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Versatile Coffee Carbon Dots as Lead (ii) and Copper (ii) ion Fluorescence Detectors and Copper Corrosion Inhibitor

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ABSTRACT

A simple and green tandem hydrothermal and pyrolysis method was developed for the synthesis of blue fluorescence carbon dots from coffee green bean powder and urea as raw materials. The carbon dots were characterized by ultraviolet visible spectrophotometer, Fourier-transform infrared spectroscopy and ocean optics spectrophotometer. The carbon dots had a bright blue emission centered at 505 nm under UV light with the excitation wavelength at 360 nm. The carbon dots also showed a quenching effect with lead (ii) ions and copper (ii) ions. Furthermore, a method for copper (ii) and lead (ii) ion detection in aqueous solution was developed with acceptable linearity of 0.961 and 0.951 respectively and selectivity to some common ions. The carbon dots also demonstrated the ability to act as copper corrosion inhibitors in 1% sodium chloride solution showing an inhibition efficiency of 76.98 % at 1000 mg/L. It is worth noting that the synthesized fluorescent carbon dots are eco-friendly and inexpensive. They could find application in chemical sensing and copper corrosion inhibition in salt environments.

Keywords : Carbon dots, Pyrolysis, Quenching, Chemical sensing and Corrosion inhibitor.

I. INTRODUCTION

Fluorescent carbon dots (C-dots) are one of the quintessential classes of carbon-based nanomaterials less than 10 nm in size that have attracted attention due to their remarkable properties such low toxicity, biodegradation ability, high water solubility, good biocompatibility, and unique photoluminescence properties (Song *et al*, 2017; Jiang *et al*. 2015; Pan *et al*. 2015; Tao *et al*. 2011). These properties support their application in biomedicine, catalysis optoelectronic devices, corrosion inhibition and metal sensing (Roy *et al*. 2015; Yang *et al*. 2014). C-dots can be synthesized from both synthetic and natural precursors. However, natural precursors are favored due to their large abundance These include; glucose (Yang *et al*. 2011), bread, jaggery and sugar caramel (Sk *et al*. 2012), banana (De *et al*. 2013), grass (Liu *et al*. 2012), orange juice (Sahu *et al*. 2012), pear juice (Liu *et al*. 2018), Nescafe original instant coffee (Jiang *et al*. 2014) etc. Synthetic methods of C-dots include arc discharge, ultrasonic/ microwaves, laser ablation, electrochemical synthesis and hydrothermal treatments (Mishra *et al*. 2018.).

Numerous fluorescent systems such as fluorescence quenching, energy transfer and the sensitivity of fluorescent spectra to molecular environments support the application of carbon dots in bio and chemo sensing. Fluorescence detection offers a fast response and facile to carry out. However, fluorescence detection with a high selectivity and sensitivity still needs more research (Liu *et al*. 2018). C-dots are widely applied in metal detection also partly due to the affinity and the coordination of the varied metal ions with the aromatic residues that make up the carbon dots (Tan *et al*. 2014). Copper II ions (Cu^{2+}) are a vital trace element.Nonetheless, elevated concentrations beyond tolerable limits of Cu^{2+} is toxic to human cells and could trigger many severe disorders and diseases like kidney failure, Wilson disease and infant liver damage (Mubarak *et al*. 2017). Thus, the study, development and fabrication of swift and efficient detection for Cu^{2+} is highly imperative. An additional noxious heavy metal that requires swift detection is lead (ii) ions (Pb^{2+}) because too much exposure to lead sparks lead poisoning, which affects multiple body systems most especially in children, and when lead accumulates in the brain liver kidney and bones the effects are lethal (Wani *et al*. 2015). Fluorescent carbon dots from different sources were efficaciously used in the detection of Cu^{2+} due to their photoluminescence property (Cotruvo *et al*., 2015) and table sugar derived Carbon dot have also been reported as naked eye sensors for toxic Pb^{2+} ions (Ansi *et al*. 2008).

Most corrosion inhibitors applied regularly are organic compounds containing some electronegative atoms Nitrogen (N), Sulphur (S), Phosphorus (P), Oxygen (O) and multiple bonds, which have free electron pairs to form a bond with the metal surface (Sigurcik *et al*. 2015; Abd El Haleem *et al*. 2013; Doner *et al*. 2013). Today, the inhibition efficiency of many organic compounds has been reported. These include; imidazoles, triazoles, ionic liquids and Schiff bases (Cui *et al*. 2017). However, due to concerns on environmental safety and human health, the application of some of those organic inhibitors is limited due to their toxicity (Quiang *et al*. 2017). Robusta coffee beans contain more caffeine (hence more nitrogen content) compared to Arabica coffee (Dias *et al* .2015; BPS, 2019). This implies that robusta offers a better choice of coffee for the synthesis of carbon dots that can be applied as metal corrosion inhibitors.

In this study, a simple synthesis of carbon dots using coffee and urea by tandem hydrothermal and pyrolysis method was established. The absorption, functional group characterizations and fluorescence property of the prepared carbon dots were investigated. The fluorescence of C-dots could be directly quenched by Cu^{2+} and Pb^{2+} linearly in the concentration range from 0.1-50.0 mg/L and 5.0 -50 µg/L respectively. The C-dots could inhibit the corrosion of copper by 76.98 % at 1000 mg/L in 1% sodium chloride solution.

II. METHODS AND MATERIAL

A. Raw materials, reagents and apparatus

Robusta Coffee (*Coffea caneophora*) was obtained from the local farmers group (Koperasi Kopi Bogor), Ethanol (96%), acetone (99.5%), copper, coupon (99.9%) , sulphuric acid (98%), potassium bromide (99%) copper II sulphate, Sodium chloride Potassium chloride, Iron II chloride, lead II nitrate, Urea. All the reagents were purchased from commercial sources and used without any further purification, Barnstead Furnace 47900, 365 nm UV lamp, 1700 UV-Vis Spectrophotometer Shimadzu in Laboratory Bersama, Chemistry Department of IPB, Ocean Optics Spectrophotometer USB 4000 in the Spectroscopy Laboratory of the Department of Physics, IPB, FTIR Spectrophotometer in Laboratory Bersama Chemistry department IPB and a potentiostat.

B. Synthesis of Carbon dots from *coffea caneophora*

The Carbon dots were prepared by tandem hydrothermal treatment and pyrolysis of green coffee powder and urea. In a typical synthesis, 3.0 g coffee

and 1.2 g urea were dissolved in 20 mL of deionized water (Shi *et al.* 2019). The sticky cloudy solution was poured into 30 mL crucible as a reactor vessel. The reactor vessel was then wrapped in Aluminium foil, placed in a furnace and heated at 220 °C for 5 hours. After cooling down to room temperature, the black residue was dissolved in water and carbon dots were obtained by removing larger particles through centrifugation at 5000 rpm for 15 minutes. The supernatant was then evaporated at 80 °C to obtain the highly fluorescent blue carbon dots.

C. Characterization

The absorption spectra of the 100 mg/L of carbon dots in aqueous solution were recorded with a Shimadzu UV-Vis Spectrometer. The absorbance measurements were carried out at a wavelength of 200-700 nm with a 0.5 nm wavelength interval to obtain the absorption spectrum. The FTIR spectrum of the C-dots was recorded on a Nicolet FTIR spectrometer (Impact-410, Madison, USA) using a KBr pellet. A total of 20 mg of C-dot was mixed with 0.2 g KBr and finally all the fluorescence intensity measurements were carried out on an ocean optics spectrophotometer USB 40000 at a wavelength of 400-700 nm.

D. Fluorescence intensity measurement of Cu^{2+} ions

Various concentrations of Cu^{2+} solutions namely; 0.1, 0.5, 1.0, 5.0, 10.0 and 50.0 mg/L were added separately to a cuvette containing 1.5 mL of carbon dots solution. The solution was vigorously shaken and immediately transferred to the fluorescent measurement with the excitation wavelength of 360 nm. The fluorescence emission spectra of carbon dots were recorded from 390 to 600 nm and the fluorescence intensity was monitored at 505 nm. The procedure was repeated with different concentrations of Pb^{2+} namely; 5, 10, 15, 20, 50 µg/L.

E. Testing selectivity of the carbon dots

To assess the selectivity of carbon dots quenching toward Cu^{2+} ion and Pb^{2+}, interference assays were performed under identical conditions using five 20 mg/L metal ions potassium ions (K^+), sodium ions (Na^+), Iron II ions (Fe^{2+}) copper II ions and lead II ions.

F. Preparation of electrode *and solutions*

The copper coupon was abraded with silicon carbide abrasive papers, degreased with ethanol and acetone, washed ultrasonically with deionized water and lastly dried at room temperature. The corrosion medium was prepared with and without various concentrations (blank, 200, 400, 500, 600, 800 and 1000 mg/L) of carbon dots. The 1 % sodium chloride was prepared by dissolving 1 g of sodium chloride crystals in 100 g of deionized water. The experiments were carried out at room temperature and freshly prepared solutions were used for each set of experiments.

G. Weight loss measurements.

The weight loss procedures were carried out according to the ASTM standard G31-72. Samples with a dimension of 1.0 cm × 1.0 cm × 0.02 cm were immersed in 1 % sodium chloride solution in a 20 mL vessel with and without various concentrations of the carbon dots for 14 days. The samples were then removed and washed with deionized water, dried and weighed at various time intervals. The average value of the weight loss obtained was used for calculations. The corrosion rate (v_{corr}, mils penetration per year) and the inhibition efficiency (ηw, %) of copper in different concentration of coffee carbon dots in 1 % sodium chloride solution and without addition of carbon dots at room temperature were calculated.

H. Electrochemical measurements

The studies were conducted in 1% NaCl test solution using three electrodes setup in one compartment cell. A platinum sheet was used as the counter electrode and Ag/AgCl as the reference. The working electrode was copper (99.99% purity) cylindrical rod embedded in a resin with one bottom surface (3.34 cm^2) exposed The DY 2300 Potentiostat/Galvanostat was utilized. The detection was carried out at room temperature using the linear sweep voltammetry technique with a

potential range of – 2.0 V to 0.5 V, a scan rate of 0.01 V/s, and a sensitivity of 0.001.

III.RESULTS AND DISCUSSION

A. Synthesis and characterization of the carbon dots

The carbon dots were prepared by carbonization of urea and green bean coffee powder. Direct hydrothermal treatment followed by pyrolysis in a furnace at 220 °C for 5 hours produced a black-coloured fine powder with a high solubility in water. The aqueous solution of C-dot exhibited a light brown colour under daylight and emits intense blue light under 365 nm UV light exposure. However there was no emission with a solution of coffee and urea therefore, the obtained results qualitatively unveiled the presence of carbon dots (Figure 1).

Figure 1. Preparation process of the carbon dots from coffee and urea

B. Spectroscopic characterization of the carbon dots

The UV-Vis absorption spectrum of the aqueous solution of carbon dots showed a strong absorption peak at around 280 nm. The peak at 280 nm (Figure 2).is attributed to n-π* transition of the C=O bonds and that indicate the presence of carboxyl group on the carbon dots surfaces The photoluminescence spectra of synthesized carbon dots showed a maximum emission at 505 nm at different carbon dot concentrations. The origin of this strong emission is probably due to the presence of several functional groups on the surface that act as emissive traps for the electronic transition (De *et al.* 2013).

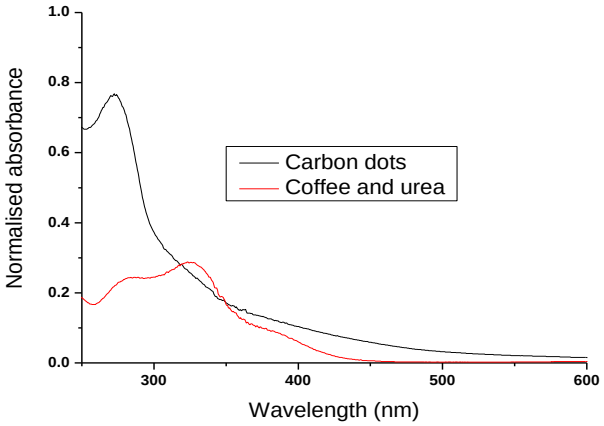

Figure 2. The UV-vis absorption spectra of carbon dots and their precursor:

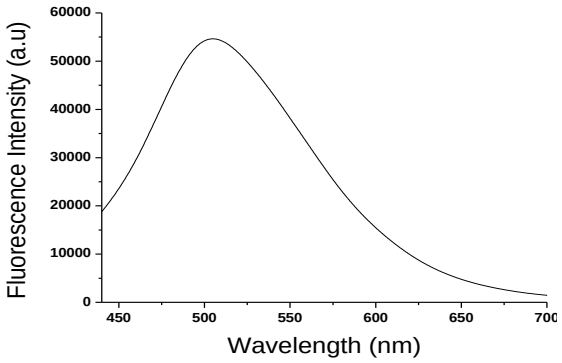

Figure 3. Fluorescence spectra of carbon dots

C. FTIR analysis

This was carried out to elucidate the functional groups and surface functionalization of C-dots. The carbon dots exhibited the presence of many hydrophilic groups at the frequencies of 3237, 1711 and 1381 correspond to the presence of –OH or –NH_2, C=O, and C-N respectively. The presence of these functional groups ensured that the synthesized C-dots have excellent water solubility (Sciortino *et al.*, 2017) (Figure 4).

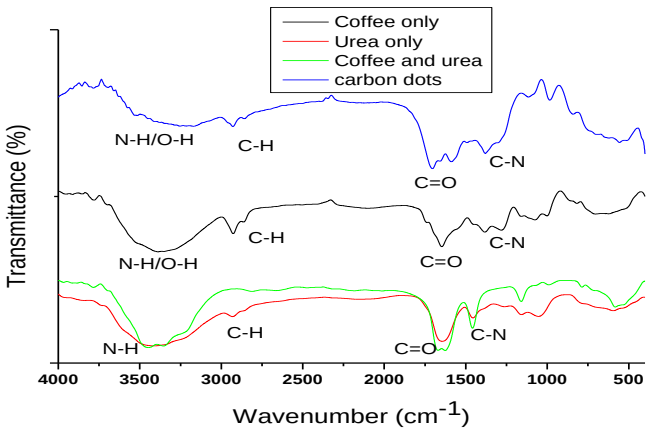

Figure 4. The FTIR spectra of the carbon dots

D. Detection of Cu^{2+} and Pb^{2+} ions using carbon dots

The emission spectra of a fixed amount of carbon dots (100 mg/L) in response to the concentrations of copper (ii) and lead (ii) ions were plotted. They showed concentration dependent trend and since this was a quenching process, a standard Stern-Volmer relationship was adopted to model the intensities recorded at various wavelength of the maximum emissions.

$$\frac{F_O}{F} = K_{sv}[C] + 1$$

Where both F_o and F denote the fluorescence intensities of carbon dots before and after adding the metal ions. Ksv denotes the Stern-Volmer quenching constant. From the equation, a linear relationship between the fluorescence intensities before and after addition of metal ions was obtained

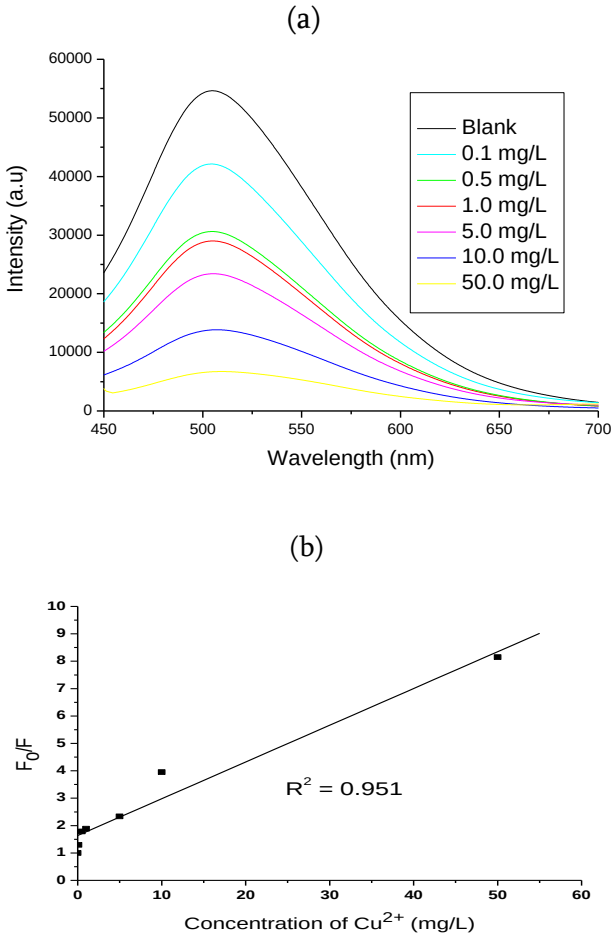

Figure 5 (a) The fluorescence spectra of the carbon dots at various Cu^{2+} concentrations (b) the linearity curve from the Stern-Volmer equation.

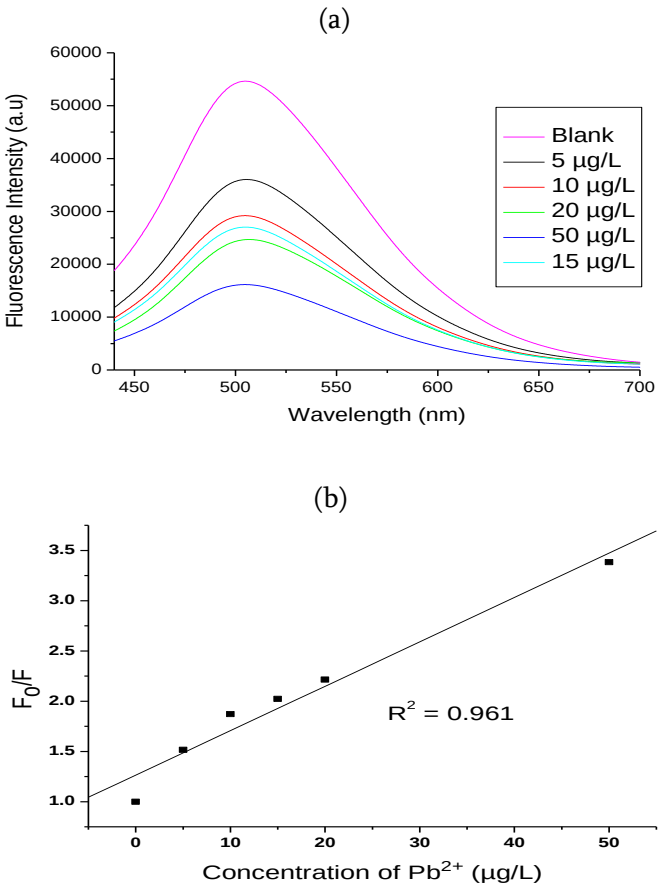

Figure 6: (a) The fluorescence spectra of the carbon dots at various Pb^{2+} concentrations: (b) the linearity curve the from the Stern-Volmer equation.

The limits of detections of the synthesized carbon dots were calculated from $\frac{3\sigma}{m}$ where σ denotes the standard deviation of the carbon dots corrected blank signals (n=4) and m denotes the slope of the linear curve from the Stern-Volmer equation. The R^2 of the linear curve of copper (ii) ions was 0.951 and that of lead (ii) ions was 0.961. The Non-specificity of the carbon dots towards lead (ii) ions and copper (ii) ions can be contributed to the effect of pyrolysis which degrades the surface of carbon dots leaving it bare to the environment hence can come into contact with the metal ions around (Tan *et al.* 2014). However, different metal ions induce different degree of disturbances on the material fluorescence process depending on their electronic configuration and the interaction affinities. Hence surface modification of carbon dots can be done improve their specificity.

The limit of detection of copper (ii) ions was 0.447 mg/L and that of lead (ii) ions was 1.358μ g/L.

E. Selectivity of the carbon dots

The Pb^{2+} and Cu^{2+} ions could quench the fluorescence intensity of the carbon dots but not any other ions. The addition of 20.0 mg/L interferents did not change the fluorescence intensity of carbon dots. However, only 20.0 mg/L of Cu^{2+} and Pb^{2+} effectively quench the fluorescence intensity of carbon dots however the lead II ions quench the fluorescence intensity of the carbon dots more than other ions. (Figure 7) The reason for the above results can be because K^+, Na^+ are diamagnetic and lack the ability of paramagnetic quenching mechanism with the carbon dots. While iron (ii) Fe^{2+} when bonded to some ligands can form a diamagnetic compound because of the creation of a low spin situation.

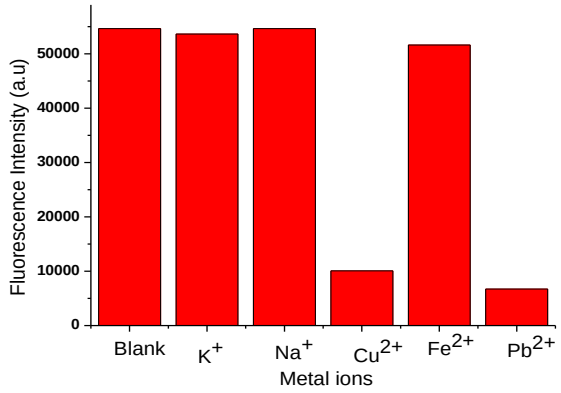

Figure 7. Selectivity of the quenching assay using carbon dots for Cu^{2+} and Pb^{2+}.

F. Corrosion inhibition studies

Weight loss measurements

Using equations (1) and (2), the inhibition efficiency of the different concentrations of carbon dots was calculated and the corrosion rate of the copper piece.

$$\nu_{corr} = \frac{K\,x\,W}{A\,x\,T\,x\,D} \qquad (1)$$

$$\eta_w\,(\%) = \frac{\nu^0_{corr} - \nu_{corr}}{\nu^0_{corr}}\,x\,100 \qquad (2)$$

Where K= 8.76×10^4, W is the copper weight loss in grams, A is the total surface area of copper (cm^2), T is the immersion time (hours), D is the density of the test specimen ν^0_{corr} and ν_{corr} are the blank corrosion rates and with inhibitor, respectively.

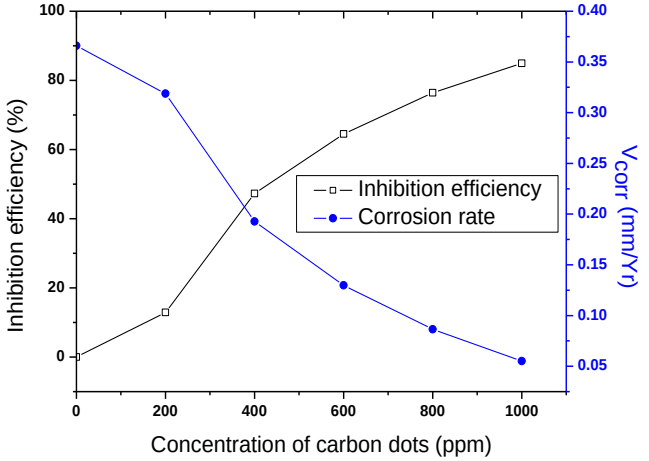

Figure 8. The corrosion rates and inhibition efficiencies for copper in different concentrations of coffee carbon dots in 1% NaCl solution.

The corrosion rate of copper decreased with the decreasing of inhibitor concentration from 0 to 100 mg/L implying that the coffee carbon dots have a corrosion inhibition activity. Therefore in aqueous solution, the π electrons and the lone electron pair oxygen and nitrogen atoms donate to the unoccupied d orbital of copper to form strong coordinate bond thereby facilitating the adsorption of the inhibitor molecules to metal surfaces (Qin *et al.*, 2011). The highest inhibition efficiency was 84.94% at the carbon dot concentration of 1000 mg/L in 1% sodium chloride solution.

G. Potentiodynamic polarization measurements

The potentiodynamic polarization measurements were carried out. The current–potential curves for copper in sodium chloride solutions were obtained in the absence and presence of various concentrations of the coffee carbon dots at room temperature. The obtained data was converted into a Tafel curves (Figure 9) in order to obtain the equation of lines on cathode and anode (Table 1). The equation of the two equations of the line produced a value of corrosion current. Corrosion currents were the basis for calculating the efficiency of inhibition.

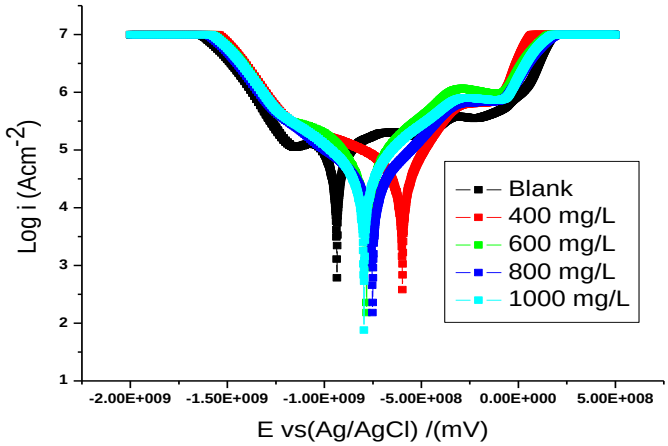

Figure 9. The Tafel polarization curves of copper in 1% NaCl at different carbon dot concentrations.

From Table 1 below, it was observed that the maximum inhibition efficiency was 76.98% at the inhibitor concentration of 1000 mg/L. However the increase in inhibitor concentration led to increase in the inhibition efficiency due to the increasing number of molecules inhibitor molecules adsorbed on the metal surfaces. Also the surface coverage (θ) increased with the increasing inhibitor concentration since the protection of the metal increases (Anindita *et al.*, 2018)

Sample (mg/L)	Tafel Equation		E (mv)	Y	I (mA)	Θ	Corr. Rate	IE
	Cathode	Anode						
Blank	Y = -0.0047x ─ 5.7879	Y = 0.0045x + 2.8683	-940.9	-1.3657	0.043	1	0.1492	0
400	Y = -0.0035x ─ 3.0199	Y = 0.0072x + 1.6899	-440.2	-1.4792	0.033	0.233	0.1145	23.26
600	Y = -0.0052x ─ 5.5516	Y = 0.0068x ─ 3.2926	-737	-1.7192	0.019	0.5581	0.06592	55.81
800	Y = -0.006x ─ 6.5246	Y = 0.0063x + 2.8067	758.6	-1.973	0.0106	0.627	0.03677	75.34
1000	Y = -0.0059x ─ 5.9374	Y = 0.0069x + 2.5963	-666.7	-2.004	0.0099	0.77	0.03435	76.98

Table-1: The copper corrosion parameters gathered from potentiodynamic polarization curves.

IV. CONCLUSION

In this study, an efficient and facile green synthetic method for highly fluorescent carbon dots using urea and green bean coffee powder as carbon source was investigated. The synthesized carbon dots exhibited a bright blue colour under UV light. The carbon dots also exhibited a high florescence emission at 505 nm after characterization with fluorescence spectra and absorption at 280 in UV-vis spectra. The as-prepared carbon dots exhibited quenching effect with Cu^{2+} ion and Pb^{2+} and a method for Cu^{2+} ion detection and Pb^{2+} in water can be developed with required selectivity. Furthermore, the coffee carbon dots could inhibit copper corrosion for as high as 77% in 1% sodium chloride solution. Therefore, coffee is an eco-friendly raw material for the preparation of carbon nanoparticles and the synthesized fluorescent carbon dots could be useful in fabricating sensors for metal detection and copper corrosion inhibitors in salt environments

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