

**KINETICS OF GOLD-BROMIDE
LOADING ONTO ACTIVATED CARBON**

by

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Abstract. The kinetics of gold-bromide loading onto activated carbon has been investigated in a batch stirred reactor. The amount of gold adsorbed by the carbon was correlated with time based on a second-order equation. The rate constant for gold-bromide species onto activated carbon ranged from 0.27 to $1.2 \times 10^{-1} \text{ (kg/t)}^{-1}(\text{min})^{-1}$ from a solution containing 25 ppm of Au, with 0.8 g carbon per liter of solution at 30°C . The equilibrium saturation capacity (Q_e) was $36 \pm 3.0 \text{ kg/t}$ from the same system. The rate of loading increased with decreasing particle size, decreasing pH, increasing temperature, and decreasing concentration of gold in solution. The calculated activation energy E_a was 8.8 kcal/mole , significantly higher than those reported for physical adsorption. The equilibrium saturation capacity (Q_e) increased with increasing concentration of gold in solution, but was independent of other variables such as temperature, agitation speed, particle size and pH. These rate constants and equilibrium saturation capacity for the gold-bromide species are higher than those reported for the gold-cyanide systems.

Introduction.

The kinetics for loading or adsorption of gold species onto ion exchange resins and activated carbon has been investigated extensively by researchers. The literature is filled with a comprehensive account of the kinetics of gold-cyanide species onto activated carbon and ion exchange resins (Adams et al, 1987; Avraamides et al, 1985; Cho et al, 1979; Hughes et al, 1989; McDougall et al, 1980; Peel et al, 1981; Faulkner et al, 1987; Van Deventer, 1986). However, there is little information in the literature about the kinetics of gold-bromide loading onto activated carbon. In the search to find alternate lixivants for the processing of gold, it is necessary to model the kinetics of these gold species onto carbon and compare them with the available models for the cyanide system. This paper evaluates the kinetics of gold-bromide loading onto activated carbon based on a second-order kinetic model.

The loading characteristics of gold-bromide species AuBr_4^- onto activated carbon have been discussed in a previous paper (Mensah-Biney, 1993). It was postulated that the loading of AuBr_4^- species onto carbon was not reversible due to the reduction of the AuBr_4^- species to elemental gold on the carbon surface. Since loading is not reversible, and equilibrium was not attainable (at least not within a reasonable period), the conventional kinetic models are not applicable for modeling the kinetics of gold-bromide loading onto activated carbon.

The kinetics of loading of gold-chloride AuCl_4^- species onto carbon was modeled by a first-order reaction equation by Hughes (1989). Nicol and Fleming (1984) modeled the kinetics of gold-cyanide species onto activated carbon by a first-order reaction equation. Faulkner (1987) from Calgon Carbon Corporation in PA, has used first and second-order equations to describe the kinetics of gold adsorption onto activated carbon in cyanide systems.

In the present study, the kinetic data have been fitted to a second-order reaction model correlating capacity (Q) and time (t). This model is similar to the model used by Faulkner et al (1987) for fitting kinetic data for gold cyanide systems. The reasons for using this model are twofold; it will allow for the comparison between the kinetics of the gold-bromide species and those of gold-cyanide species; and this model appears to offer a more practical approach for the determination of kinetic rate constants and apparent equilibrium capacity (it has been used routinely by Calgon Carbon Corporation for determination of rate for gold-cyanide systems).

The rate constant for gold-bromide species onto activated carbon ranged from 0.27 to $1.2 \times 10^{-1} \text{ (kg/t)}^{-1}(\text{min})^{-1}$ when using a solution containing 25 ppm of Au with 0.8 g carbon per liter of solution at 30°C . The equilibrium saturation capacity (Q_e) was $36 \pm 3.0 \text{ kg/t}$ from the same system.

The rate of loading increased with decreasing particle size, decreasing pH, decreasing agitation speed, decreasing gold concentration and increasing temperature. The equilibrium saturation capacity (Q_e) increased with increasing concentration of gold in solution, but was independent of the other variables such as temperature, agitation speed, particle size and pH. The rate constant and equilibrium saturation capacity for the gold-bromide species are higher than those reported for the cyanide system (Faulkner, 1987).

Theory

In comparing kinetic rates, it is usually convenient to use linear relationships rather than the measured exponential or polynomial relationships. With gold adsorption, it is appropriate to compare straight line relationships between loading capacity and time rather than the exponential curves that result from the measurement of loading capacity and time. Therefore, the measured curves are usually transformed to linear relationships by use of kinetic models. In order to convert the measured (experimental) curves to linear

relationships, it is customary to apply rate equations used in chemical kinetics to study the velocity of chemical reactions. In the present study, a second-order rate equation was used to model the relationship between loading capacity (Q) in kg/t and time (t) in minutes. The loading of gold bromide species onto activated carbon is postulated to involve at least two different mechanisms: mass transfer, and chemical reduction. Therefore, it seems appropriate to model the kinetic data with a second order rate equation.

If Q_e is the equilibrium saturation capacity and Q_t is the capacity at any time t, then the rate for the accumulation of gold onto the carbon can be derived in terms of the second-order kinetic equation as:

$$\frac{dQ}{dt} = k(Q_e - Q_t)^2 \quad (1)$$

$$\frac{dQ}{(Q_e - Q_t)^2} = k dt \quad (2)$$

$$\int \frac{dQ}{(Q_e - Q_t)^2} = k \int dt \quad (3)$$

$$\frac{1}{(Q_e - Q_t)} = kt + c \quad (4)$$

$$\text{at } t = 0, Q_t = 0$$

$$\frac{1}{Q_e} = c \quad (5)$$

$$\frac{1}{(Q_e - Q_t)} = kt + \frac{1}{Q_e} \quad (6)$$

$$Q_t = (Q_e - Q_t)(ktQ_e + 1) \quad (7)$$

$$Q_t = Q_e^2 kt - ktQ_e Q_t - Q_t = Q_e \quad (8)$$

$$ktQ_e Q_t = Q_e - Q_e^2 kt \quad (9)$$

$$Q_t(ktQ_e + 1) = Q_e^2 kt \quad (10)$$

$$\frac{ktQ_e + 1}{Q_e^2 k} = \frac{t}{Q_t} \quad (11)$$

$$\frac{t}{Q_e} + \frac{1}{Q_e^2 k} = \frac{t}{Q_t} \quad (12)$$

$$\frac{t}{Q_t} - \frac{t}{Q_e} = \frac{1}{Q_e^2 k} \quad (12a)$$

A plot of t/Q_t versus t is linear with a slope = $1/Q_e$ and intercept = $1/(Q_e^2 k)$. The rate constant k in $(\text{kg/t})^{-1} \text{min}^{-1}$ can thus be calculated from the slope(s) and intercept(1) as:

$$t = \frac{1}{Q_e^2 k} \quad (13)$$

$$s^2 = \left(\frac{1}{Q_e}\right)^2 \quad (14)$$

$$t = \frac{s^2}{k} \quad (15)$$

$$k = \frac{s^2}{t} \quad (16)$$

Experimental

Analytical Methods.

Gold analysis was performed by flame atomic absorption spectroscopy (AAS) on solutions only, and no analysis of solid material was performed.

Preparation of Activated Carbon.

The present research was carried out using coconut-shell activated carbon (Calgon PCB). The preparation and treatment of the activated carbon have been described elsewhere (Mensah-Biney, 1991)

Gold Solution.

A gold-bromide stock solution containing about 1000 ppm of gold was prepared according to procedure described in previous publications (Mensah-Biney, 1993).

Procedure:

The experimental apparatus used for this kinetic study is shown schematically in Figure 1, and the experimental conditions are tabulated in Table 1. The procedure involved stirring a carefully weighed sample of conditioned carbon in 500 ml. of a gold-bromide solution at a constant temperature. The initial pH and redox potential were measured before adding the carbon, and the mixture was agitated at constant temperature for specified contact time. All the tests used a nominal 25 mg/L Au standard gold solution prepared by diluting 1000 mg/L Au gold-bromide stock solution with deionized water, except in tests where the concentration of gold was the dependent variable. The pH and redox potential of this standard solution were 4.35 and 850 mV (SCE)

respectively. This solution will be referred to as standard solution in the text. Approximately 0.8 g carbon per liter of solution of minus 20 mesh size activated carbon was used for each test unless particle size or carbon concentration was a dependent variable.

At specified intervals, the agitation was stopped and a sample of the solution was taken using an Oxford micropipet with disposal tips. The solution sample was filtered through a 0.35 micron filter paper before submitting for gold analysis. The concentration of gold in solution as a function of time was thus measured. There was no analysis of gold on the carbon and the amount of gold adsorbed by the carbon was determined from solution analysis only.

Discussion of Results

The loading of gold-bromide species onto activated carbon was studied in a batch stirred reactor with a motorized stirrer and three-blade impeller. The amount of gold adsorbed by the carbon was correlated with time based on a second-order equation represented by equation (12a). Figure 2 shows a typical plot of t/Q versus t demonstrating second order kinetics fit of the data with the model. The derived model constants are tabulated in Table 2 and the effect of the following variables on the kinetic rate is discussed below:

Agitation Speed

Figure 3 shows the plot of t/Q versus t for the varying agitation speeds used for the tests. These tests were performed at 30° C constant temperature using the standard solution with agitation speeds of 480, 625 and 850 RPM. The data listed in Table 2 show that there was only a small change in the rate constant with varying agitation speed measured in RPM. The rate constant decreased from 8.87×10^{-1} to 6.57×10^{-1} (kg/t)⁻¹min⁻¹ when the agitation speed was increased from 480 to 850 RPM. The ratio of the rate constants was 1.4.

However, the decrease in the rate constant, as measured by R-value, was substantial, decreasing from 1.227 to 0.647 when the agitation speed was increased from 480 to 850 RPM. The ratio of the rate constants based on R-values was 1.9.

This confirms that the theoretical rate constant based on this model is not directly proportional to the R-value, and their absolute values may not be equal. Therefore, the use of R-value for comparing the rate of adsorption for activated carbons based on this model should be interpreted accordingly.

Temperature

The correlation between t/Q and t for three varying temperatures investigated (30, 45, 65° C) is shown in Figure 4 and the derived constants are listed in Table 2. These tests used minus 20 mesh size carbon in a standard solution with agitation speed of 625 RPM. The kinetic rate constant is clearly dependent on temperature. The rate constant increased from 7.4×10^{-1} to 34.2×10^{-1} (kg/t)⁻¹min⁻¹ for a temperature increase of 30° C to 65° C. This increase in the kinetic rate with increasing temperature is inconsistent with the adsorption behavior of activated carbon for gold-cyanide species. The Arrhenius plot correlating the rate constant with temperature is shown in Figure 5. The activation energy (E_a) determined from the plot was 8.8 kcal/mole which is significantly higher than those reported for physical adsorption. This high activation energy value may be an indication of a chemical reaction controlled mechanism.

The recovery of gold from solution after 2-hour contact time also increased from 87% to 99.7 % when the temperature was increased from 30° C to 65° C. This is a significant finding because it shows that practical adsorption rates for the recovery of gold-bromide species are feasible at moderate temperatures of 30 to 65° C.

Carbon Particle Size

The effect of particle size on the kinetics of loading is shown in Figure 6 and the derived model constants are also listed in Table 2. The tests used standard solutions at a constant temperature of 30° C and agitation speed of 625 RPM. There was a significant increase in the loading rate for gold as the particle size of the carbon decreased. This is in agreement with conventional mass transfer or chemical reaction mechanisms and is due to the increase in the surface area of the particles as the size is reduced. The rate constant increased from 2.7×10^{-1} to 12.4×10^{-1} (kg/t)⁻¹min⁻¹ when the carbon particle size was decreased from + 10 mesh to - 20 mesh. The corresponding gold recovery from solution in two hours was 85% and 95% respectively.

Initial Gold Concentration

The dependence of the kinetic rate on initial gold concentration on was evaluated using minus 20 mesh carbon at a constant temperature of 30° C and agitation speed of 625 RPM. Figure 7 shows the plot of t/Q versus t for the three initial concentrations investigated (12.5, 35.0, and 50.0 mg/L Au). The derived rate constants are listed in Table 2. The data show that the rate constant increased with decreasing initial gold

concentration. The rate constant was 3.8×10^{-1} (kg/t)⁻¹min⁻¹ and 29.6×10^{-1} (kg/t)⁻¹min⁻¹ for initial gold concentration of 50.0 mg/L and 12.5 mg/L, respectively. This dependence of the kinetic rate on initial gold concentration is due to the relative availability of adsorption sites. For the same weight of mono sized carbon, the available site per gold species in solution is higher when the initial concentration is lower. The driving force for the transfer from bulk solution to the carbon surface is stronger resulting in a faster rate of adsorption.

pH

The effect of pH on the kinetics of loading was also evaluated during the present study. The tests evaluated three pH values (1.58, 4.35, 9.50) in a standard solution at constant temperature of 30° C and agitation speed of 625 RPM. The effect of pH on the rate of loading is shown in Figure 8 and the derived model constants are listed in Table 2. The data show that the rate of loading was faster at low pH values. The rate constant was 12.2×10^{-1} (kg/t)⁻¹min⁻¹ at 1.58 pH and decreased to a low value of 2.3×10^{-1} (kg/t)⁻¹min⁻¹ at 9.50 pH. This agrees with the typical mechanism of adsorption of gold species by activated carbon. It is well established that adsorption rates for gold species by activated carbon are faster in acidic medium. This phenomenon has been explained in terms of the functionality groups proposed to be present at the carbon surface and are responsible for the adsorption of the gold species. The results also show that 95% of the gold in solution was recovered by the carbon in two hours at 1.58 pH compared to 87% within the same period at 9.5 pH.

Excess Bromine (as Geobrom 55)

The dependence of the rate of loading on excess bromine addition on was evaluated using a standard solution at a constant temperature of 30° C and agitation speed of 850 RPM. Excess bromine was added to the solution as 0.00, 0.25, and 0.50 g/L Geobrom 55. Figure 9 shows graphically the dependence of excess bromine addition on the rate of loading, and the derived model constants are listed in Table 2. The rate constant decreased from 9.97×10^{-1} to 6.57×10^{-1} (kg/t)⁻¹min⁻¹ when the excess bromine addition was increased from 0.00 to 0.25 g/L Geobrom 55. This finding confirms our previous finding that too much bromine in solution is detrimental for gold loading onto activated carbon. This is explained in terms of oxidation of the carbon surface by the excess bromine resulting in poor adsorption of gold species. Too much of the bromine will in fact oxidize and degrade the carbon surface to such an

extent that adsorption of gold species will cease. Such a phenomenon was experienced during the loading capacity study reported previously (Mensah-Biney, 1993). Gold recovery from solution onto the carbon was only 54% in two hours when the excess bromine addition was 0.50 g/L Geobrom 55 compared to 87% when there was no excess bromine addition.

Summary

The kinetic data generated from this study fitted the derived model fairly well. The model constants derived during these tests are valid for the initial period investigated. They may change with time especially during the extended loading times, such as those usually used for loading capacity measurements. This is true because the loading of gold-bromide species onto activated carbon involves at least two mechanisms. The initial period of loading is postulated to be dominated by a mass transfer type mechanism such as ion exchange. This type of mechanism can thus be described by conventional equilibria models. The model constants for this type of mechanism will remain constant and not vary with time under normal conditions. The other type of mechanism which is postulated to predominate during the long and extended period of loading is chemical, irreversible and cannot be described by conventional models. The model constants for that will not remain constant and vary with time. In essence the model constant for the loading of gold-bromide species onto carbon is made up of the sum of at least two different constants. The present study was not designed to distinguish between these two constants.

The rate constant(k) obtained during this initial period is the maximum rate constant under the specified conditions. This rate constant would decrease with time under same conditions and will approach zero. The increase in rate constant will be in steps as the loading time increases. At an extended long period of loading, the decrease in the rate constant will become negligible as shown by the results of the loading capacity measurements described elsewhere (Mensah-Biney, 1993).

The reported equilibrium saturation capacities are the initial values and are expected to represent the minimum values. As the loading time increases, these values will increase. However, these increases will also be in steps, such that a constant value will be obtained for one period and then increase to another higher constant value for an increased period. The loading capacity measurements reported elsewhere(Mensah-Biney,

1991) show that an equilibrium saturation capacity of more than 800 kg/t was achieved for a loading period lasting more than 100 hours.

It is important to note that the R-value is not directly proportional to the derived rate constant (k). However under proper conditions, the R-value is proportional to the theoretical rate constant (k), and the R-value may be used to compare the rate of loading for different carbons. The R-value has been used by Calgon Corporation for estimating the rate of loading for activated carbon.

CONCLUSIONS

The second-order rate constant for gold-bromide species onto activated carbon ranged from 0.27 to $1.2 \times 10^{-3} \text{ (kg/l)}^{-1} \text{ (min)}^{-1}$ from a solution containing 25 ppm of Au, with 0.8 g carbon per liter of solution at 30° C. The saturation equilibrium loading capacity Q_e was $36 \pm 3.0 \text{ kg/t}$ from the same system. The rate of loading increased with decreasing particle size, decreasing pH, decreasing agitation, increasing temperature, and decreasing concentration of gold in solution. The calculated activation energy (E_a) was 8.8 kcal/mole. The equilibrium saturation capacity (Q_e) increased with increasing concentration of gold in solution, but was independent of the other variables such as temperature, agitation speed, particle size and pH. This agrees with the typical adsorption behavior of gold cyanide species by activated carbon.

The results of these kinetic tests show that the data fitted the proposed model fairly well with correlation coefficient of fit about 0.99. Thus, the kinetic of gold-bromide loading onto activated carbon can be described by a second-order reaction model that correlates capacity (Q) with time (t). This model appears to offer a more practical approach for the determination of the kinetic rate for the loading of gold bromide species onto activated carbon, since the model uses data that cover relatively longer contact time between the adsorbent and solution. The data presented in Figure 2 cover a loading period of 2.5 hours. Moreover, the model relates capacity rather than concentration and time.

The determined rate constants were moderately high, and are significantly higher than those reported for the adsorption of gold cyanide species by activated carbon. The rate of recovery of gold from solution, particularly the 99.5 % recovery in two hours at 65° C, seems practical and may render the recovery of gold from bromine solution by activated carbon as a feasible process.

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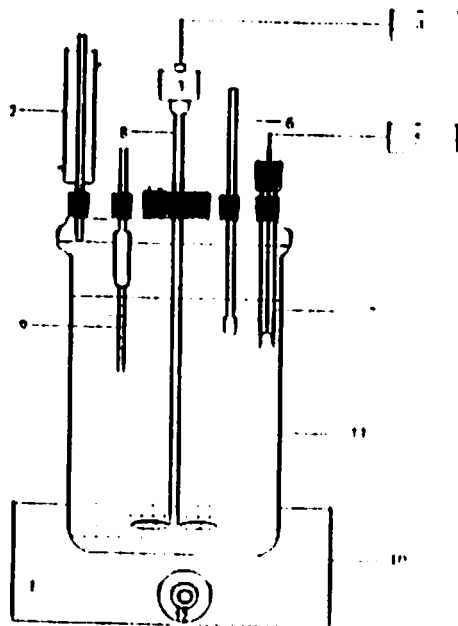
TABLE 1. Experimental Conditions for the Kinetics of Gold-bromide Loading onto Activated Carbon.

Volume of solution (mL.)	500
Particle Size, (mesh)	+ 10, 10 x 14, - 20
Initial Solution Conc.(mg/L Au)	12.5, 25, 50
Initial pH	1.58, 4.35, 9.50
Initial Redox potential(mv. vs SCE)	730 to 840
Excess Geobrom 55 Conc.(g/L)	0.05, 0.25, 0.50
Temperature (° C)	30, 45, 65
Agitation speed (RPM)	480, 625, 800, 850

TABLE 2. Kinetic Data and Derived Constants for the loading of Gold-bromide Species onto Activated Carbon

Variable	Value	Q_c	R-Value	$k \times 10^4$	% $[Au]_0$ Loaded	Corr. Coeff.
Agitation Speed(RPM)	480	37.2	1.227	8.87	91.2	0.9984
	625	36.3	0.975	7.40	87.0	0.9917
	850	31.4	0.647	6.57	68.7	0.9936
Temperature (° C)	30	36.4	0.974	7.40	87.0	0.9917
	45	35.4	2.284	18.00	97.5	0.9998
	65	34.2	4.009	34.20	99.6	0.9996
Particle Size(mesh)	+ 10	41.5	0.465	2.70	75.3	0.9819
	10 x 14	37.5	0.612	4.35	82.0	0.9974
	- 20	35.9	1.592	12.40	95.3	0.9996
Initial Conc. $[Au]_0$ (mg/L)	12.5	17.4	0.900	29.60	98.4	0.9995
	35.0	30.6	0.933	9.97	87.0	0.9818
	50.0	57.9	1.267	3.77	68.5	0.9969
pH	1.58	35.7	1.556	12.50	95.3	0.9996
	4.35	36.3	0.975	7.40	87.0	0.9917
	9.50	36.8	0.406	3.00	62.3	0.9635
Excess Bromine (g/L DDMI)	0.00	30.6	0.933	9.97	87.0	0.9818
	0.25	31.4	0.647	6.57	68.7	0.9936
	0.50	26.2	0.478	6.96	54.3	0.9631

- (Q_c): is the equilibrium saturation capacity in kg Au/t carbon.
(k): is the second-order rate constant in $(\text{kg/t})^{-1} \text{min}^{-1}$.
(R-value): is the reciprocal of intercept (1/I) in $(\text{kg Au/t carbon})\text{min}^{-1}$.
(DDMI): is 5-dimethylhydantoin, the bromine carrier (Geobrom 55).
(Corr. Coeff.): is the correlation coefficient of fit based on linear regression analysis, a value of 1.000 represents a perfect fit.
% $[Au]_0$ Loaded: is the fraction (as %) of initial gold in solution loaded onto the carbon in 120 min.



- | | |
|--------------------------------------|----------------------------|
| 1. Heating Mantle | 7. pIV Eh Probe |
| 2. Condenser | 8. 3 blade PTFE Stirrer |
| 3. Stirring Motor | 9. Sampling Tip |
| 4. Self Constant Voltage Transformer | 10. Voltage Transformer |
| 5. pIV Eh Meter | 11. Reactor (Resin Kettle) |
| 6. Thermometer | 12. Temperature Controller |

Fig.1 Schematic Diagram of the Apparatus for the Kinetic of Loading

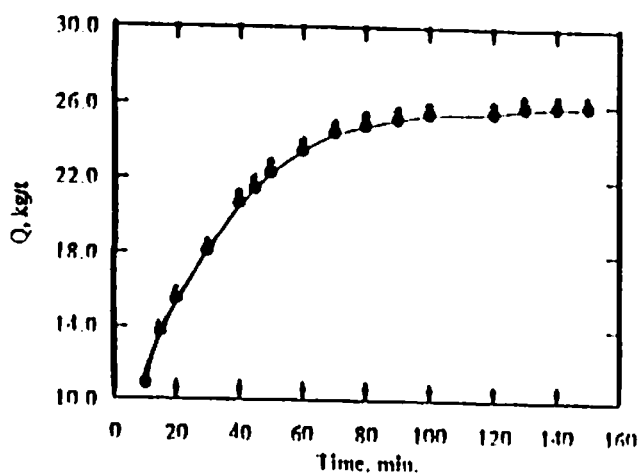


Figure 2. Typical Kinetic Plot for the Loading of Gold bromide onto Activated Carbon

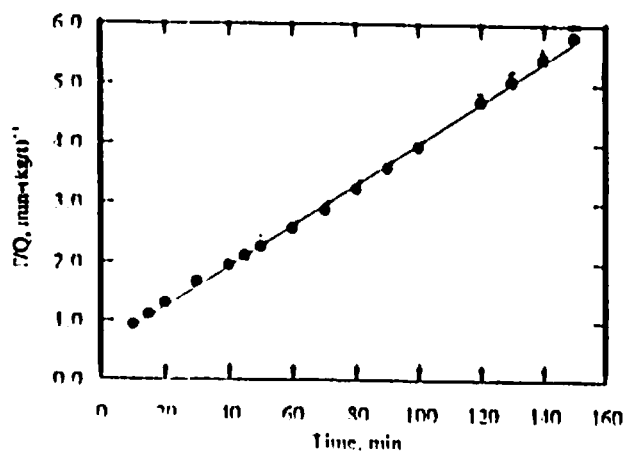


Figure 3. Typical Plot of $1/Q$ vs. t for fitting the kinetic data from Fig 2 above using a second-order model

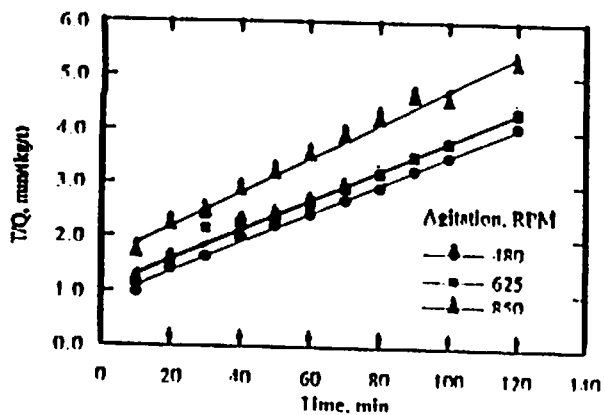


Figure 3. Effect of Agitation Speed on the Kinetics of Loading. 25 mg/L Au, 30 °C, 20 mesh

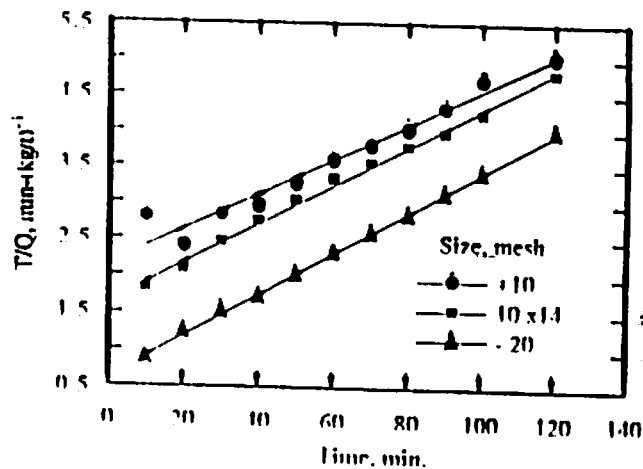


Figure 6. The Effect of Carbon Size on the Kinetics of Loading. 25 mg/L Au, 30 °C, 625 rpm.

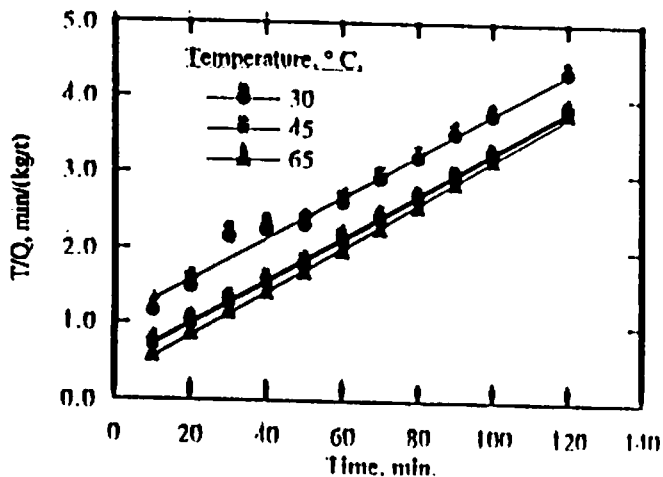


Figure 4. Dependence of Temperature on the Kinetics of Loading. 25 mg/L Au, 625 rpm speed, -20 mesh

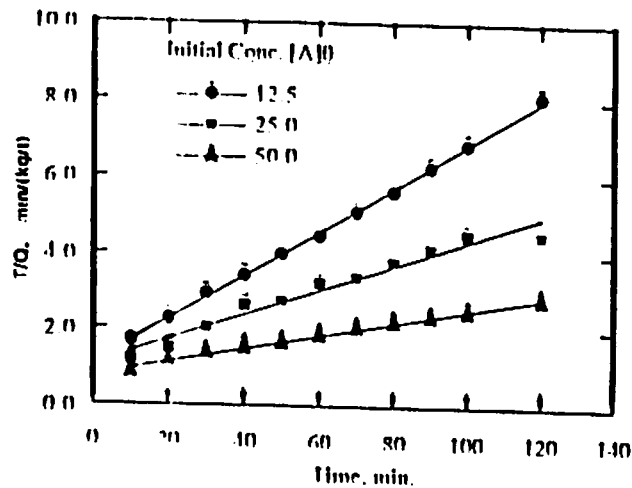


Figure 7. The effect of Initial Concentration of Gold on the Kinetics for Loading: 30 °C, 625 rpm, -20 mesh size.

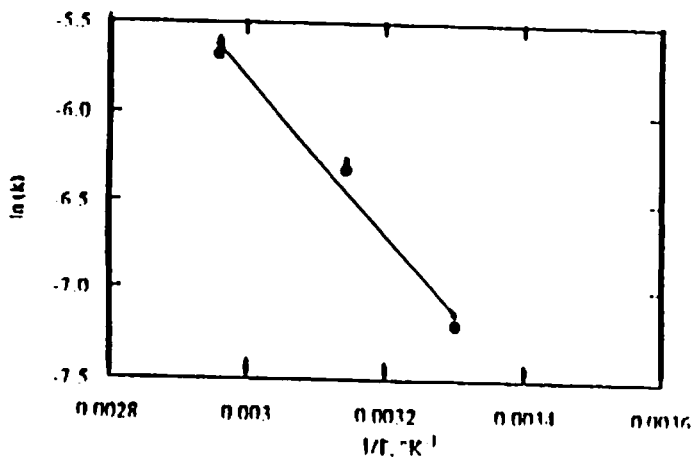


Figure 5. Effect of Temperature on Rate Constant. Arrhenius Plot; 625 RPM, 25 mg/L Au, 20 mesh

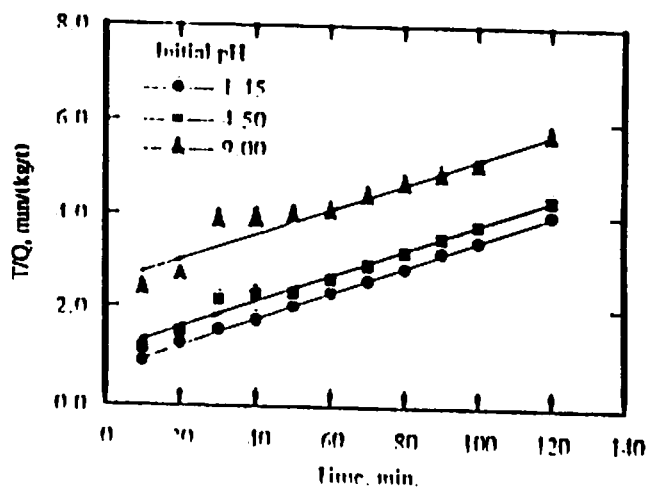


Figure 8. The influence of pH on the Kinetics of Loading: 25 mg/L Au, 30 °C, 20 mesh size, 625 rpm

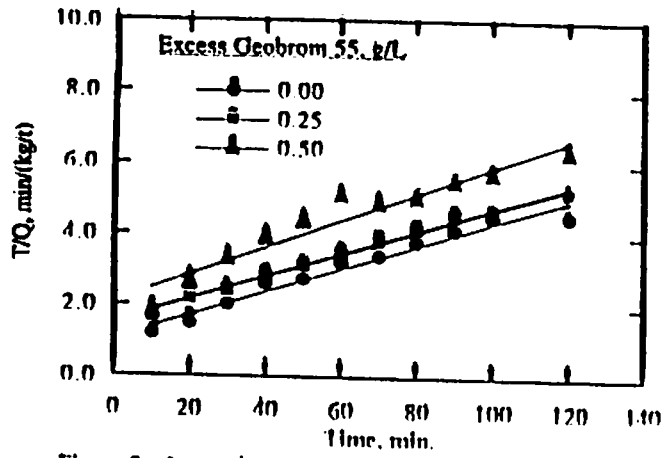


Figure 9. Dependence of Excess Bromine Concentration on Kinetics of Loading