

Model for the Calculation of Heat Absorbed by Oxalic Acid Solution during Leach-Enhanced Dissolution of Iron from Iron Oxide Ore

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Abstract: The model for the calculation of the quantity of heat absorbed by oxalic acid solution as a result of the dissolution of iron during leaching of iron oxide ore has been derived. The triple exponential model; $Q = \text{Exp}[\exp(1.12e + 0.8342)]$ was found to predict the quantity of heat absorbed by the oxalic acid solution as a result of dissolution of iron depending on the mass-input of iron oxide ore during the leaching process (providing that $e = 1$). The validity of the model is believed to be rooted on the expression $e = N \ln[\text{Log}Q]$ where both sides of the expression are correspondingly approximately equal to 1. The maximum deviation of the model-predicted quantity of absorbed heat from the corresponding experimental values is less than 4% which is quite within the acceptable range of deviation limit of experimental results. The positive values of heat absorbed as obtained from experiment and model indicate and agree that the leaching process is endothermic in nature. The quantities of absorbed heat per unit mass-input of iron oxide ore as obtained from experiment and derived model were evaluated as 7.315 J/g and 6.84 J/g respectively. Similarly, the quantities of absorbed heat per unit percent rise in the concentration of dissolved iron as obtained from experiment and derived model were evaluated as 10837.04 J/% and 10133.33 J/% respectively, indicating proximate agreement. [Journal of American Science. 2010;6(9):431-437]. (ISSN: 1545-1003).

Keywords: Model, Heat Absorbed Iron Dissolution, Oxalic Acid, Iron Oxide Ore, Leaching.

1. Introduction

The possibility of using different organic and inorganic acids has been evaluated in several studies. The dissolution of iron oxides and oxyhydroxides in hydrochloric and perchloric acids was evaluated by Sidhu et al. (1981) while Lim-Nunez and Gilkes (1987) used synthetic metal-containing goethite and haematite in their evaluation. Studies were carried out (Borghi et al. (1989)) to investigate the effect of EDTA and Fe(II) during the dissolution of magnetite. Even though industrial use of sulphuric acid and other inorganic acids to dissolve iron oxide has not fared too well, Chiarizia and Hotwitz (1991) studied the dissolution of goethite in several inorganic acids in the presence of reducing agents. These acids belong to the families of the carboxylic and diphosphoric. The practical effectiveness of several organic acids (such as acetic, formic, citric, ascorbic acids etc.) used for dissolving iron from iron compounds has also been evaluated. (Ambikadevi and Lalithambika (2000)). Oxalic acid was found to be the most promising because of its acid strength, good complexing characteristics and high reducing power, compared to other organic acids.

Taxiarchou et al, (1997a) discovered that on using oxalic acid, the dissolved iron can be precipitated from the leach solution as ferrous oxalate, which can be re-processed to form pure haematite by calcinations. Several studies have been carried out (Vaglio et al.,1998; Segal et al., 1984; Frenier and Growcock, 1984; Jepson, 1988; Panias et al., 1996; Blesa et al., 1994; Cornell and Schindler,1987) to investigate the use of oxalic acid to dissolve iron oxide on a laboratory scale. Lee et al. (2006) used 0.19-0.48M oxalic acid to dissolve hydrated iron oxide. The dissolved iron was found to reach 90% for a 20% slurry within 60mins. using 0.19M oxalic acid for the finer fraction ($< 150\mu\text{m}$) containing 0.56% Fe_2O_3 . Lower iron removal was achieved using the coarser fraction ($>150\mu\text{m}$) containing 1.06% Fe_2O_3 and then reached a steady state of only 78% after 1 h of leaching. It was expected that the liquor pH is < 1 at the oxalic acid concentration range studied (0.19-0.48), even though the pH was not measured or controlled. Taxiarchou et al. (1997a) found that the maximum iron dissolution of only 40% is within 3 h at temperatures in the range 90-100°C. At 0.5M oxalate and all temperatures (25, 60 and 80°C), the

dissolution of iron was faster at a lower pH in the range pH 1-5 studied.

Nwoye and Mbuka (2010) derived a model for computational analysis of heat absorbed by hydrogen peroxide solution (relative to the weight of iron oxide ore added). The model;

$$Q = e^{1.04(W)} \quad (1)$$

was found to be dependent on the weight-input of iron oxide ore. The values of the heat absorbed Q as predicted by the model were found to agree with those obtained from the experiment that the leaching process is endothermic in nature, hence the positive values of the absorbed heat Q_{exp} and Q_M . The validity of the model was found to be rooted on the expression $\ln Q = N(W)^{1/2}$ where both sides of the expression are corresponding almost approximately equal. Maximum deviation of model-predicted quantities of heat absorbed (from the corresponding experimental values) was found to be less than 33% which is quite within the acceptable deviation range of experimental results.

Nwoye et al., (2010) derived a model for evaluating the quantity of heat absorbed by hydrogen peroxide solution relative to solution temperature (during leaching of iron oxide ore). The values of the absorbed heat as predicted by the model:

$$Q = e^{(0.195T)} \quad (2)$$

was found to agree with those obtained from the experiment in showing that the leaching process is endothermic in nature, culminating in the positive values of the absorbed heat. It was found that the validity of the model stems on the expression $\ln Q = NT$ where both sides of the equation are approximately equal to 5. The maximum deviation of the model-predicted quantities of absorbed heat from the corresponding experimental values is less than 25% which is quite within the acceptable range of deviation limit of experimental results.

Model for predictive analysis of the quantity of heat absorbed by oxalic acid solution during leaching of iron oxide ore has been derived (Nwoye et al., 2009). The model is expressed as;

$$Q = e^{(0.987(T))} \quad (3)$$

Where

T = Solution temperature during leaching of iron oxide ore in oxalic acid solution. ($^{\circ}C$)

N = 0.987(Temperature coefficient for oxalic acid solution during leaching of iron oxide ore) determined in the experiment (Nwoye,2005).

Q = Quantity of heat energy absorbed by the oxalic acid solution during the leaching process.(J)

The model was found to depend on the value of the final solution temperature measured during the experiment. It was observed that the validity of the model is rooted in the expression $(\ln Q)/N = T$ where both sides of the relationship are correspondingly almost equal. The respective deviation of the model-predicted Q values from the corresponding experimental values was found to be less than 21% which is quite within the acceptable range of deviation limit of experimental results. The positive values of heat absorbed as obtained from experiment and model indicate and agree that the leaching process is endothermic in nature.

Nwoye (2008b) has reported that the heat absorbed by oxalic acid solution during leaching of iron oxide ore can be predicted using the model he derived which works under the process condition; initial pH 6.9, average ore grain size; 150 μ m and leaching temperature; 30 $^{\circ}C$. The model (Nwoye, 2008b) can be stated as

$$Q = K_N \left(\frac{\gamma}{\%Fe_2O_3} \right) \quad (4)$$

Where

Q = Quantity of heat absorbed by oxalic acid solution during the leaching process. (J)

γ = Final pH of the leaching solution (at time t).

$\%Fe_2O_3$ = Concentration of haematite dissolved in oxalic acid solution during the leaching process.

K_N = 4.57(Haematite dissolution constant in oxalic acid solution) determined in the experiment (Nwoye, 2008c).

Nwoye (2008b) carried out further work on the model using the same process conditions and observed that on re-arranging the model as;

$$\%Fe_2O_3 = K_N \left(\frac{\gamma}{Q} \right) \quad (5)$$

the concentrations of haematite predicted deviated very insignificantly from the corresponding experimental values. In this case, the value of Q was calculated by considering the specific heat capacity of oxalic acid. Values of heat absorbed by the oxalic acid solution during the leaching of iron oxide ore as predicted by the model (Nwoye, 2008b) agree with the experimental values that the leaching process is endothermic. This is because all the predicted values of the heat absorbed by the oxalic acid solution were positive. The model shows that the quantity of heat absorbed by oxalic acid solution during the leaching process is directly proportional to the final pH of the solution and inversely proportional to the concentration of haematite dissolved. Model for

calculating the quantity of heat absorbed by oxalic acid solution during leaching of iron oxide ore has been derived (Nwoye et al.,2009). It was observed that the validity of the model is rooted on the expression $\ln Q = N$ where both sides of the expression are approximately equal to 7. The mode;

$$Q = e^{(1.02)} \quad (6)$$

was found to depend on the value of the final solution pH measured during the experiment. The maximum deviation of the model-predicted Q values from the corresponding experimental values was found to be less than 11% which is quite within the acceptable range of deviation limit of experimental results. The positive values of heat absorbed as obtained from experiment and model agree and show that the leaching process is endothermic in nature.

The mixed potential model of leaching assumes that the charge transfer processes occurring at the mineral surfaces are those that control the rate of dissolution (Kanevskii et al, 1963).

The aim of this work is to derive a model for the calculation of heat absorbed by oxalic acid solution during leach-enhanced dissolution of iron from iron oxide ore (mined in Itakpe (Nigeria)).

2. Model

The solid phase (ore) is assumed to be stationary, contains the un-leached iron remaining in the ore. Hydrogen ions from the oxalic acid attack the ore within the liquid phase in the presence of oxygen.

2.1 Model Formulation

Results of previous research work (Nwoye, 2000) carried out were used for this work. Statistical and computational analysis of these results (Nwoye, 2000) presented in Table 1, gave rise to Table 2 which indicate that;

$$e = N \ln[\text{Log}Q] \quad (\text{approximately}) \quad (7)$$

Introducing the value of N into equation (7)

$$e = 0.8929 \ln[\text{Log}Q] \quad (8)$$

Mathematically, since $2.303 \text{Log}Q = \ln Q$

$$e = 0.8929 [\ln 0.4342 + \ln(\ln Q)] \quad (9)$$

$$e = -0.7449 + 0.8929[\ln(\ln Q)] \quad (10)$$

Equation (10) predicts the concentration of dissolved iron associated with absorbed heat by the oxalic acid solution (providing that e = 1 and the value of Q known).

Re-arranging equation (10)

$$0.8929[\ln(\ln Q)] = e + 0.7449 \quad (11)$$

$$[\ln(\ln Q)] = \left[\frac{e + 0.7449}{0.8929} \right] \quad (12)$$

$$[\ln(\ln Q)] = \left[\frac{e + 0.8342}{0.8929} \right] \quad (13)$$

$$[\ln(\ln Q)] = 1.12e + 0.8342 \quad (14)$$

$$\ln Q = \exp(1.12e + 0.8342) \quad (15)$$

$$Q = \text{Exp}[\exp(1.12e + 0.8342)] \quad (16)$$

Equation (16) is the derived model which is triple exponential in nature.

Where

= Percentage concentration of dissolved iron in oxalic acid solution.(%)

N=0.8929(Dissolution coefficient of iron in oxalic acid solution during the leaching process) determined in the experiment (Nwoye,2000).

Q=Quantity of heat energy absorbed by the oxalic acid solution during dissolution of the iron.(J)

Table 1: Variation of percentage concentration of dissolved iron with quantity of heat absorbed by oxalic acid solution. (Nwoye,2000)

M (g)	(%)	Q (J)
22	0.0083	1228.92
26	0.0110	1258.18
30	0.0069	1254.00
34	0.0077	1195.48
38	0.0095	1254.00
42	0.0040	1224.74

Where M = Mass of iron oxide ore used for the leaching process (g)

3. Boundary and Initial Condition

Consider iron ore in cylindrical flask 30cm high containing leaching solution of oxalic acid. The leaching solution is stationary i.e (non-flowing). The flask is assumed to be initially free of attach bacteria. Initially, atmospheric levels of oxygen are assumed. Range of mass of iron oxide ore was used: (4-14g). The initial pH of leaching solution; 6.5 and leaching time of 30 minutes was used for all samples. A constant leaching temperature of 25°C was used. Ore grain size; 150µm, and oxalic acid concentration; 0.1mol/litre were used. These and other process conditions are as stated in the experimental technique (Nwoye, 2000).

The boundary conditions are: atmospheric levels of oxygen (since the cylinder was open at the top) at the top and bottom of the ore particles in the liquid and gas phases respectively. At the bottom of the particles, a zero gradient for the liquid scalar are assumed and also for the gas phase at the top of the particles. The leaching solution is stationary. The sides of the particles are taken to be symmetries.

4. Model Validation

The formulated model was validated by direct analysis and comparison of Q values from model data and those from the experimental data for equality or near equality. Analysis and comparison between these data reveal deviations of model data from experimental data. This is believed to be due to the fact that the surface properties of the ore and the physicochemical interactions between the ore and leaching solution which were found to have played vital roles during the leaching process (Nwoye, 2000) were not considered during the model formulation. This necessitated the introduction of correction factor, to bring the model data to that of the experimental values. (Table 3)

Deviation (Dv) of model predicted Q values from experimental Q values is given by

$$Dv = \left(\frac{Dp - DE}{DE} \right) \times 100 \quad (17)$$

Where Dp = Predicted data from model
DE = Experimental data

Correction factor (Cf) is the negative of the deviation i.e

$$Cf = -Dv \quad (18)$$

Therefore

$$Cf = -100 \left(\frac{Dp - DE}{DE} \right) \quad (19)$$

Introduction of the corresponding values of Cf from equation (19) into the model gives exactly the corresponding experimental Q values. (Nwoye, 2000)

5. Results and Discussion

The derived model is equation (16). Computational analysis of values in Table 1 resulted to Table 2.

Table 2: Variation of e with $0.8929 \ln[\text{Log}Q]$

e	$0.8929 \ln[\text{Log}Q]$
1.0083	1.0072
1.0111	1.0102
1.0069	1.0097
1.0077	1.0037
1.0095	1.0097
1.0040	1.0068

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he quantity of heat absorbed by oxalic acid solution per unit mass-input of iron oxide ore during the leaching process was determined following comparison of the quantity of heat absorbed by oxalic acid solution per unit mass-input of iron oxide ore obtained by calculations involving experimental results as well as derived model.

Determination of the quantity of heat absorbed by oxalic acid solution per unit mass-input of iron oxide ore.

Quantity of heat absorbed by oxalic acid solution per unit mass-input of iron oxide ore during leaching Q_1 (J/g) is calculated from the equation;

$$Q_1 = Q/m \quad (20)$$

Therefore, a plot of quantity of heat absorbed by oxalic acid solution against mass-input of iron oxide ore (as in Figure 1, line ExD) gives a positive slope, S between the first rising phase of the curve at points (1228.92, 22) and (1258.18, 26) respectively following their substitution into the mathematical expression;

$$S = Q/m \quad (21)$$

Eqn. (21) is detailed as

$$S = Q_2 - Q_1 / m_2 - m_1 \quad (22)$$

Where

Q = Change in the quantities of heat absorbed by oxalic acid solution Q_2, Q_1 at mass-input values m_2, m_1 . Considering the points (1228.92, 22) and (1258.18, 26) for (Q_1, Q_1) and (Q_2, Q_2) respectively, and substituting them into eqn. (22), gives the slope as 7.315 J/g which is the quantity of heat absorbed by oxalic acid solution per unit mass-input of iron oxide ore during the actual experimental leaching process (Nwoye, 2000). Similar plot (as in Figure 1, line MoD) of model-predicted results also gives a positive slope. Considering points (1241.98, 22) and (1269.34, 26) for (Q_1, m_1) and (Q_2, m_2) respectively and substituting them into eqn. (22) gives the value of slope, S as 6.84 J/g. This is the model-predicted quantity of heat absorbed by the oxalic acid solution per unit mass-input of iron oxide ore.

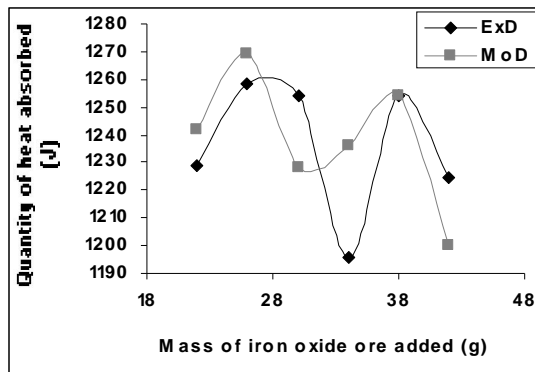


Figure 1- Comparison of the quantities of heat absorbed by oxalic acid solution relative to the mass-input of iron oxide ore (as obtained from experiment (Nwoye, 2000) and derived model)

Determination of the quantity of heat absorbed by oxalic acid solution per unit percent rise in the concentration of iron dissolved in the solution.

From Figure 2, line ExD, consideration of points (1228.92, 0.0083) and (1258.18, 0.011) for (Q_1, m_1) and (Q_2, m_2) respectively, and substituting them into eqn. (22), gives a positive slope as 10837.04 J%. This is the quantity of heat absorbed by oxalic acid solution per unit percent rise in the concentration of dissolved iron during the actual experimental (Nwoye, 2000) leaching process (Nwoye, 2000). Also similar plot (as in Figure 2, line MoD) of model-predicted results gives a positive slope. Considering points (1241.98, 0.0083) and (1269.34, 0.011) for (Q_1, m_1) and (Q_2, m_2) respectively and substituting them into eqn. (22) gives the value of slope, S as 10133.33 J%. This is the model-predicted quantity of heat absorbed by oxalic acid solution per unit percent rise in the concentration of dissolved iron. A comparison of these two values of quantity of absorbed heat show proximate agreement.

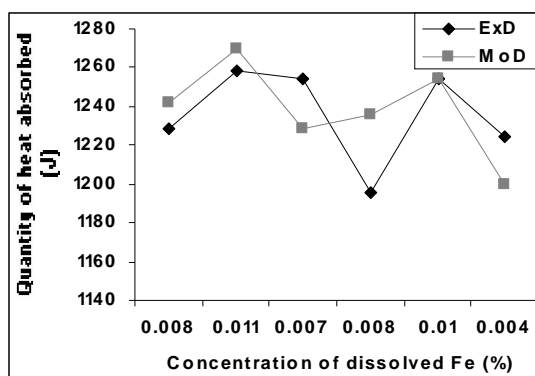


Figure 2- Comparison of the quantities of heat absorbed by oxalic acid solution relative to the concentration of dissolved iron (as obtained from experiment (Nwoye, 2000) and derived model)

Figures 1 and 2 show very close alignment of the curves from model-predicted values (line MoD) and that from the corresponding experimental values (line ExD). The degree of alignment of these curves is indicative of the proximate agreement between both experimental and model-predicted values of quantity of absorbed heat. The model predicts (as shown in Figures 1 and 2) positive values of the quantity of heat absorbed. This is in accordance with experimental results (Nwoye, 2000). The positive values of heat absorbed as obtained from experiment and model indicate and agree that the leaching process is endothermic in nature.

Table 3. Variation of model-predicted quantity of absorbed heat with the associated deviation and correction factors

Q (J)	Dv(%)	Cv (%)
1241.98	+1.06	-1.06
1269.34	+0.89	-0.89
1228.09	-2.07	+2.07
1236.01	+3.40	-3.40
1254.05	0.00	0.00
1199.94	-2.03	+2.03

Table 3 shows that the maximum deviation of the model-predicted values of Q from the corresponding experimental values (Nwoye, 2000) is less than 4% which is quite insignificant and within the acceptable range of deviation limit of experimental results hence depicting the reliability and validity of the model. The validity of the model is believed to be rooted on equation (7) where both sides of the equation are correspondingly approximately equal to 1. Table 2 also agrees with the expression $e = N \ln[\text{Log}Q]$ in equation (7) following the values of e and N $\ln[\text{Log}Q]$ evaluated from Table 1 as a result of computational and statistical analysis. Table 3, Figures 3 and 4 show that the least and highest magnitude of deviation of the model-predicted Q (from the corresponding experimental values) are +0.89% and +3.4% which correspond to the quantities of absorbed heat: 1269.34 and 1236.01 J, concentrations of dissolved iron: 0.011 and 0.0077% (Figures 1 and 2) and mass-input of iron oxide ore (Table 1): 26 and 34g respectively. Table 3, Figures 5 and 6 indicates that the least and highest correction factor: -0.89% and -3.4% make up for the least and highest deviation of +0.89% and +3.4%. These correction factors also corresponds to quantities of absorbed heat: 1269.34 and 1236.01J, concentrations of dissolved iron: 0.011 and 0.0077% and mass-input of iron oxide ore: 26 and 34g respectively.

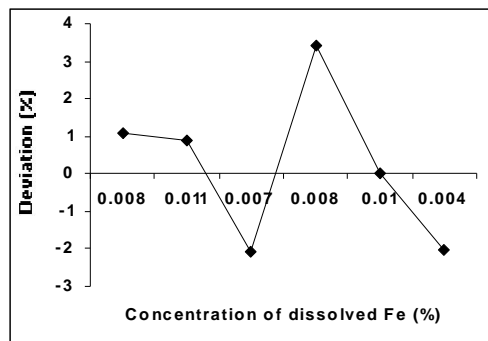


Figure 3- Variation of deviation (of model-predicted quantity of absorbed heat from experimental values) with the concentration of dissolved iron obtained from experiment (Nwoye, 2000)

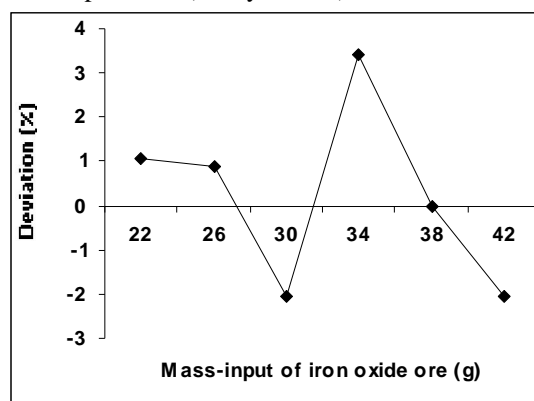


Figure 4- Variation of deviation (of model-predicted quantity of absorbed heat from experimental values) with the mass-input of iron oxide ore for the experiment (Nwoye, 2000)

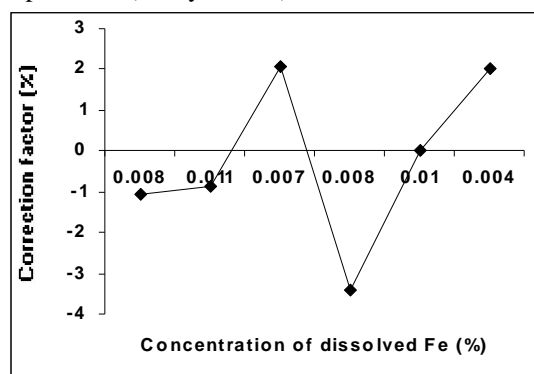


Figure 5- Variation of correction factor to (model-predicted quantity of absorbed heat) with the concentration of dissolved iron obtained from experiment (Nwoye, 2000)

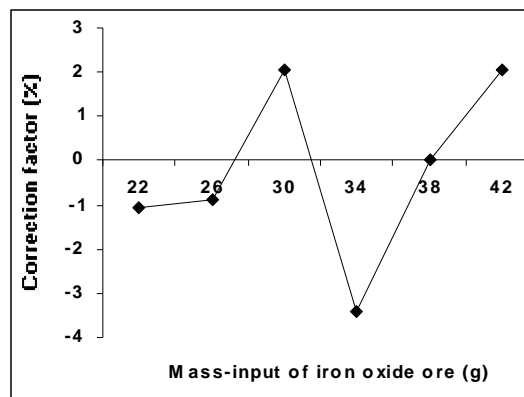


Figure 6- Variation of correction factor to (model-predicted quantity of absorbed heat) with the mass-input of iron oxide ore for the experiment (Nwoye, 2000)

6. Conclusion

The model calculates the quantity of heat absorbed by oxalic acid solution as a result of the dissolution of iron during leaching of Itakpe iron oxide ore. The triple exponential model can also predict the concentration of dissolved iron associated with heat absorbed by the oxalic acid solution during the leaching process (providing that $e = 1$ and the value of Q known). The validity of the model is believed to be rooted on the expression $e = N \ln [\text{Log}Q]$ where both sides of the equation are correspondingly approximately equal to 1. The deviation of the model-predicted Q values from the corresponding experimental Q values is less than 4% which is quite within the acceptable range of deviation limit of experimental results. The positive values of heat absorbed as obtained from experiment and model indicate and agree that the leaching process is endothermic in nature. The quantities of absorbed heat per unit mass-input of iron oxide ore as obtained from experiment and derived model were evaluated as 7.315 J/g and 6.84 J/g respectively. Furthermore, the quantities of absorbed heat per unit percent rise in the concentration of dissolved iron as obtained from experiment and derived model were evaluated as 10837.04 J/% and 10133.33 J/% respectively, indicating proximate agreement.

It is expected that more process parameters should be incorporated into the model in further works with the aim of reducing the deviations of the model-predicted Q values from those of the experiment.

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