has been discussed earlier in Figure 6. It can be observed that there exists high content of copper and oxygen which validates that the particles shown can be particles of copper oxide. A small amount of Fe and Cr resembles the SS in the base substrate. A low carbon content may correspond to the presence of carbon residual compounds as a result of the breakdown of PEG from the structure where voids are left behind.

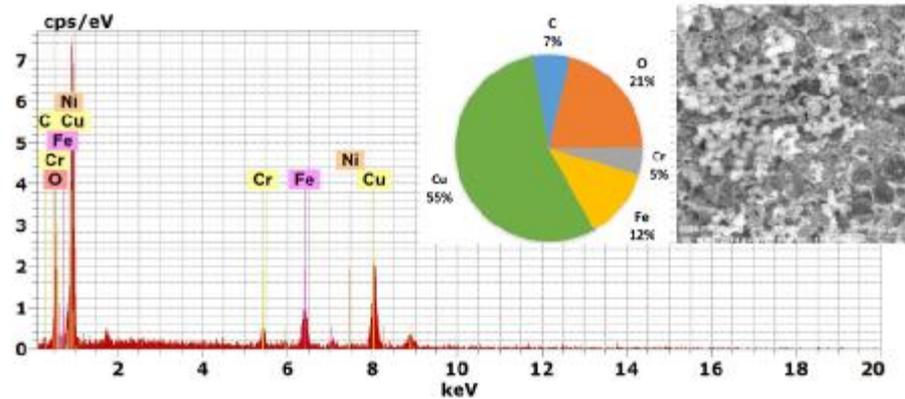


Figure 7 - Area a EDX analysis

Investigating area b where the voids are seen, EDX analysis is shown in Figure 8. A lower content of Cu and Oxygen can be seen which means that the voids may not contain much copper oxide content. However, the presence of some percentages of Fe, Ni and Cr correspond to the base metal which verifies that there's a very thin layer of coat at the voids areas that can be visually distinguished through the SEM images.

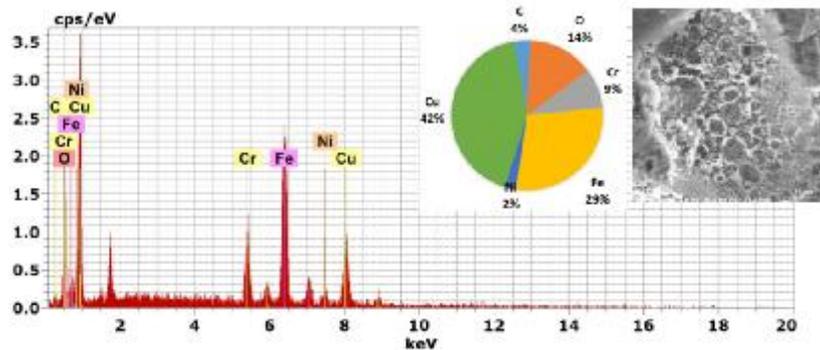


Figure 8 - Area b EDX analysis

5 CONCLUSION

Successful sol-gel preparation of copper oxide was experimentally carried on with variable PEG wt%. Stainless steel samples were dip coated with the prepared solution and heat treated up to 600°C. Visual inspection has concluded that the coats were fully adherent and smooth on the surface of the substrate with some excess carbon at the surface for an increased amount of PEG. Best absorptivity and emissivity results were for the solution prepared with the highest PEG wt% that produced a selectivity S of $\alpha/\varepsilon = 96.38/69.4$. The high emissivity strongly recommends the usage of a highly reflective layer underneath the copper oxide film in order to suppress those high emissions in the IR range. Measured bandgaps was between 2.5eV- 3.03 eV. SEM analysis validated the existence of spherical particles of copper oxide in addition to pores in higher PEG content which was not seen in the low content samples. EDX analysis validated the presence of copper oxide through the presence of copper and oxygen content in addition to some carbon corresponding to the presence of a bond between the PEG and the copper particles during sol-gel preparation.

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