Effect of $CuSO_4$ Content in the Plating Bath on the Properties of Composites from Electroless Plating of Ni-Cu-P on Birch Veneer

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A wood-based composite was prepared *via* simple electroless Ni-Cu-P plating on birch veneer for EMI shielding. The effects of $CuSO_4{\cdot}5H_2O$ concentration on the metal deposition, elemental composition, phase structure, surface morphology, wettability, surface resistivity, and shielding effectiveness of coatings were investigated. The coatings were characterized using X-ray diffraction (XRD), X-ray photoelectron spectroscopy (XPS), and scanning electron microscopy (SEM). When the $CuSO_4{\cdot}5H_2O$ concentration was increased from 0.6 g/L to 2.2 g/L, the metal deposition was decreased from 79.61 g/m^2 to 66.44 g/m^2. Elemental composition showed that the copper content in the coating increased significantly, whereas the nickel content was reduced significantly and the phosphorus content was slightly reduced. The crystallinity of coatings increased, and fine-grain structure was observed, with higher copper content. Ni-Cu-P deposition improved the hydrophobic properties when the maximum static contact angle increased from 77.5° to 116.5°. The lowest surface resistivity was 367.5 mΩ/cm^2, and the EMI shielding effectiveness of Ni-Cu-P-coated veneers was higher than 60 dB in frequencies ranging from 9 kHz to 1.5 GHz.

Keywords: Birch veneer; $CuSO_4$ content; Electroless Ni-Cu-P plating; Electromagnetic shielding effectiveness; Morphology; Wettability

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INTRODUCTION

Extensive use of electronic equipment has brought about unwanted and even harmful electromagnetic interference (EMI). An effective method to prevent these harmful effects is through the use of shielding material. Wood is a traditional biomass material used for its heat insulation, sound insulation, humidity control, and prominent mechanical strength. However, its potential application in EMI shielding is limited due to the non-conductivity of dry wood. To fabricate EMI shielding wood-based material, the electroless plating technique has been introduced by many researchers to deposit metallic films on wood veneers (Nagasawa *et al.* 1990, 1991, 1992, 1999; Huang and Zhao 2004; Wang *et al.* 2006a, 2006b, 2008, 2011a, 2011b, 2011c; Wang and Li 2007a, 2007b; Sun *et al.* 2012).

Electroless Ni-P alloy is most commonly deposited on the surface of metal and nonmetal substrates (Yang *et al.* 2009; Srinivasan and John 2009). Nickel, however, has relatively low electrical conductivity. Recent studies have shown that electroless nickel ternary alloy deposition is the most effective method of improving the properties of the binary Ni-P alloy system.

The inclusion of Cu in Ni-P coatings substantially improves the microstructure and properties of the coating. Krasteva *et al.* (1994) reported that the introduction of copper into electroless deposited Ni-P alloy conferred higher thermal and nonmagnetic stability. Abdel Aal and Shehata Aly (2009) confirmed that finer-grained structure was observed by increasing Cu content into the Ni-P matrix. Ashassi-Sorkhabi *et al.* (2002) concluded that Ni-Cu alloys with Cu content around 30% are highly resistant to corrosive environments such as in a halide solution such as seawater. Guo *et al.* (2009) demonstrated that the crystallinity of deposits increased with increasing copper ion concentration in the solution. In addition, a model for co-deposition has been proposed by Armyanov *et al.* (1999). It is claimed that the copper added to the solution for electroless plating plays three different roles: as a stabilizer (Cu^{2+}) and as an accelerator due to the catalytic properties of Ni-Cu-P alloys; it also affects the solution stability because of the formation of randomly dispersed copper particles in the solution. Electroless Ni-Cu-P has been extensively researched on substrates such as Cu, Al, and polyester fabric (Liu and Zhao 2004; Ranganatha *et al.* 2012; Guo *et al.* 2009). However, there have been few reports on the application of Ni-Cu-P coatings onto wood as a substrate. The obtained composite not only can prevent EMI, but also is still lightweight and maintains a beautiful wood texture. Hence, its potential applications are promising in EMI field and decoration field.

Electroless metal deposition is a heterogenetic catalytic electron transfer reaction in which electrons are transferred from a reducing agent to metal ions at an interface. Therefore, nonmetallic materials such as plastics, glass, or wood have to be catalyzed to produce catalytic nuclei prior to electroless plating. These nuclei act as nucleation centers to initiate the subsequent autocatalytic stage. Good catalysis is of great importance to form a high-quality coating. Traditionally, palladium colloid served as the catalyst in electroless plating due to its high reactivity. However, because of the lack of chemical bonding forces between the colloid particles and the matrix, very small amounts of Pd colloidal particles fall off from the matrix. This results in the self-decomposition of the plating solution.

In the past few years, $PdCl_2$ has been used as an activator for electroless nickel or copper plating due to its controllability (Liu *et al.* 2010; Sun *et al.* 2012). With the objective of having a low-cost and simple activation process, many attempts have been made to employ Ni activation. Two methods have been widely studied. In one method, Ni^{2+} was loaded on the substrate and Ni^{o} was produced *via* thermal treatment at high temperatures (Li *et al.* 2006; Tang *et al.* 2009; Shao *et al.* 2007; Li and An 2008). In the other method, Ni^{2+} on the surface of the substrate was reduced to Ni^{o} with sodium borohydride ($NaBH_4$) (Lai *et al.* 2006; Hu *et al.* 2006; Gao and Huang 2007). In our previous research, a novel and simple activation method served as the preparation of Ni-P coating (Li *et al.* 2010). Because of the low-cost and simple method, we used this technique for depositing Ni-Cu-P coating on wood veneers. In this paper, the effect of $CuSO_4$ content on the metal deposition, elemental composition, phase structure, surface morphology, wettability, surface resistivity, and shielding effectiveness of coatings was investigated. The coating on the wood veneer was characterized by X-ray diffraction (XRD), scanning electron microscopy (SEM), and X-ray photoelectron spectroscopy (XPS).

EXPERIMENTAL

Materials

The substrates used were birch veneers with a thickness of 0.6 mm. The source of all chemicals (analytical reagent grade and used as received) was Tianjin Kermel Chemical Reagents Development Center. These included nickel sulfate [$NiSO_4 \cdot 6H_2O$], copper sulfate [$CuSO_4 \cdot 5H_2O$], sodium hypophosphite [$NaH_2PO_2 \cdot H_2O$], sodium citrate [$Na_3C_6H_5O_7 \cdot 2H_2O$], ammonium acetate [$CH_3COONH_4$], $NH_3 \cdot H_2O$, and deionized water.

The veneers were obtained from Harbin and polished by emery papers to remove fine fibers from the surface. The specimens used for measuring electrical conductivity and wettability had dimensions of 50 mm × 50 mm, and the specimens used for testing the electromagnetic shielding effectiveness were cut as shown in Fig. 1.

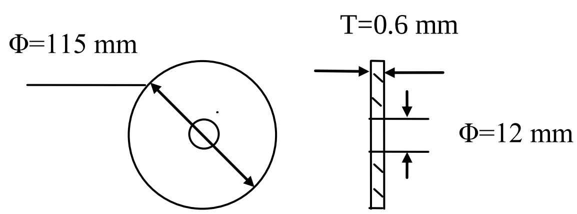

Fig. 1. Samples used for testing electromagnetic shielding effectiveness

Preparation of Wood-based Ni-Cu-P Composite

First, wood samples were dipped in $NaBH_4$ solution containing 5 g/L sodium hydroxide (NaOH) for a specific length of time at room temperature. Next, the veneers of $NaBH_4$ and NaOH loadings were left in open air for 1 min to allow for $NaBH_4$ diffusion into the inner pores. The samples were then put directly into the plating solution. The composition of the electroless bath is listed in Table 1. The pH of the bath was adjusted using $NH_3 \cdot H_2O$, and the operation temperature was controlled in an appropriate range.

Table 1. Composition of Electroless Ni-Cu-P Plating Solution

Chemicals	Content (g/L)
$NiSO_4 \cdot 6H_2O$	10~40
$CuSO_4 \cdot 5H_2O$	0.5~2.5
$NaH_2PO_2 \cdot H_2O$	20~50
$NaC_6H_5O_6 \cdot 2H_2O$	20~60
CH_3COONH_4	10~40

Measurement of Metal Deposition

The wood veneers were dried at 103 ± 2 °C to constant weight (G_0). Corresponding samples plated with Ni-Cu-P alloys were also dried at 103 ± 2 °C to constant weight (G_1). The metal deposition was calculated as

$$\textit{Metal deposition}\ (\text{g/m}^2) = (G_1 - G_0) / 0.005 \qquad (1)$$

where 0.005 is the total area of the sample (m^2).

Surface Resistivity and Shielding Effectiveness

The surface resistivity of the metallized wood veneers was evaluated using the Chinese National Military Standard method GJB2604-96. The shielding effectiveness (SE) of the metallized wood veneers was measured with an Agilent E4402B spectrum analyzer and standard butt coaxial cable line with a flange according to the Chinese industrial standard SJ20524-95. The *SE* value was calculated as,

$$SE \text{ (dB)} = -10 \times \lg (P_{out} / P_{in}) \quad (2)$$

where P_{out} and P_{in} are the incident and transmitted power, respectively.

Wettability of the Plated Veneers

The water contact angles were measured with 5 μL of deionized water droplets at room temperature, using an optical contact angle meter (Hitachi, CA-A). The water contact angles were obtained as averages of five measurements.

Characterization Methods

XPS was used for the elemental composition analysis of Ni-Cu-P coatings. XPS signals were recorded with a K-Alpha XPS Analyzer (ThermoFisher Scientific Company), using an Al K_α source. The surface morphology was characterized by scanning electron microscopy (SEM, Quanta 200). Specimens were not sprayed with gold prior to analysis. The phase structure of the coating was investigated by X-ray diffraction (XRD, Rigaku D/max2200 diffractometer) using a Cu K_α radiation generator operated at 1200 W (40 kV × 30 mA).

RESULTS AND DISCUSSION

Effect of $CuSO_4{\cdot}5H_2O$ Concentration on Metal Deposition

The $CuSO_4{\cdot}5H_2O$ concentration in the plating solution was found to be a key factor for electroless Ni-Cu-P alloys.

At pH value of 9.5 and operation temperature of 90 ^oC, the effect of $CuSO_4{\cdot}5H_2O$ concentration on the metal deposition is shown in Fig. 2. With increasing $CuSO_4{\cdot}5H_2O$ concentration from 0.6 g/L to 2.2 g/L, the metal deposition was decreased from 79.61 g/m^2 to 66.44 g/m^2.

When the bulk concentration of Cu^{2+} increased, the metal deposition decreased as a result of the deposited nickel being displaced by the copper ions. The competition between the oxidation of nickel and the reduction of copper ions can be understood using the constructed reaction of the redox couple, expressed as Eq. 3. Because of the coverage of Cu instead of Ni, the reaction slowed. Therefore, the metal deposition ostensibly decreased.

$$Cu^{2+} + Ni \rightarrow Cu + Ni^{2+} \qquad [E^{\ominus}_{25°C}(NHE) = 0.597\,V] \quad (3)$$

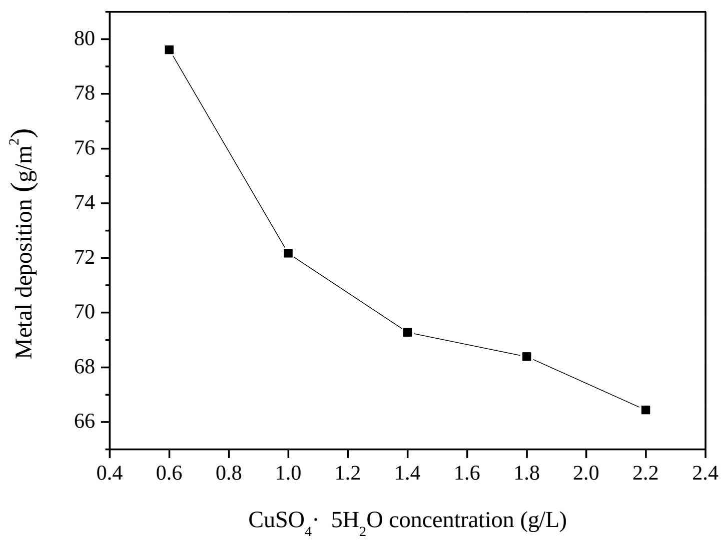

Fig. 2. Effect of $CuSO_4·5H_2O$ concentration on metal deposition (pH 9.5, temperature 90 ^oC)

Elemental Composition and Phase Structure

The $CuSO_4·5H_2O$ concentration had a significant effect on the elemental composition and phase structure of the Ni-Cu-P alloys. The effect of $CuSO_4·5H_2O$ concentration in the plating bath on the composition of Ni-Cu-P coatings is illustrated in Fig. 3. With increasing $CuSO_4·5H_2O$ concentration from 0.6 g/L to 2.2 g/L, the Cu content in the coating increased from 3.96 wt% to 20.73 wt%, while the Ni content decreased from 73.52 wt% to 63.00 wt% and the content of P reduced from 22.52 wt% to 16.27 wt%. A likely reason for this is that the Cu is preferentially deposited because its reduction potential is greater than that of the Ni and P, as shown by the following equations (4, 5, and 6):

$$Ni^{2+} + 2e^- \rightarrow Ni \qquad [E^{\ominus}_{25°C}(NHE) = -0.257\,V] \qquad (4)$$

$$Cu^{2+} + 2e^- \rightarrow Cu \qquad [E^{\ominus}_{25°C}(NHE) = 0.340\,V] \qquad (5)$$

$$H_2PO_2^{\,2-} + e^- \rightarrow P + 2OH^- \qquad [E^{\ominus}_{25°C}(NHE) = -1.820\,V] \qquad (6)$$

Figure 4 shows the XRD patterns of Ni-Cu-P-coated veneers with various copper content deposits. The peak in the curve at 2θ=22.43° is characteristic of cellulose in birch veneer. Furthermore, the peaks became smaller with lower copper content, indicative of thicker coatings being deposited on the veneer. For copper content below 20.73 wt%, a wide peak at 2θ=43.91° was observed, corresponding to the (111) plane of Ni-Cu. For deposits containing 20.73 wt% of copper, a sharp peak was obtained at 2θ=43.91°. In addition, a peak at 2θ=50.80° appeared and was attributed to Ni-Cu (200). This can be explained by the formation of a face-centered cubic Ni-Cu solid solution. An increase in the Cu content of such deposits can enhance their crystallinity. Therefore, their structure was amorphous at low Cu content and microcrystalline or crystalline at higher contents. Similar results were obtained by Guo *et al.* (2009) for a polyester fabric substrate.

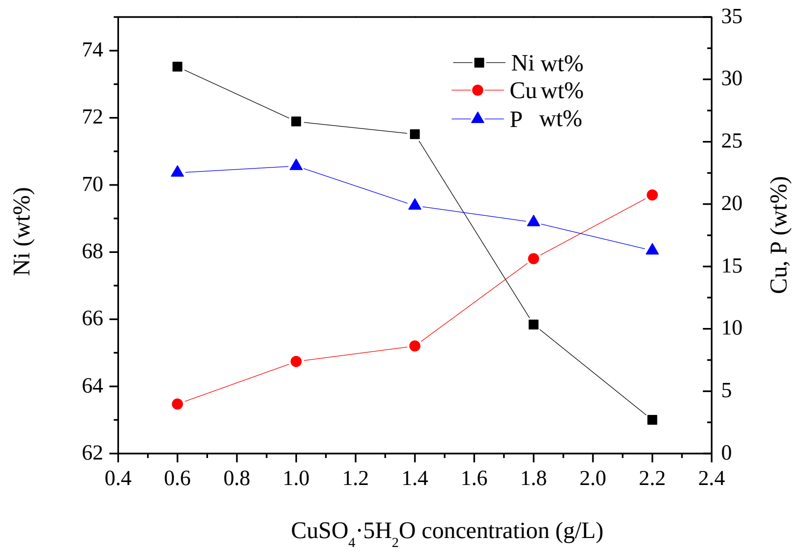

Fig. 3. Effect of $CuSO_4{\cdot}5H_2O$ concentration on elemental composition of the coating (pH 9.5, temperature 90 °C)

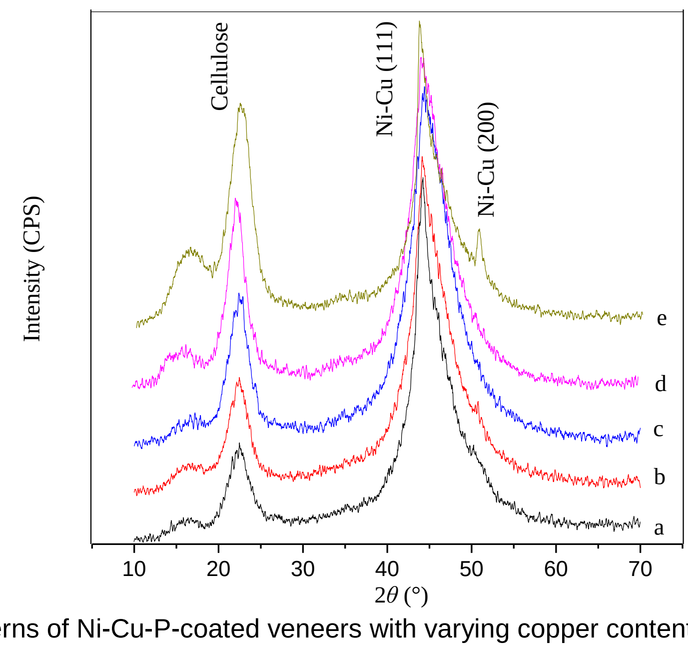

Fig. 4. XRD patterns of Ni-Cu-P-coated veneers with varying copper content: (a) 3.96 wt%, (b) 7.38 wt%, (c) 8.62 wt%, (d) 15.62 wt%, and (e) 20.73 wt%

Surface Morphology

The surface morphology of the Ni-Cu-P coating with varying copper content was investigated using SEM, as shown in Fig. 5. The surface morphology exhibited a nodular structure. Furthermore, as the $CuSO_4{\cdot}5H_2O$ concentration increased from 0.6 g/L to 2.2 g/L, the copper content in the deposits increased from 3.96 wt% to 20.73 wt% (Fig. 3) and the corresponding nodular structure became less conspicuous. A finer-grained structure was observed for the higher copper content specimen, as shown in Fig. 5e. The result agreed with the result obtained by Abdel Aal and Shehata Aly (2009) on the open cell stainless steel foam. Copper deposition played a key role in the reduction or inhibition of additional nodule sites. The nodular deposition in a coating depended on both the nucleation rate and the growth of the deposit. The introduction of copper to the deposit held up the growth of the nodules by inhibiting further growth. This is probably the main reason for the smooth and compact Ni-Cu-P coatings.

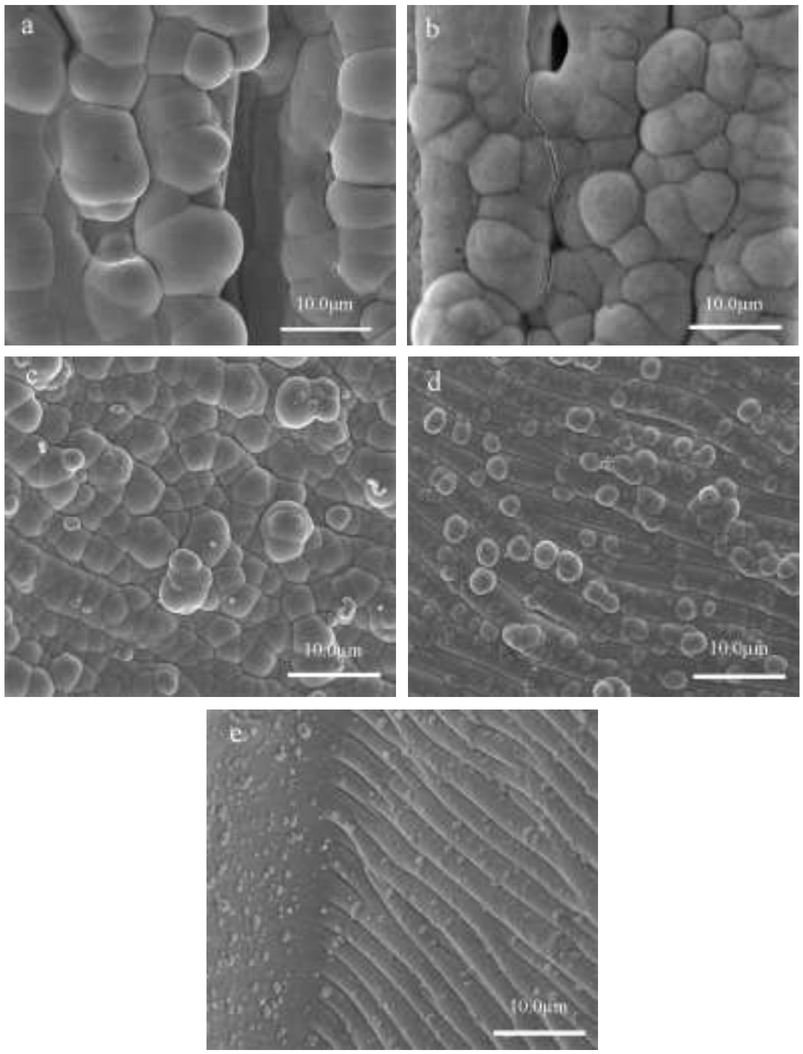

Fig. 5. SEM images of Ni-Cu-P-coated veneers with varying copper content: (a) 3.96 wt%, (b) 7.38 wt%, (c) 8.62 wt%, (d) 15.62 wt%, and (e) 20.73 wt%

Wettability

The static contact angles were measured to evaluate the wettability of the as-prepared Ni-Cu-P alloy surfaces. Figure 6 shows the effect of $CuSO_4 \cdot 5H_2O$ concentration on contact angle of the coating. As $CuSO_4 \cdot 5H_2O$ concentration was increased from 0.6 g/L to 2.2 g/L, the corresponding contact angle increased from 99.5° to 116.5°. The pristine veneer was a hydrophilic material and showed a water contact angle less than 90°. Therefore, the wettability of Ni-Cu-P-coated veneer was reduced, indicating some enhancement of hydrophobic properties. Note that the wettability of a solid surface mostly depends on the geometrical and elemental composition. Hence, the morphological and compositional differences caused by $CuSO_4{\cdot}5H_2O$ concentrations should be taken into consideration.

It can be seen in Fig. 5 that the formed microstructure was similar to the binary micro-structure in the lotus effect. In addition, the introduction of copper into electroless deposited Ni-P decreased the free energy of the surface, leading to the decrease in wettability. The highest wt% of Cu, 20.73, was obtained at a $CuSO_4{\cdot}5H_2O$ concentration of 2.2 g/L. Therefore, the corresponding hydrophobic property of the coating was markedly increased.

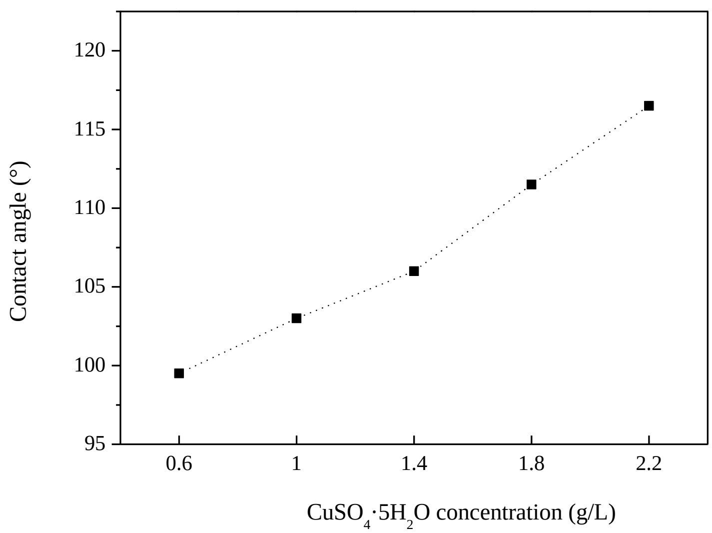

Fig. 6. Effect of $CuSO_4{\cdot}5H_2O$ concentration on contact angle of the coating (pH 9.5, temperature 90 ^oC)

Surface Conductive and Electromagnetic Shielding Performances

The electromagnetic shielding results of the pristine and plated birch veneer are illustrated in Fig. 7. The raw birch veneer, with surface resistivity of more than 10^{10} Ω/cm^2, had no shielding performance, as shown in Fig. 6f. With increasing $CuSO_4{\cdot}5H_2O$ concentrations from 0.6 g/L to 2.2 g/L, the electromagnetic shielding effectiveness of the plated veneers was lower at frequencies from 9 kHz to 1.5 GHz (Fig. 7, a, b, c, d, and e). However, the lowest shielding effectiveness value exceeded 30 dB in all frequencies. According to Schelkunoff's theory, better conductivity in Ni-Cu-P-coated veneers resulted in a higher level of SE (Wu *et al.* 2002; Guo *et al.* 2009).

As shown in Table 2, the surface resistance decreased gradually with lower $CuSO_4{\cdot}5H_2O$ concentration, indicative of improvement in conductivity. At a $CuSO_4{\cdot}5H_2O$ concentration of 0.6 g/L, the surface resistivity reached its lowest value of 367.5 $m\Omega/cm^2$ and the corresponding shielding effectiveness value was the highest, exceeding 60 dB in all frequencies.

In general, a material is regarded as an effective shield when the shielding value exceeds 35 dB. Therefore, the Ni-Cu-P-coated veneers can meet some EMI shielding application requirements.

Table 2. Effect of $CuSO_4 \cdot 5H_2O$ Concentration on Surface Resistivity

$CuSO_4 \cdot 5H_2O$ concentration (g/L)	0.6	1.0	1.4	1.8	2.2
Surface resistivity (mΩ/cm^2)	367.5	438.9	528.2	572.3	580.9

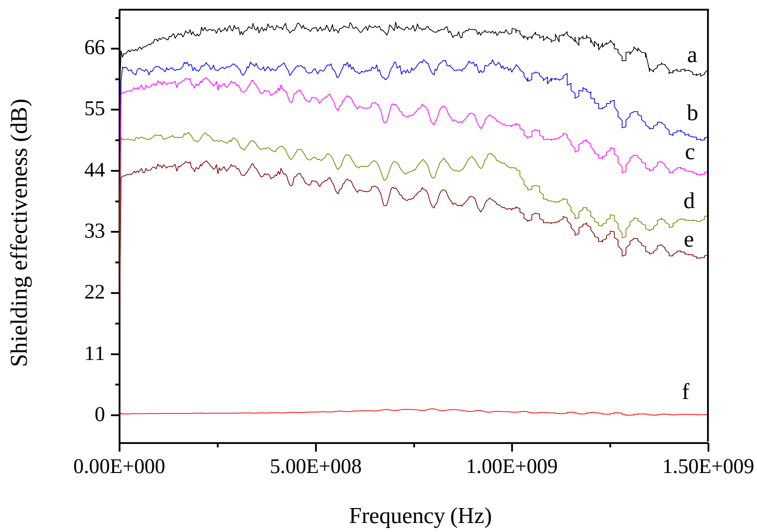

Fig. 7. Shielding effectiveness of Ni-Cu-P-coated veneers with different $CuSO_4 \cdot 5H_2O$ concentrations: (a) 0.6 g/L, (b) 1.0 g/L, (c) 1.4 g/L, (d) 1.8 g/L, (e) 2.2 g/L, and (f) pristine veneer

CONCLUSIONS

The $CuSO_4 \cdot 5H_2O$ concentration in the plating bath had a notable effect on the metal deposition, elemental composition, phase structure, surface morphology, wettability, surface resistivity, and shielding effectiveness of the coatings. The conclusions are summarized as follows:

1. With increasing $CuSO_4 \cdot 5H_2O$ concentrations from 0.6 g/L to 2.2 g/L, the metal deposition decreased from 79.61 g/m^2 to 66.44 g/m^2.
2. The copper content in the coating increased from 3.96 wt% to 20.73 wt%, while the nickel content decreased from 73.52 wt% to 63.00 wt% and the phosphorus content was reduced from 22.52 wt% to 16.27 wt%.
3. XRD results showed that the structure of the coatings was amorphous at low Cu content and microcrystalline or crystalline at higher contents.
4. SEM analysis showed that a finer-grained structure was attained with higher copper contents.

5. The wettability tests demonstrated that the Ni-Cu-P coating induced increased hydrophobicity.
6. The lowest surface resistivity was 367.5 mΩ/cm^2, and the electromagnetic shielding effectiveness exceeded 60 dB at frequencies from 9 kHz to 1.5 GHz.

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