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A STUDY ON THE PERFORMANCE OF SECOND ORDER MODELS AND TWO PHASE MODELS IN IRON ORE FLOTATION

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In this study, the fit to the experimental data of two second order models and two phase model were compared with first order models. The best fit was observed with the two parameter model with rectangular distribution of floatabilities (model II) and the three parameter fast/slow floating particles model (model I). Also, the three parameter gamma distribution model (model VI) showed good fit to the experimental data. The worst fit was observed with first order two stage kinetic model (model V) which indicates that there is no need to divide the flotation rate into two flotation rates. Model VII which includes six parameters gave acceptable fit to experimental data. This result is in contrast with the results obtained by other investigators which states that increase of parameters in model leads to parameter dilution and increase on model error. Hence, it may be concluded that the number of parameters to obtain an adequate model needs more investigation. The confidence intervals for the three best models (model I, model II and model VI) were estimated at 95% confidence level. Also, model II shows more discrete regions with regard to flotation rate and recovery than model VI. This result indicates the ability of such model to express the changes in flotation reagents and/or flotation conditions over other tested models.

key words: flotation, modeling, second order models, two phase models

INTRODUCTION

The importance of describing the flotation process by a mathematical model has been realized by numerous researchers (Arbiter and Harris 1962, Jowett and Ghosh 1965). Models provide the worker with a means to assess and predict flotation cell performance and hence the opportunity to improve operation of the cell.

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Distinguishing between the performance of different flotation cells and evaluating the effects of operating parameters (Such as aeration rate and agitation intensity) can also be accomplished by use of a flotation model (Mehrotra and Kapur 1974, Mika and Fuerstenau 1969). Examination of the literature dealing with the modeling of flotation process reveals that a number of varying models have been presented (Woodburn et al. 1976, Woodburn 1970). No particular model has gained predominant acceptance amongst flotation researchers.

The mathematical modeling of flotation was applied in many areas in the flotation literature. Gulsoy (2005) analyzed the entrainment behaviour of both hydrophilic and hydrophobic particles in flotation. Empirical model equation was proposed and checked with experimental observations. The results provided accurate interpretation of hydrophilic and hydrophobic particles entrainment. Mathe et al. (2000) studied also the effect of entrainment of hydrophilic particles on modeling of recovery of mineral grains in concentrate. Heindel (1997) analyzed the microprocesses associated with the deinking flotation. He made a comparison between deinking and mineral flotation and concluded that modeling deinking flotation is possible. Heindel and Bloom (2006) mentioned that although there are many differences between conventional mineral flotation and dispersed air flotation, the latter can be modeled and they gave some guidelines for modeling such complex process.

Yianatos (2007) reviewed the flotation models that depend on fluid flow and models that depend on kinetics of related processes in conventional and column flotation. He mentioned that due to deviation of mechanical cells from perfect mixing conditions, the fluid flow regime, mass transport between pulp/froth interface and froth transport mechanism are better understood. The simulation of flotation process/circuits was also attempted. The results of such simulations are found for example in (Harris 1997, Alexander et al. 2000).

In a previous study (Saleh 2009) statistical model analysis was carried out on single phase first order models. The results showed that the three parameter fast/slow floating particles model and the two parameter model with rectangular distribution of floatabilities were the best models. In the present investigation, models of second order and two phase type are tested, evaluated and compared with first order models in iron ore flotation.

CONSIDERED FLOTATION MODELS

The following models are tested and analyzed in this work.

1 – Three parameters fast/slow floating particles model (named model I). This model mathematically expressed as:

$$r = (1-\Phi)[1-\exp(-K_f\, t)] + \Phi[1-\exp(-K_s\, t)] \tag{1}$$

where r is recovery at time (t), Φ is the fraction of flotation components with slow rate constant, K_f, K_s are the rate constants for fast and slow components (min^{-1}). This model incorporates two rate terms instead of one rate constant (Kelsall 1961). The model does not include an ultimate recovery parameter but rather the ultimate fractional recovery is assumed to be 1.0. When K_s parameter approaches 0, the parameter *(Φ)* will then represent the fraction not recovered and the term *(1 - Φ)* becomes analogous to ultimate recovery and the model reduces to two parameter model.

2 – Two parameter model with rectangular distribution of floatabilities (named model II). Mathematically, this model is described as:

$$r = R[1 - \frac{1}{Kt}(1 - \exp(-Kt))] \tag{2}$$

where R is the ultimate recovery at long flotation times, K is the rectangular rate constant representing the largest allowed value of rectangular distribution (min^{-1}) and r as in model I. This model assumes a rectangular distribution of rate constants (Meyer and Klimpel 1984). The model describes a first order reaction. The predicted recovery is based on both ultimate recovery and a rectangularly distributed rate constant.

3 – Second order kinetic model (named model III). Because a first order equation appears to apply to a limited number of time – recovery profiles, it is not a sufficient criterion alone to establish the order of a flotation rate equation. This model is developed by assuming n=2 in the rate equation ($dc/dt = -Kc^n$) where c is the concentration of a particular floatable component, K is the rate constant and n is the order of rate constant. Mathematically it is expressed as:

$$r = \frac{R^2 Kt}{1 + RKt} \tag{3}$$

R is the ultimate recovery and K is the flotation rate constant. This model is a two parameter expression describing the flotation of a monodisperse feed with particles having a constant floatability (Arbiter, 1951). The terms r, R and K are the same as mentioned in model II.

4 – Second order kinetic model with rectangular distribution of floatabilities (named model IV). This model was proposed by Klimpel (1980). The mathematical form of this two parameter model is given as:

$$r = R\{1 - \frac{1}{Kt}[\ln(1 + Kt)]\} \tag{4}$$

The terms r, R and K are the same as in model II. This model assumes a rectangular distribution of floatabilities.

5 – First order two stage kinetic model (named model V). This model was developed by considering the flotation system as being composed of discrete pulp and froth volumes. The model as proposed by Klimpel (Klimpel, 1984) incorporates two rate terms describing the mass transfer of a component from the pulp to the froth and finally to the concentrate. By assuming that the rate of drainage from the froth is minimal, the mathematical form of this model is derived as:

$$r = R\{(\frac{K}{K-K'})(1-\exp(-K't)) - (\frac{K'}{K-K'})(1-\exp(-Kt))\} \quad (5)$$

K is the rate of transfer from pulp to froth and K' is rate of transfer from froth to concentrate. Equation (5) is a three parameter model, describing a first order, two stage reaction. The particles are assumed to be present in a monodisperse feed with constant floatabilities. As K (rate of transfer from pulp to froth) is always much greater than K' (rate of transfer from froth to concentrate), transfer from froth to concentrate is the rate limiting step.

6. Three parameter gamma distribution model (named model VI). This model assumes a gamma distribution of floatabilities instead of rectangular distribution and was developed by (Huber–Panu et al., 1976). It is mathematically expressed in the following form:

$$r = R\{1-[\frac{\lambda}{\lambda+t}]^P\}. \quad (6)$$

The gamma distribution can be described as being composed of the summation of P exponential distributions. When P = 1.0, this model will be reduced to a two parameter form.

7. Six parameter model (named model VII). This model discretises the floating material into fast, medium and slow floating components (Apling and Ersayin ,1986). It may be represented by the equation:

$$\mathrm{r} = \sum_{i=1}^{n} \mathrm{a_i}(1-\exp(-K_i t) \quad (7)$$

where r is the recovery of the _grains at time *(t)* and a_i is the fraction of the grains that is characterized by rate constant K_i such that:

$$\sum_{i=1}^{n} \mathrm{a_i} = 1.0. \quad (8)$$

Models I and II were tested for coal flotation in a previous study (Saleh 2009) and obtained the best fit among other models tested, with model I better than model II. It was aim of this investigation to test these models and compare them to the performance of second order and two phase models for iron flotation.

EXPERIMENT

MATERIAL

Iron ore sample used in this study was obtained from Um-Hebal region, South-East of Aswan, Egypt. The sample was first crushed in a hammer mill and then ground in a disc crusher. The ground product was screened on a 0.5 mm wedge wire screen using Rotap Shaker. The undersize material was collected and the oversize product was returned to disc mill and screened again until all material passed 0.5 mm sieve. The collected material [-0.5 mm] represents the flotation feed (-500 µm). A representative sample from the flotation feed was taken and analyzed. The chemical analysis of this sample are: K_2O=0.1%, Na_2O<0.1%, TiO_2 = 0.55, MnO = 1.57, Fe_2O_3 = 57.5%, SiO_2 = 17%, Al_2O_3 = 3.57, CaO = 5.38%, P_2O_5 = 3.02% and L.O.I. = 9.35%. The flotation feed was screened and analyzed for Fe content. The size distribution and iron content of different size fractions are shown in Table 1. Oleic acid was applied as a collector and pine oil was used a frother.

Table 1. Size distribution and iron content of flotation feed

Size, micron	Yield *(γi)* wt%	Cumulative yield *($\Sigma\gamma_i$)*	Fe content %
-500+355	9.38	100	35.90
-355+250	15.48	90.62	38.10
-250+125	35.54	75.14	38.05
-125+62	32.82	39.60	40.00
-62+32	11.88	15.78	44.70
-32	3.90	3.90	48.00
Feed Sample	100		38

METHODS

Flotation experiments were carried out in Denver laboratory sub aeration machine of one dm^3 capacity using tap water. The operating conditions are shown in Table 2.

The ore was well mixed and sampled using standard riffling procedure. The 100 g samples were taken, placed in bags and stored in a dry environment until testing. The froth was removed manually. After the ground material was transferred to the cell, the pulp level was adjusted to the appropriate height. It was found that the applied frother concentration was sufficient to produce a stable froth persistent for entire flotation time (8 min).

Table 2. Operating conditions

Operating Variable	Value
Oleic acid collector	2 kg/Mg and 3 kg/Mg
Pine oil frother	60 mg/l
Solid/liquid ratio	10%
Aeration rate	6 l/min
Flotation time	0.5, 1.0, 2.0, 4.0, 6.0, 8.0 min
r. p. m.	1000
pH	8

The collector reagent is used industrially for this type of ore. The collector was provided by the supplier in a purified state, used as it received and added to the flotation cell using clean, calibrated pipette.

In carrying out a flotation test, the pulp was first agitated for 5 min and then conditioned with oleic acid collector for another 5 min. The pine oil frother was added 1 min before aeration.

To study the flotation kinetics, the collection of concentrate was taken at time intervals 0.5, 1.0, 2.0, 4.0, 6.0 and 8.0 min. The concentrate and tailing from each test were filtered, dried, weighed, sampled and assayed for Fe content and the fractional recovery.

RESULTS AND DISCUSSION

The optimal flotation model parameters were determined by a generalized parameter estimation computer program (Ureka-the solver). The criteria used for estimation of parameter values is the minimization of the absolute sum of the squares of the deviations at a given time between observed (experimental) and calculated recovery. Having the optimal parameters for a given model, a procedure for selecting the best flotation model remains to be found.

EVALUATION OF FLOTATION MODELS

A method of model discrimination used is based on a statistical analysis of the model error resulting from curve fitting as compared to the true experimental error. The requirements of a floatation model are first: a model must fit the observed data and, second: each model parameter must have a range of statistical significance narrow enough such that changes in flotation system can be confidently assessed. By using standard deviation of estimate, $S_{r,}$ the fit of the observed to the calculated data can be measured (Eq. 9).

$$S_r = \sqrt{\frac{\sum_i^n (r_{i,cal} - r_{i,\,obs})^2}{n-m}} \tag{9}$$

In this equation, n equal to number of data points, m is the number of model parameters; $r_{i,cal}$ is the calculated recovery at time i and $r_{i,obs}$ is the observed or experimental recovery rate at time i.

Hence, flotation models have been evaluated on the basis of the quality of fit of the experimental data. Model that gives parameters when evaluated at various flotation times will give a predicted flotation time-recovery profile that is nearly identical to the observed flotation time-recovery profile is assumed to be the best model. Because the experimental error is constant in all tests, the difference between experimental profile and predicted profile of two models will be a direct measure of the difference in the model errors.

Figure 1 shows the flotation response of the iron ore sample tested. It represents the flotation recovery % versus flotation time (min) at collector dosage of 2 kg/Mg and 3 kg/Mg. It is clear that the recovery increases with increasing flotation time. The increase in flotation recovery is small at high flotation times and the recovery tends to be, more or less, constant after 6 minutes. The flotation response of feed sample is poor where the maximum obtained flotation recovery (at 2 kg/Mg collector) is about 50% after long flotation time (about 8 min). The poor flotation response may be related to the geological nature of the flotation feed, i.e. the valuable mineral (hematite) may be finely disseminated in the ore matrix which needs extensive grinding to achieve acceptable mineral liberation. This is clear from Fig. 1 where increase collector dosage from 2 kg/Mg to 3 kg/Mg does not lead to a significant increase of recovery (55% vs. 50%) with the same concentrate grade 48% Fe compared with the feed grade 38% Fe.

The optimal model parameters for models (I-VII) as well as the maximum error in predicted flotation recovery and the standard error of *estimate* (S_r) are shown in Table 3. The results of simulation of the investigated models are shown in Figures 2 through 5. All tested flotation models expressed the recovery-time profile successfully as the standard error of estimate may be more or less acceptable where it is ranging from 0.010 to 0.034, i.e., from 1%-3%. The lowest performance is observed with the first order two stage kinetic model (model V) which include two stage rate constants. The first expresses the transfer of particles from pulp to froth and the second expresses the rate of transfer of particles from froth to concentrate.

Table 3. Model parameters, maximum error and standard error of estimate (S_r) at flotation dose 2 kg/Mg collector

Model	Parameters	Max. error	S_r
I	K_f=1.3374 K_s=0.0349 Φ =0.6222	0.0254	0.0130
II	K =2.0522 R =0.5221	0.0129	0.0100
III	K=2.1663 R=0.5494	0.0231	0.0148
IV	K=2.5609 R=0.5901	0.0286	0.0184
V	K=2.3432 K'=2.4014 R=0.4637	0.0342	0.0341
VI	λ=2.6595 P=2.9025 R=0.4959	0.0128	0.0130
VII	a_1=0.4095 a_2=0.0106 a_3=0.5800 K_1=1.2020 K_2=0.7050 K_3=0.0171	0.0098	0.0138

This indicates that dividing flotation rate constant into two rate constants, i.e. considering that flotation process consists of two stages instead of single stage is useless or have no additional benefit. The best fit to the experimental data is obtained with the first order two parameters model with rectangular distribution of floatabilities (model II) and the three parameter fast/slow floating particles model (model I). This result is in agreement with the obtained results in a previous work carried out on coal flotation (Saleh 2009). In that investigation, these models recorded the best performance among other tested first order models. This work indicates that the performance of first order models is better than second order models as better fit to experimental data is observed.

Three parameter gamma distribution model gave better fit than second order models. This result shows that the flotation process can be better described as first order process than other high order processes. The six parameter model (model VII) which includes classification of floating particles into three fractions *(a_1, a_2, a_3)* with fast, medium and slow flotation rates *(K_1, K_2, K_3)* has good fit to the experimental results better than second order models. This result is in contrast with other results obtained by other investigators (Apling and Ersayin 1986) which stated that no benefit is gained from increasing number of parameters in the flotation model. Hence, it seems that this conclusion needs more investigation in the future studies. The performance of tested models with regard to fit is in following order: model II > model I = model VI > model VII > model III > model IV > model V.

The best two models (model I and model II) were tested also with dosage 3 kg/Mg oleic acid collector. The standard error of estimate *(Sr)* were 0.024 and 0.023 respectively compared with 0.013 and 0.010 at 2 kg/Mg collector. It is clear that the standard error of estimate depends on the flotation conditions and increases with collector dosage increase. It's worth to mention that among the tested models, two parameters model with rectangular distribution of floatabilities (model II) and second order kinetic model with rectangular distribution of floatabilities (model IV) don't respond at the point of zero time. Among tested second order models (models III, IV), second order kinetic model (model III) obtained better results.

Better performance of model II over model I may be related to the assumption that the ultimate recovery is 1.0. It is believed that the rectangular distribution of floatabilities gives this model flexibility and therefore, should be a better form of the first order process. Quality of fit for model II is the best of all models. Because the mathematical form of model III includes the square of the ultimate recovery parameter and due to assumption of the second order, additional model error over and above that inherent in first order models may be introduced. However, as in model III, the second order form in model IV introduced additional parameter dilution. In model VI the fit to the observed data is excellent and better than of some two parameter forms. Although it was concluded that little or no benefit is derived from increasing number of parameters in the model, i.e., the smaller the number of parameters that are required by a model to achieve an adequate fit to experimental data, the easier the model relates changes in their magnitude to changes in operational variables (Apling and Ersayin 1986), six parameter model (model VII) shows good fit to the observed data.

PARAMETER CONFIDENCE REGIONS

The parameter confidence regions were estimated for the three best models, namely, model I, model II and model VI.

Due to the non-linear nature of the flotation models, confidence limit estimates of parameters based on linear model methodology can not be applied. Instead, logical sensitivity test procedure was applied (Klimpel and Austin 1984).This procedure involved a stepwise change (±10%,±25%,±50%) in one of the optimal model parameters values. By holding the changed parameter constant and allowing the computer program to reoptimize, a new estimate of model parameters and the sum of squares between observed recovery and calculated recovery was determined. The sum of squares is then used to calculate the model variance (S^2_r) which used in statistical *F-Test*. By comparing the optimal model variance to the model variance from a given change in the parameter being assessed, a calculated *F*-value can be determined. By repeating this process, a plot of calculated *F* versus the parameter value can be made. If calculated *F*-value is less than the *F*-distribution (*F*-value from the tables, i.e., F_{crit}) then there is no significant difference between

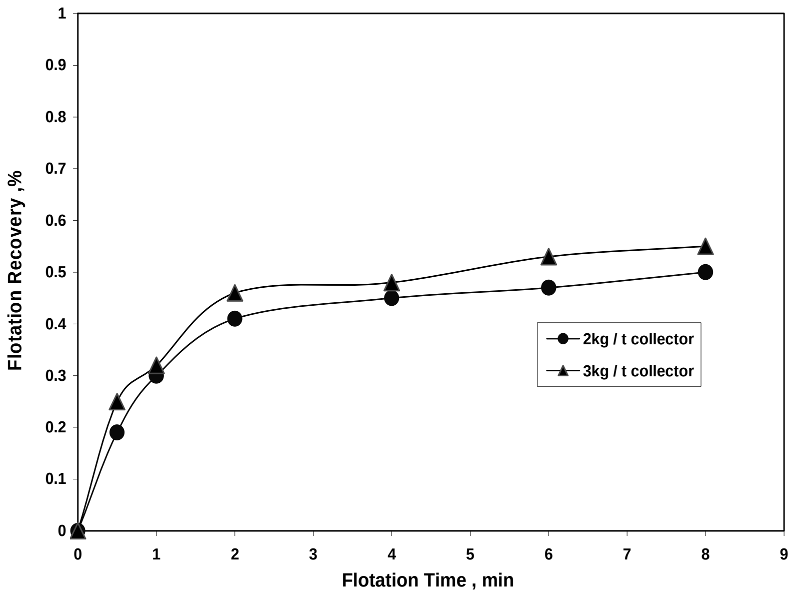

Fig. 1. Flotation response of considered feed sample at 2 kg / Mg and 3 kg / Mg collector

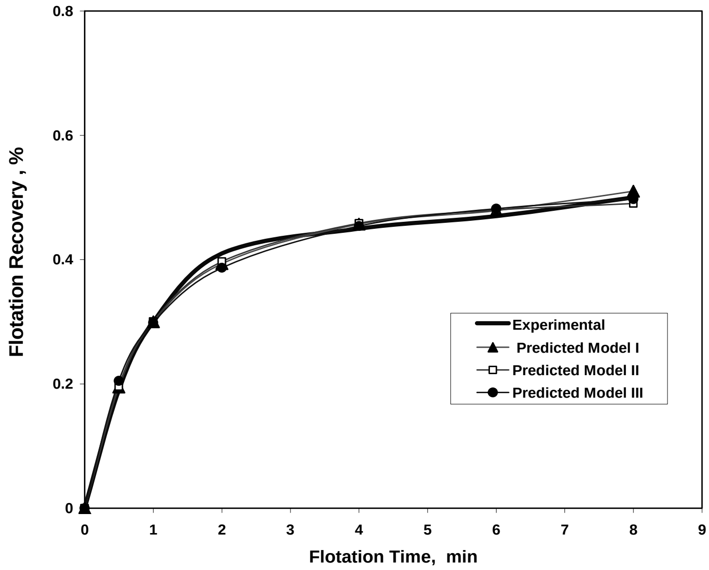

Fig. 2. Experimental and predicted flotation recovery for models I , II and III

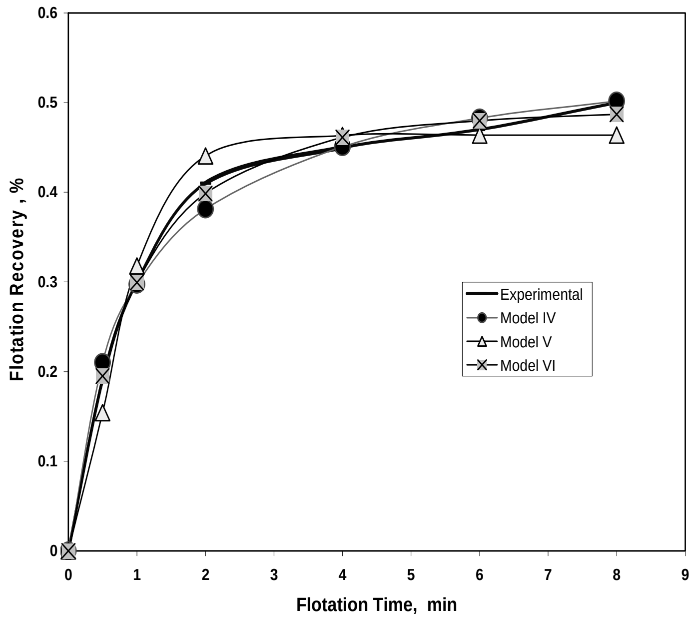

Fig. 3. Experimental and predicted flotation recovery for models IV, V, and VI at 2kg / Mg collector

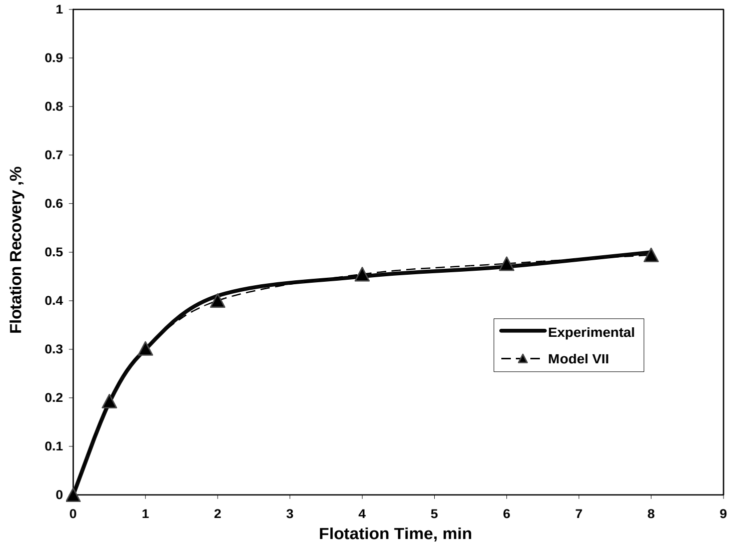

Fig. 4. Experimental and predicted flotation recovery for model VII at 2 kg / Mg collector

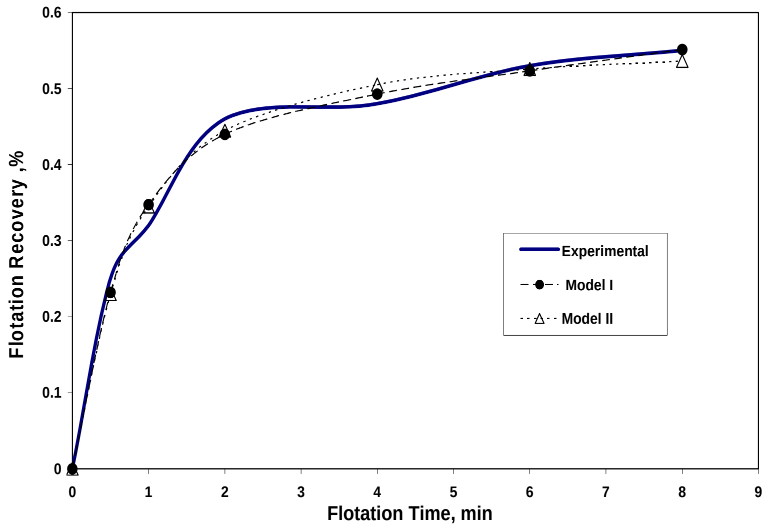

Fig. 5. Experimental and predicted flotation recovery for models I and II at 3 kg / Mg collector

the optimum parameter value and the changed parameter value and null hypothesis *(H_o)* is applied. On the other hand, if calculated *F*-value is greater than the $F_{crit,}$ the alternative hypothesis *(H_1)* is applied which states that there is a significant difference between the optimum parameter value and the charged parameter value.

If a model gives parameters with a wide range of statistical significance, the differences that can be observed between different tests become diluted or completely masked and it's impossible to make firm quantitative comparison of various test conditions. On the other hand, if the model gives parameters with a narrow range of statistical significance, differences between tests become apparent.

This method of calculation is illustrated in a working example for model II in tables (4, 5). The confidence limits for K and R parameters were determined by the graphical technique at 95% confidence level and shown in Figures (6, 7).

Figure (8) shows comparison of confidence intervals for ultimate recoveries of models II and VI. The approach was continued for each parameter in each of considered models (I, II, VI). Table (6) shows the confidence limits of the three best models, i. e, model I, model II and model VI.

It's clear that for model II while the confidence regions of K is symmetrical, the confidence region of R is not. The confidence region of R of model VI is also, more or less, symmetrical as shown on Fig. 8.

Investigating the above results indicates that with regard to *(R)*, the range of statistical significance in model VI is less than model II. The confidence limits for R and K are very narrow for model II. The confidence limits for Φ and R in models I and VI are, more or less, the same. It is expected that additional parameters in model VI may introduce model error and parameter dilution comparing with simpler form of model II. Determination of confidence intervals for second order models are recommended in future work to be compared with that of first order models. Among the tested models and with regard to quality of fit and confidence intervals, the two parameter model with rectangular distribution of floatabilities (model II) is the best model. The increase in model parameters still needs more investigation in the future studies. The first order models recorded better performance than second order models. Three parameter gamma distribution (model VI) shows a fit to the experimental data more or less similar to that observed with three parameter fast/slow floating particles model (model I). Models I, II obtained the best fit to the experimental data as previously observed in coal flotation (Saleh 2009). The worst fit was observed with the first order two stage kinetic model (model V).

Table 4. Determination of parameter confidence range of K by F-Test at 95% confidence level for model II

	K	R	SSQ	S_r^2	F
Opt.	2.0522	0.5221	0.00045	0.00010	1.0
+10%	2.2574	0.5129	0.00070	0.00018	18
-10%	1.8470	0.5329	0.00077	0.00019	19
+25%	2.5653	0.5013	0.00182	0.00045	45
-25%	1.5391	0.5531	0.00286	0.00072	72

Table 5. Determination of parameter confidence range of *R* by *F*-Test at 95% confidence level for model II

	K	*R*	*SSQ*	S_r^2	*F*
Opt.	2.0522	0.5221	0.00045	0.00010	1.0
+10%	1.8316	0.5743	0.00109	0.00027	2.7
-10%	2.2855	0.4699	0.00048	0.00012	1.2
+25%	1.5163	0.6526	0.00484	0.00121	12.1
-25%	2.6676	0.3916	0.00409	0.00102	10.2

SSQ=sum of squares of differences

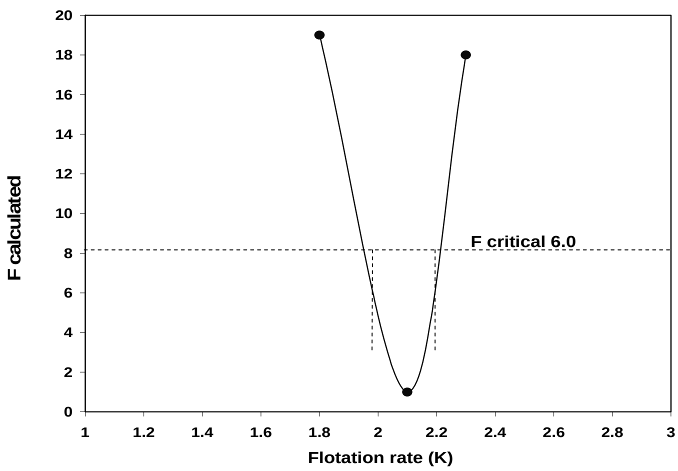

Fig. 6. Graphical determination of parameter confidence ranges for flotation rate (*K*) for model II

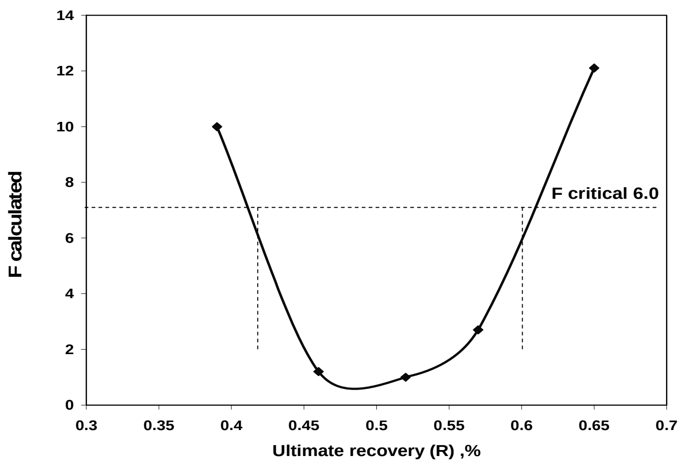

Fig. 7. Graphical determination of parameter confidence ranges for ultimate recovery (*R)* for model II

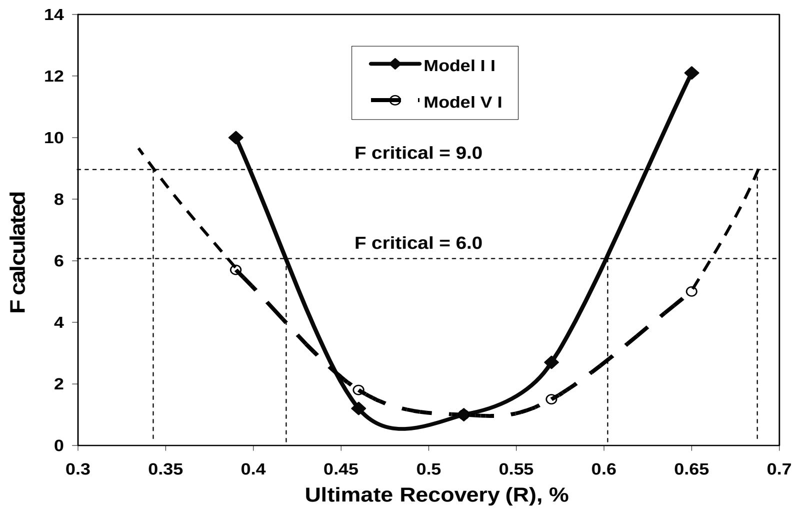

Fig. 8. Comparison of confidence ranges for ultimate recovery (R) for models II and VI

Table 6. Confidence intervals for model I, model II and model VI

Model	Parameter	Optimum	Range
I	K_f	1.3374	0.75 - 2.25
	K_s	0.0349	*
	Φ	0.6222	0.40 - 0.75
II	K	2.0522	1.97 - 2.20
	R	0.5221	0.42 - 0.60
VI	λ	`2.6595	*
	P	2.9025	*
	R	0.4959	0.34 - 0.68

* Parameter insensitive to 50% change

CONCLUSIONS

In this investigation, seven flotation models were tested in iron ore flotation. These models include, three parameter fast/slow floating particles model (model I), two parameter model with rectangular distribution of floatabilities (model II), second order kinetic model (model III), second order kinetic model with rectangular distribution of floatabilities (model IV), first order two stage kinetic model (V), three parameter gamma distribution model (model VI) and six parameter model (model VII). The best fit was observed for models I and II with model II better than model I.

The fit to the experimental data is found in following order: model II > model I = model VI > model VII > model III > model IV > model V. The first order models obtained better performance than second order models. The tested six parameter model (VII) showed also good fit to the experimental data. This result shows need for more investigation to illustrate the effect of increase parameters in model and to determine their upper limit to provide an adequate model. The worst fit was observed with the first order two stage kinetic model (model V) which indicates that dividing flotation rate into two rates, the first for transfer particles from pulp to froth and the second for transfer particles from froth to concentrate is incorrect.

The confidence intervals for the three best models (model I, II, VI) were estimated by the graphical technique at 95% confidence level. It was found that model II shows discrete confidence regions which illustrates the ability of this model to express changes in flotation conditions. In comparison with model II, and with regard to flotation recovery, model VI showed less discrete (wide) confidence regions.

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Porównano wierność opisu danych pomiarowych za pomocą kinetycznych modeli drugiego rzędu i modeli dwufazowych. Najlepszy opis odnotowano za pomocą modelu dwuparametrowego z potęgową dystrybucją flotowalności (model II) oraz trójparametrowego modelu dla szybko/wolno flotujących ziarn (model I). Także trójparametrowy model oparty o γ-dystrybucję (model VI) dobrze opisywał dane pomiarowe. Najgorsze dopasowanie obserwowano dla modelu pierwszego rzędu dwustopniowego modelu kinetycznego (model V), co wskazuje, że nie ma potrzeby dzielenia szybkości flotacji na dwie części. Model VII, który zawiera sześć parametrów, także daje poprawny opis danych. Wynik ten kontrastuje z wynikami otrzymanymi przez innych badaczy twierdzących, że wzrost liczby parametrów modelu prowadzi do rozmycia oraz wzrostu błędu modelu. Przy 95% poziomie ufności wyznaczono przedziały ufności dla najlepszych modeli I, II oraz VI. Ponadto model II wykazał więcej dyskretnych obszarów w odniesieniu do prędkości flotacji i uzysku, niż model VI. Wskazuje to na lepszą zdolność tych modeli do opisu zmian w procesie flotacji.

słowa kluczowe: flotacja. modelowanie, modele drugiego rzędu, model dwufazowe