



## Adsorption behaviour of copper and gold glycinate complexes in alkaline media onto activated carbon. Part 1: Isotherms



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### ABSTRACT

Alkaline glycine/glycinate solutions have been shown to have significant potential in the leaching of gold and copper from ores, concentrates, and wastes bearing these metals. An attempt was made to understand the adsorption behaviour of the gold glycinate complex onto activated carbon in the presence of copper for a pure alkaline glycine/glycinate system, after the gold has been dissolved using hydrogen peroxide as an oxidant and caustic soda as pH modifier. The adsorptive behaviour under various process conditions was evaluated using equilibrium loading isotherms. The linear regression ( $R^2$ ) values for the Freundlich isotherm for all tested parameters are close to unity, suggesting the model to be well suited for gold glycinate adsorption onto activated carbon. The maximum adsorption capacity of the gold complex on the activated carbon was determined as a function of free glycine concentration, solution pH, initial gold and copper concentration and calcium chloride salt concentration using synthetic solutions. The adsorption capacity increased with increasing free glycine, copper and calcium chloride concentrations, but decreased with increasing solution pH and initial gold concentration. This information can be used (with adsorption kinetics) to design and evaluate Carbon-in-Pulp (CIP), Carbon-in-Leach (CIL) and Carbon-in-Column (CIC) circuits for gold when copper may be present.

### 1. Introduction

The increased interest in non-cyanide technology for gold ore processing can be attributed to a number of reasons. From an environmental perspective, cyanide is toxic, and the adverse environmental impacts resulting from its discharge have led to stringent jurisdiction regarding its usage (Eisler et al., 1999; Jeffrey et al., 2002). In some parts of the world, the use of cyanide for gold mining is prohibited while in other places approval for any gold cyanidation project is proving to be extremely difficult (Mudder and Botz, 2004). Geologically, due to the growing demand for gold, exploitation is shifting from cyanide-amenable ores to increasingly refractory or polymetallic ores, increasing the need for alternative processing routes (Hiskey and Atluri, 1988; La Brooy et al., 1994; Breuer et al., 2005; Dai et al., 2012; Jeffrey et al., 2002). Ammonia, thiocyanate, thiourea and thiosulfate are some of the alternative lixiviants studied thus far (Pyper, 1981; Aylmore and Muir, 2001; Grosse et al., 2003; Molleman and Dreisinger, 2002; Rezai and Peikary, 2002; Muir and Aylmore, 2004). More recently, the ability of glycine to leach gold and gold-copper ores was shown by a number of researchers (Perea and Restrepo, 2018; Eksteen and Oraby, 2015;

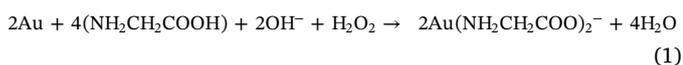
Oraby and Eksteen, 2014; Eksteen et al., 2017a,b; Tanda et al., 2017; Barton et al., 2018). As a leaching agent, glycine has environmental, technical and economic advantages over cyanide. It is environmentally benign and stable, yet easily biologically destructible under neutral and mild acidic conditions, but quite stable compared to sodium cyanide under alkaline conditions. From an economic perspective, apart from it being cheaper than cyanide (the bulk cost of glycine and NaCN respectively is around USD 1000–1800 per tonne (FOB, Feed Grade glycine) versus USD 2000 - USD2500 per tonne (FOB, Industrial grade sodium cyanide, 98%)) at the time of writing and compared to many other alternative plausible lixiviants, glycine can easily be recovered, regenerated and reused. Cyanide tends to convert to cyanate, thiocyanate, volatile HCN, and ferrocyanide, making recovery and reuse uneconomical in many cases. To reduce cyanide consumption of oxide gold-copper ores, these ores are conventionally first acid leached prior to neutralisation and cyanidation. However, being an alkaline leach, glycine leaching of gold ores is less susceptible to acid consuming gangue and co-leaching of Fe, Mn, Mg and Al (Eksteen and Oraby, 2015; Tanda et al., 2017). Glycine therefore allows the leaching of copper and gold at the same pH.

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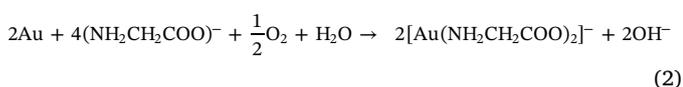
E-mail address: [jacques.eksteen@curtin.edu.au](mailto:jacques.eksteen@curtin.edu.au) (J.J. Eksteen).

The alkaline glycine system is characterised by simple chemistry (i.e. not prone to the side reactions as cyanide). Glycine, also known as aminoethanoic/aminoacetic acid, is the simplest neutral amino acid made up of one amine (–NH) and one carboxyl (COOH<sup>–</sup>) functional groups. It can, depending on solution pH, exist as either a glycinium cation (<sup>+</sup>H<sub>3</sub>NCH<sub>2</sub>COOH), a glycinate anion (H<sub>2</sub>NCH<sub>2</sub>COO<sup>–</sup>) or zwitterion (<sup>+</sup>H<sub>3</sub>NCH<sub>2</sub>COO<sup>–</sup>). In the glycine leach system, owing to the alkaline nature of the leach, at pH 10.5–11, the most predominant specie is the glycinate anion (Rega et al., 1998). The leaching process involves metal dissolution in a neutral to alkaline heated (23–60 °C) glycine solution in the presence of a soluble oxidant (Eksteen and Oraby, 2015). The oxidants successfully employed in previous studies include dilute hydrogen peroxide (Eq. (1)) and dissolved oxygen (Eq. (2)). The possible mechanism of gold dissolution in glycine has been proposed as:

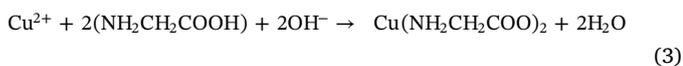
a) Using hydrogen peroxide as oxidant



b) Using oxygen as oxidant



The leaching behaviour of various copper oxide minerals in alkaline glycine systems has also been studied and is reasonably well understood. Results from studies indicate that, at optimum conditions (pH 11, glycine to copper molar ratio of 8:1), the copper extraction was over 80.0% for azurite, malachite and cuprite and < 20.0% for chrysocolla in 24 h. The copper complexing mechanism is shown in Eq. (3) (Tanda et al., 2017):



Methods for gold recovery from resultant polymetallic leachates include precipitation, ion exchange, solvent extraction and adsorption. Preliminary studies have confirmed that activated carbon adsorb gold glycinate complexes extremely well, possibly better compared to gold cyanide (Oraby and Eksteen, 2015). NaSH precipitation and solvent extraction were exploited for copper glycinate recovery and it was found out that it was possible to selectively recover over 99% copper using either method (Eksteen et al., 2016). Based on these results, activated carbon adsorption, currently being the main technique applied in cyanide systems for recovery of gold from cyanide leach liquors, is the starting point of our consideration for the newly developed glycine system. Industrially, it is preferred over other methods since it is cost effective, simple and has a high removal capacity owing to the large surface area of the carbon (Wan and Miller, 1990; Tu et al., 2011). Metals recovery from glycine leachates is still at an early stage and to date, fundamental understanding concerning the adsorption behaviour of metal glycinate complexes on activated carbon under different process conditions is lacking.

The interaction between an adsorbate and an adsorbent for any new system is best understood through the use of adsorption equilibrium information. Adsorption isotherms, their proper understanding and interpretation, are critical in the effective design and optimisation of any adsorption system. The Freundlich and Langmuir adsorption isotherms have been used to describe gold cyanide adsorption onto activated carbon from synthetic solutions with the former giving a much more accurate description of the equilibrium gold adsorption behaviour (Davidson, 1974; Cho et al., 1979). Previous studies have shown that these isotherms are well-suited for low concentration systems (Dixon

et al., 1976; Van Deventer, 1986; Woollacott, 1990) as encountered in most gold leaching operations from its ores. This is usually the case in the carbon-in-pulp (CIP) and the carbon-in-leach (CIL) processes, the two most dominant downstream processes used for gold extraction after cyanide leaching.

The main objective of the current study was to model the adsorption behaviour of metals onto activated carbon from a pure glycine system by generating isotherm parameters. The effects of pH, glycine concentration, ionic strength, initial gold and copper concentrations on the competitive loading of gold and copper glycinate complexes onto activated carbon are evaluated below.

## 2. Material and methods

### 2.1. Sorbent characterisation

The adsorbent used in this work was fresh activated carbon: PICA-GOLD® G210AS produced by Jacobi Carbons. The sorbent was manufactured from raw high grade coconut shell as per specifications from the manufacturer. Prior to use, the activated carbon was pulverised, mechanically sieved to –45 µm, oven dried at 70 °C for 16 h and preserved at ambient temperature in a desiccator. The surface area of the carbon as determined by the N<sub>2</sub>BET method was 840.6881 ± 46.5496 m<sup>2</sup>/g. The BET surface area of the carbon enables the loading capacity to be presented as grams of gold per metre squared of carbon (g/m<sup>2</sup>). The activated carbon is pulverised to reduce kinetic effects and allow the carbon to reach equilibrium as quickly as possible. The resultant isotherm results can then be used to calculate the theoretical amount of granular activated carbon needed to meet the treatment objectives industrially.

### 2.2. Adsorbates

All experiments were carried out using synthetic solutions prepared from Millipore water and analytical grade reagents. The gold glycinate solution was prepared by leaching a 24 ct (99.99%) gold sheet (10 cm × 2.5 cm × 0.03 cm) supplied by A&E Metals in an alkaline glycine solution. Gold dissolution was done in a glass beaker which contained 15 mL hydrogen peroxide (30% w/v, Rowe Scientific), 15 g/L glycine (> 99%, Sigma-Aldrich) dissolved in 500 mL of distilled water and buffered to a pH of 11 by adding NaOH (ThermoFisher Scientific). The alkaline solution was heated to 60 °C and stirred at 400 rpm using Teflon coated magnetic stirrer bars. The beaker was covered to prevent evaporation. Copper was added to the prepared gold glycinate solution as CuSO<sub>4</sub>·5H<sub>2</sub>O (Asia Pacific Specialty Chemicals).

### 2.3. Adsorption experiments

The traditional bottle-on-rolls method was employed for the metal glycine complexes adsorption onto activated carbon. The experimental runs were carried out in sealed 2.5 L Winchester bottles (OD 44 cm and L 30 cm) at room temperature (approx. 23 °C). The development of equilibrium isotherms was effected by contacting weighted samples of 0.040, 0.128, 0.256, 0.404 and 0.640 g/L powdered activated carbon with 250 mL of gold and copper synthetic solution. The initial pH of the solutions was adjusted to 11 and controlled during the experiments by using NaOH or H<sub>2</sub>SO<sub>4</sub>. The mixture was bottle rolled for 24 h at 105 rpm. Preliminary tests indicated that 24 h was sufficient to reach equilibrium conditions. When equilibrium was reached, samples were filtered using a 0.22 µm FilterBio® CA syringe filter to remove any present carbon fines and prevent further adsorption. Total metal concentration in solution was determined by atomic adsorption spectrophotometry by using an Agilent 55B AAS.

### 3. Results and discussion

#### 3.1. Freundlich isotherm

The experimental adsorption data on activated carbon for gold at temperature range from 22 °C to 25 °C and equilibrium pH 11 was fitted to the Freundlich model which is defined by Eq. (4) below:

$$Q_e = kC_e^n \tag{4}$$

where  $Q_e$  (mg g<sup>-1</sup>) and  $C_e$  (mg L<sup>-1</sup>) represent quantity of adsorbate per unit mass of the adsorbent and concentration of adsorbate in solution respectively at equilibrium (Juarez and Oliveira, 1993). Empirical constants  $k$  and  $n$ , derived from the intercept and slope of the linear plot of  $\log Q_e$  vs  $\log C_e$ , are reflective of adsorption equilibrium gold loading capacity and adsorption intensity or surface heterogeneity respectively (Cermakova et al., 2017). A value of  $1/n$  below unity is indicative of chemisorption while above one indicates cooperative adsorption (Haghseresht and Lu, 1998).

#### 3.2. Effect of glycine concentration

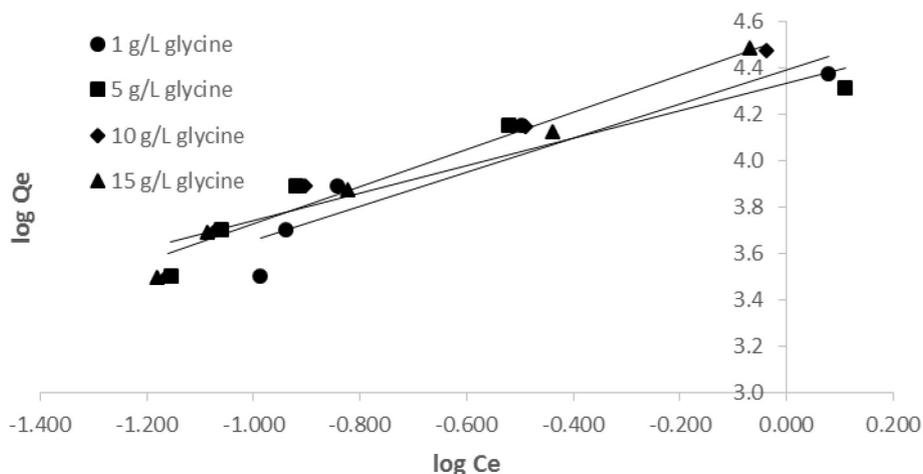
The carbon adsorption capacity of the gold glycinate complex at different free glycine concentrations but otherwise identical conditions was explored and the resultant isotherms are shown in Fig. 1. The corresponding Freundlich constants were computed and are given in Table 1. Generally, the loading capacity of gold on carbon increases with increasing glycine concentration. As reflected by the  $k$  values in Table 1, an increase in glycine concentration from 5 g/L to 10 g/L increased the adsorption capacity significantly. However, outside this concentration bracket, the glycine concentration sensitivity is lost as the gold adsorption capacity is practically the same for concentrations below and above 5 and 10 g/L respectively. This result is an indication that, unlike is the case with cyanide, the free glycine species does not have a deleterious effect as it does not appear to adsorb and compete for adsorption sites on the carbon. This is an added advantage as leaching systems are expected to contain an excess of free glycine.

**Table 1**  
Numerical Freundlich isotherm constants for different glycine concentrations for gold.

Glycine conc (g/L)	Freundlich $k$		$n$	$R^2$
	(kg/t)	g/m <sup>2</sup>		
1	24.6	$2.93 \times 10^{-5}$	0.7304	0.8705
5	21.6	$2.57 \times 10^{-5}$	0.5923	0.8737
10	34.2	$4.06 \times 10^{-5}$	0.8038	0.9697
15	33.9	$4.03 \times 10^{-5}$	0.8244	0.9838

#### 3.3. Effect of solution pH

The pH range for alkaline glycine leaching which results in practical significance is 9–12 with gold leaching maximised at pH 11 (Oraby and Eksteen, 2015). The effect of pH on the adsorption of gold glycinate onto activated carbon was therefore studied within this range at pHs 9, 10, 11 and 12. pH adjustment was done by addition of sodium hydroxide with other parameters kept constant. The loading isotherms shown in Fig. 2 confirm that the loading capacity of the gold complex in glycine systems is affected by solution pH. Overall, an increase in pH decreases the loading capacity with optimal adsorption observed at pH 10 as shown by  $k$  values listed in Table 2. A decrease in the loading capacity of gold by increasing pH can be explained by the reduction of active sites on the carbon due to the increased concentration of hydroxide ions. Hydroxide ions adsorb fairly strongly onto activated carbon (Davidson, 1974). At higher pH values more hydroxide anions and the metal glycinate complexes compete for active sites on the carbon thereby retarding gold adsorption. The solution pH is also known to affect the properties of the adsorbent (Nouri et al., 2002). From the results, it can be inferred that the optimal pH for the adsorption of gold glycinate species is pH 10. The surface groups of the activated carbon can be protonated or not (i.e. positively or negatively charged on average) depending on the equilibrium solution pH. The variation on the net surface charge consequently affects the electrostatic attraction and adsorption of metal species onto activated carbon. In the presence of copper, gold glycinate-carbon surface interaction is favoured and maximised between pH 9 to 10.



**Fig. 1.** Adsorption isotherms for gold glycinate complex on carbon as a function of glycine concentration (Freundlich model). Initial [Au] 2 ppm, Initial [Cu] 40 ppm, pH 11, 23 °C, 24 h contact time.

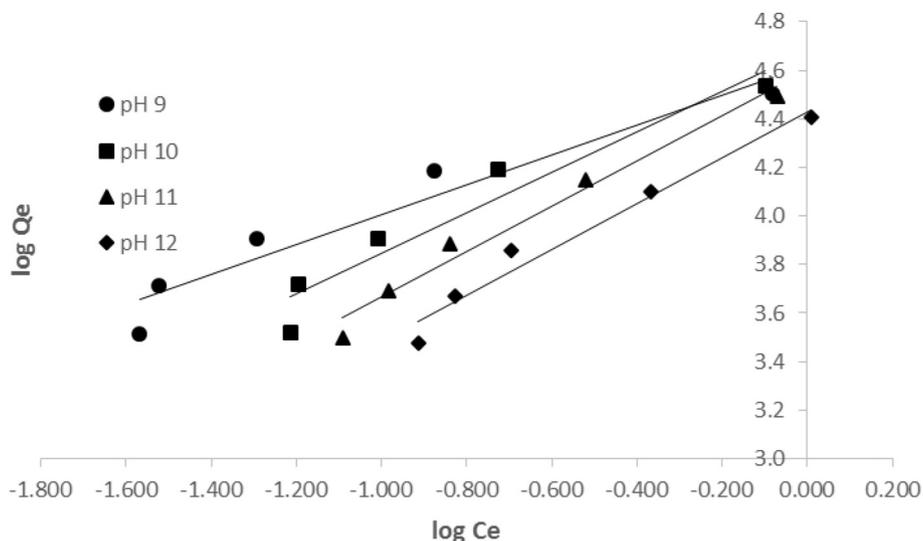


Fig. 2. Adsorption isotherms for gold glycinate complex on carbon as a function of solution pH (Freundlich model). Initial [Au] 2 ppm, Initial [Cu] 40 ppm, [Gly] 5 g/L, 23 °C, 24 h contact time.

Table 2  
Numerical Freundlich isotherm constants at various solution pHs for gold.

pH	Freundlich k		n	R <sup>2</sup>
	(kg/t)	g/m <sup>2</sup>		
9	41.8	4.97 × 10 <sup>-5</sup>	0.6139	0.9297
10	47.8	5.68 × 10 <sup>-5</sup>	0.8321	0.9289
11	39.6	4.71 × 10 <sup>-5</sup>	0.9342	0.9772
12	27.0	3.21 × 10 <sup>-5</sup>	0.9458	0.9685

Table 3  
Numerical Freundlich isotherm constants for gold adsorption on activated carbon.

Initial gold conc ppm	Freundlich k		n	R <sup>2</sup>
	(kg/t)	g/m <sup>2</sup>		
2	39.6	4.71 × 10 <sup>-5</sup>	0.9342	0.9772
4	20.6	2.45 × 10 <sup>-5</sup>	0.7190	0.9709
8	15.8	1.88 × 10 <sup>-5</sup>	0.6688	0.9475
16	14.0	1.67 × 10 <sup>-5</sup>	0.5689	0.9510

3.4. Effect of initial gold concentration

Fig. 3 displays gold adsorption isotherms obtained at various initial gold concentrations. It can be seen that as the initial gold concentration increases from 2 to 16 ppm, the isotherms show progressively lower gold loading capacity. This observation is reflected by the k values as

seen in Table 3. This trend is inconsistent with that of a pure cyanide system. For a pure cyanide system, gold loading capacity was found to increase with increasing gold concentration in solution (Marsden and House, 2006). A similar trend was however observed with the thio-sulphate ammoniacal system where an increase in the initial gold

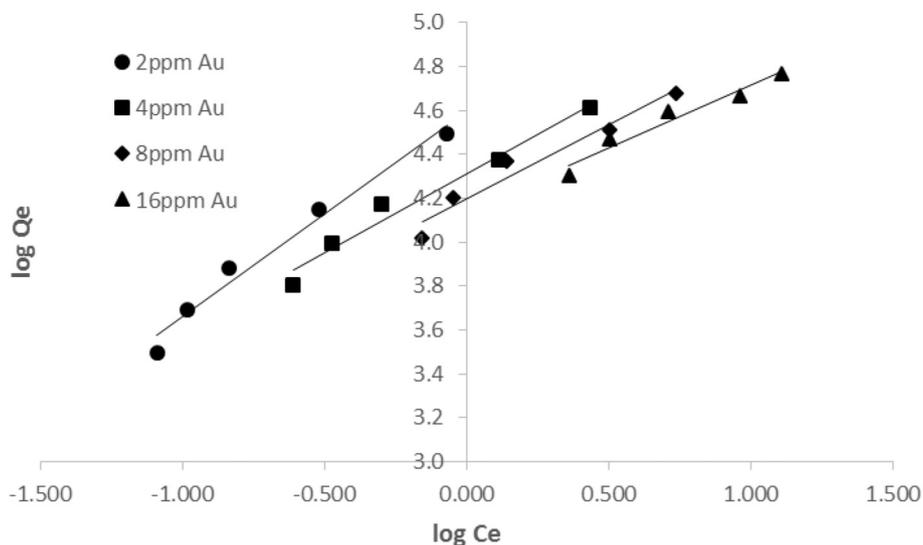


Fig. 3. Adsorption isotherms for gold glycinate complex on carbon as a function of initial gold concentration (Freundlich model). Initial [Cu] 40 ppm, [Gly] 5 g/L, pH 11, 23 °C, 24 h contact time.

concentration decreased the percentage of gold adsorbed onto carbon (Navarro et al., 2006). The authors stated that this behaviour was possibly due to insufficient active sites on carbon that can increase loading proportionately to the increasing gold concentration in solution. Another interesting observation from Fig. 3 is that as the concentration of gold increases, the isotherm is shifted to the right. This is coupled with a significant decrease in the slope. This implies that the adsorption is better suited to leaching from ores rather than concentrates, due to the lower metal concentrations after leaching.

### 3.5. Effect of initial copper concentration

Cu<sup>2+</sup> ions were found to significantly enhance gold dissolution in alkaline glycine peroxide systems (Eksteen and Oraby, 2015). Copper is normally present as one of the three major copper-glycine species, Cu(H<sub>2</sub>NCH<sub>2</sub>COO)<sub>2</sub>, Cu(H<sub>2</sub>NCH<sub>2</sub>COO)<sub>2</sub><sup>-</sup>, Cu(H<sub>2</sub>NCH<sub>2</sub>COO)<sup>+</sup> (Aksu and Doyle, 2001). The influence of copper on gold loading capacity onto activated carbon was determined at various copper concentrations and the loading isotherms are depicted in Fig. 4. There is a notable increase in the gold loading as the copper concentration increases with an intermediate loading maximum observed at a copper concentration of 20 ppm as reflected by the *k* values in Table 4. This behaviour might be explained by the difference in stability of the gold and copper glycine complexes. The copper glycine complex is significantly stronger (log K = 15.6) and more stable than the gold glycine complex (log K = 1.26) (Aksu and Doyle, 2001; Oraby and Eksteen, 2015). This means that glycine has an affinity for copper over gold. This implies if there is copper adsorbed on carbon and sufficient free glycine in the system, the copper may be eluted back into the bulk of the solution and be replaced by the gold. This contention is further supported by preliminary studies on elution which confirmed that glycine does not elute the gold from carbon but can elute a portion of the copper. Also, the strength of the bond between the gold and carbon is much stronger than that between the copper and carbon making gold detachment from carbon much more difficult.

**Table 4**  
Numerical Freundlich isotherm constants for gold glycinate complex at varying initial Cu concentrations.

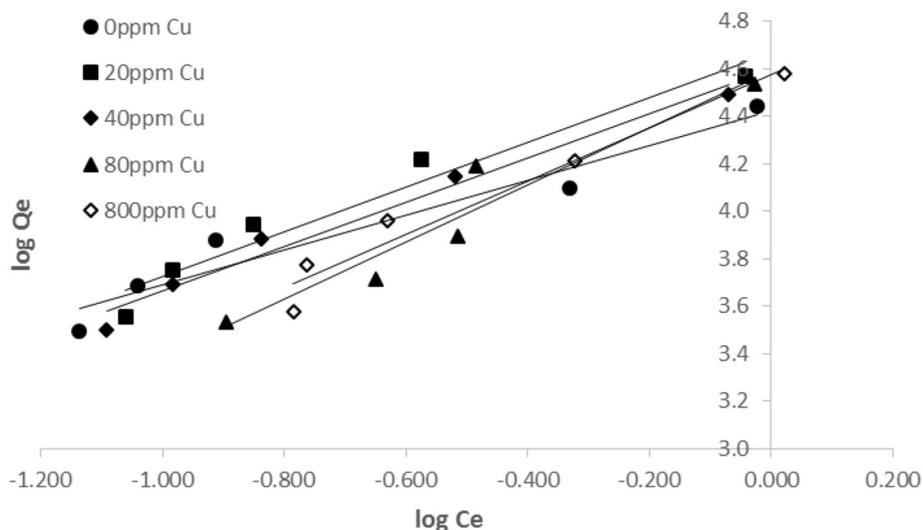
Initial Cu conc	Freundlich <i>k</i>		<i>n</i>	R <sup>2</sup>
	ppm	(kg/t) g/m <sup>2</sup>		
0	26.6	3.16 × 10 <sup>-5</sup>	0.7333	0.9361
20	46.5	5.53 × 10 <sup>-5</sup>	0.941	0.951
40	39.6	4.71 × 10 <sup>-5</sup>	0.9342	0.9772
80	39.2	4.67 × 10 <sup>-5</sup>	1.2019	0.9209
800	37.6	4.47 × 10 <sup>-5</sup>	1.1181	0.9577

### 3.6. Effect of Ca<sup>2+</sup>

Salts of calcium are known to enhance adsorptive capacity of adsorbents, including that of activated carbon (Randtke and Jepsen, 1982). To study the effect of Ca<sup>2+</sup> on the adsorptive capacity of activated carbon for gold glycine complexes, adsorption was determined at various concentrations of calcium chloride (See Table 5). As can be seen from Fig. 5, the presence of Ca<sup>2+</sup> in solution as CaCl<sub>2</sub> affects the adsorption capacity of gold onto carbon. The results indicate that the adsorptive capacity is enhanced by increasing concentrations of calcium. Presence of cations in solution are said to improve adsorption in one of the following three ways (Randtke and Jepsen, 1982):

**Table 5**  
Numerical Freundlich isotherm constants for gold adsorption at varying Ca<sup>2+</sup> concentrations.

CaCl <sub>2</sub> conc	Freundlich <i>k</i>		<i>n</i>	R <sup>2</sup>
	(g/L) Kg/t	g/m <sup>2</sup>		
0.0	39.6	4.71 × 10 <sup>-5</sup>	0.9342	0.9772
0.5	42.2	5.02 × 10 <sup>-5</sup>	0.8244	0.9813
2.0	49.8	5.92 × 10 <sup>-5</sup>	0.7927	0.9905



**Fig. 4.** Adsorption isotherms for gold glycinate complex on carbon as a function of initial copper concentration (Freundlich model). Initial [Au] 2 ppm, [Gly] 5 g/L, pH 11, 23 °C, 24 h contact time.

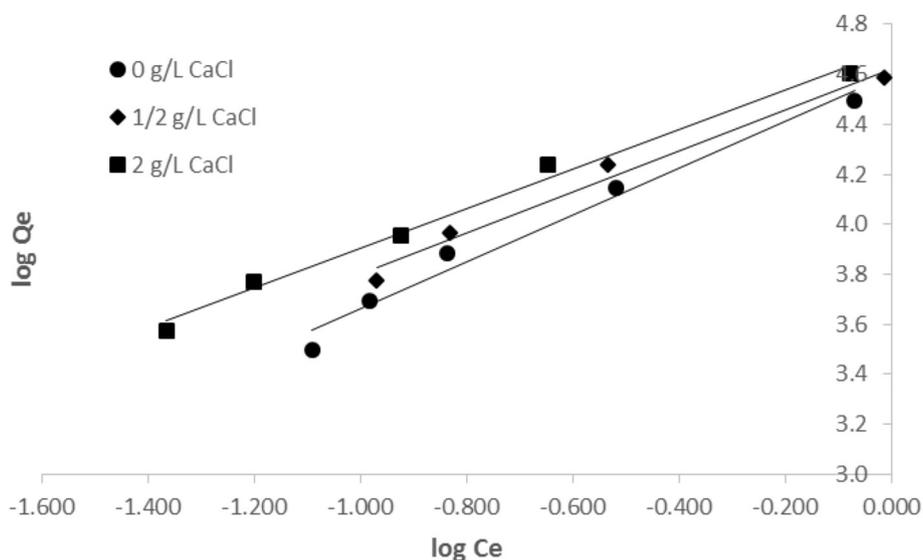


Fig. 5. Adsorption isotherms for gold glycinate complex on carbon as a function of ionic solution strength (Freundlich model). [Gly] 2 g/L, Initial [Au] 2 ppm, Initial [Cu] 40 ppm, [Gly] 5 g/L, pH 11, 23 °C, 24 h contact time.

- i) Interaction of the cation and functional groups on the adsorbent surface resulting in the neutralisation of repulsive forces between adsorbate and adsorbent and possible creation of new and or favourable active adsorption sites.
- ii) Interaction of the cation with the already adsorbed species possibly resulting in the rearrangement of the packing and alignment of the adsorbed species.
- iii) Interaction of the cation salt with the adsorbate prior to adsorption likely resulting in the alteration of the solubility of the adsorbate.

#### 4. Conclusions

The adsorption isotherms of gold complexes onto activated carbon from alkaline glycine solutions containing copper were studied and modelled using the Freundlich isotherm model. A summary of the results is given below:

- Increasing the glycine concentration up to 10 g/L increases the carbon loading significantly. Above 10 g/L free glycine concentration, the carbon loading capacity is not greatly affected by glycine concentration in solution.
- Increasing the pH from 10 through 11 to 12 decreased the carbon loading capacity due to increased hydroxide ions which compete with gold complexes for active sites on the carbon. pH value that gave the highest gold loading is 10.
- High initial gold concentration lowered the carbon's adsorption capacity. Increasing the initial gold concentration from 2 to 16 ppm reduced the adsorption capacity of the carbon by over 50%.
- Surprisingly, the gold loading capacity of the carbon increased with increasing copper concentration in solution, up to a maximum after which a small decrease was noted. Instead of copper competing for adsorption sites with gold on the carbon, it somehow enhances gold adsorption.
- A higher ionic strength increased the carbon's loading capacity for gold glycinate complexes.

#### Acknowledgements

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