CANADA

DEPARTMENT OF ENERGY, MINES AND RESOURCES

OTTAWA

MINES BRANCH INVESTIGATION REPORT IR 68-50

FACTORIAL DESIGN AND REGRESSION ANALYSIS OF COPPER-ORE FLOTATION TESTS

by

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EXTRACTION METALLURGY DIVISION

COPY NO. 17

SEPTEMBER 4, 1968

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SUMMARY of RESULTS

A designed series of flotation tests was performed with a chalcopyrite-bearing ore to determine if the operating variables and responses could be related quantitatively by regression-analysis techniques. The test results permitted the development of only one completely satisfactory mathematical relationship. This model shows the relationship of particle size, pulp density and ore grade to the grade of copper concentrate produced by flotation. Other mathematical models developed failed to account for more than 68 per cent of the response variation over the range of the variables investigated.

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INTRODUCTION

Makepeace[1] states that "the objective of designed experiments is to obtain more information at less cost than can be obtained by classical experimentation". With this thought in mind, we decided to design a series of experiments to determine to what extent operating variables and responses of the rougher step in a chalcopyrite flotation-system could be related quantitatively by regression techniques.

SAMPLES AND MINERALOGY

Two samples were received from Opemiska Copper Mines (Quebec) Limited. One of these contained 1.44 per cent copper, while the other sample contained 6.31 per cent Cu. Samples from the Opemiska Mine were chosen for this work because the copper content of the ore is due to a single mineral, chalcopyrite, which is amenable to flotation.

The mineralogical examination of the ores by Messrs. M. R. Hughson and S. Kaiman showed that, in the high-grade ore, chalcopyrite was the major mineral and occurred as coarse masses along with minor amounts of pyrite, pyrrhotite, magnetite, molybdenite and linnaeite. The low-grade sample contained chalcopyrite more commonly as small blebs, usually less than ½ mm across, in massive pyrite. There were also lesser amounts of magnetite, molybdenite and pyrrhotite in this sample. The gangue in both samples was chiefly biotite and amphibole with minor amounts of quartz, feldspar, calcite, sphene, chlorite, apatite and sericite.

PROCEDURE

The series of tests was done in a 1000-gram glass Fagergren cell on a batch basis. The volume of air was measured by a "Precision" wet test meter which was attached to the air intake valve of the cell. A few exploratory flotation-tests showed the optimum float-time to be about 6 minutes and therefore this time interval was adopted for all tests.

The range of the variables bracketed the operating conditions used at the Opemiska mill. The variables investigated along with the levels used are shown in Table 1.

TABLE 1

Independent Variables Investigated

Independent Variable	Levels Used
Particle Size of Ore (%-200 mesh)	63, 72, 81
Pulp Density (% solids)	19, 26, 33
Lime Added (lb/ton dry ore)	0.6, 0.9, 1.2,
Dow Z-200 (lb/ton dry ore)	0.075, 0.11, 0.15,
Cyanamid R-208 (lb/ton dry ore)	0.015, 0.030, 0.045,
Triethoxy-butane (lb/ton dry ore)	0.06, 0.09, 0.12,
Air (cfm)	0.13, 0.18, 0.25
Copper Content of ore	1.44, 6.31
Promoter Z6 (same for each test lb/ton dry ore)	0.003

The statistical design used on the high-grade sample is a 1/16 fraction of a 2^7 factorial design. A 1/8 fraction of a 2^7 factorial design was used with the low-grade sample to reduce the confounding of the variables in the mathematical models of an overall copper system. Four tests with each ore sample were replicate tests done at an intermediate level to provide an estimate of the experimental error in the systems. The total number of tests done on the high-grade sample is, therefore, 12, and on the low-grade, 20. Two system-responses, concentrate grade of copper and copper recovery were measured for each run.

The experimental design allows for the development of first-order relationships between the independent variables and the measured responses. Assuming that all the variables are significant, the statistical model for each response in the low- and the high-grade ore systems would contain the following terms:

$$\text{Response} = Bo + B_1 X_1 + B_2 X_2 + B_3 X_3 + B_4 X_4 + B_5 X_5 + B_6 X_6 + B_7 X_7 + \text{error} \quad (1)$$

The copper grade of the two ore-samples is treated as a variable in the overall design to correlate the test results from both series of tests and thereby obtain mathematical models for the overall range. Because the overall system contains the additional independent variable, namely, copper grade, the general model for this system would be:

$$\text{Response} = \text{equation (1)} + B_8 X_8 + \text{error} \quad (2)$$

In equation (1) and (2), Bo is a constant term, B_1 to B_8 are the regression coefficients or parameters to be determined, while X_1 to X_8 are the independent variables.

The specific numbers for the coefficients shown in the general models were calculated with the Departments' CDC-3100 computer using a program developed by the staff of the Extraction Metallurgy Division.

RESULTS AND DISCUSSION

The results of the flotation tests done with the various levels of the independent variables are shown in Table 2. The predicted amounts for the concentrate grade and recovery in the overall copper system, which were calculated from the regression equation, are also given in this table. The regression equations and their supporting statistics are shown in the Appendix by Tables A to F.

Empirical models that define the relationship between each measured response (concentrate grade and recovery) and the independent variables (X_1 to X_8 of Table 2) have been fitted to the data for each of the three systems (low-grade copper, high-grade copper and overall copper). Statistically, only one of the models can be classified as a predictive equation. This best fit to the total test-results was obtained when the correlation was between the concentrate grade, as the response, and the operating variables. The resulting model; Conc. grade, % Cu = 15.21 + 0.081 (% minus 200 mesh) - 0.22 (% solids) + 0.95 (% Cu in feed), accounts for 83% of the observed variation in the response where the actual test results show concentrate grades ranging from 13.7 to 24.9% Cu. This model indicates that changes in reagent additions within the ranges investigated have no effect on the concentrate grade. It also shows that the concentrate grade increases with an increase in fineness of grind of the ore and per cent Cu in feed but decreases with an increase in pulp density.

The remaining models all fail to explain more than 68 per cent of the response variation over the range of the variables investigated. These remaining models, therefore, can be used only as an approximate description of the behaviour between the response and the independent variable in each system. If a variable is insignificant it means that the variable, in the range investigated, has no effect on the observed variation in the response greater than that due to experimental error. It does not mean that the variable can be eliminated from the process. Although the range of the variables bracketed those conditions used at the Opemiska mill, it may have been too restricted to permit its effect to register, statistically (3).

The analysis of data from the tests with the low-grade sample by regression methods shows that the most significant variables affecting the grade of concentrate are pulp density and collector, Dow Z-200, in that order, (Appendix Table A) while the most significant variables affecting the recovery are pulp density, R-208 addition and grind, in that order (Appendix Table D). Analysis of the results of the high-grade sample shows that the significant variables affecting the concentrate grade are pulp density and grind, in that order, (Appendix Table B), while the pulp density, amount of frother and lime added are significant variables affecting the copper recovery. (Appendix Table E)

Since we are considering the rougher step in the flotation study, we are more concerned with recovery of copper than with concentrate grade. However, in our study there is no significant correlation between the operating variables and copper recovery. In the overall-copper system, the most significant variables are pulp density, frother addition and copper grade of feed but these account for less than 50% of the variation in the copper recovery. The range of recoveries obtained in the 24 tests under different conditions was from 95.4 to 98.6% while the range of recoveries obtained in replicate tests was from 97.3 to 98.6%.

The results of this work suggest that the most efficient operation of the Opemiska rougher-flotation circuit would depend on grinding the feed to as near 80% minus 200 mesh as the process economics will allow and operating the circuit at as low a pulp density as the flotation-cell capacity will allow. The amounts of reagents used can vary within the limits investigated in this series of tests without significantly affecting the flotation results. The indications are that under these conditions the rougher circuit would handle Opemiska ore ranging from 1.4 to 6.1% Cu.

TABLE 2

Regression Input Data And Responses

INDEPENDENT VARIABLES									RESPONSES				
Run No	X_1 Grind (%-200)	X_2 Pulp Solids (%)	X_3 Lime (lb/ton)	X_4 Z-200 (lb/ton)	X_5 R-208 (lb/ton)	X_6 TEB Frother (lb/ton)	X_7 Air cfm	X_0 Ore Cu (%)	Copper Grade Measured (%)	Copper Recovery Measured (%)	Copper Grade Predicted (%)	Copper Recovery Predicted (%)	Run No
	Low Grade Copper System								Low Grade Cu System		Overall Cu System		
1	63	19	0.6	0.075	0.015	0.06	0.11	1.44	19.1	95.5	17.5	96.2	1
2	80	19	0.6	0.15	0.015	0.06	0.14	1.44	17.9	96.8	18.8	96.2	2
3	63	32	0.6	0.075	0.045	0.12	0.13	1.44	14.2	97.6	14.6	97.7	3
4	80	33	0.6	0.15	0.045	0.06	0.23	1.44	13.7	98.1	15.6	97.3	4
5	63	19	1.2	0.15	0.045	0.12	0.25	1.44	16.4	96.8	17.5	96.7	5
6	80	19	1.2	0.075	0.045	0.12	0.25	1.44	20.7	97.5	18.8	96.7	6
7	63	32	1.2	0.15	0.015	0.06	0.23	1.44	16.2	96.9	14.6	97.2	7
8	80	32	1.2	0.075	0.015	0.12	0.13	1.44	17.2	97.5	16.0	97.7	8
9*	73	25.4	0.9	0.10	0.03	0.08	0.18	1.44	17.4	97.5	16.9	96.9	9
10*	73	25.4	0.9	0.10	0.03	0.08	0.18	1.44	14.9	97.6	16.9	96.9	10
11*	73	25.4	0.9	0.10	0.03	0.08	0.18	1.44	17.0	97.5	16.9	96.9	11
12*	73	25.4	0.9	0.10	0.03	0.08	0.18	1.44	16.6	97.3	16.9	96.9	12
13	80	32	1.2	0.15	0.045	0.12	0.23	1.44	14.5	97.5	16.0	97.7	13
14	63	32	1.2	0.075	0.045	0.12	0.23	1.44	15.0	97.0	14.6	97.7	14
15	80	19	1.2	0.15	0.015	0.06	0.23	1.44	17.8	96.1	18.8	96.2	15
16	63	19	1.2	0.075	0.015	0.12	0.13	1.44	10.5	96.0	17.5	96.7	16
17	80	33	0.6	0.075	0.015	0.06	0.13	1.44	17.9	96.7	15.8	97.3	17
18	63	32	0.6	0.15	0.015	0.06	0.13	1.44	14.0	97.1	14.6	97.2	18
19	80	19	0.6	0.075	0.045	0.12	0.13	1.44	19.1	96.0	18.8	96.7	19
20	63	19	0.6	0.15	0.045	0.06	0.23	1.44	16.5	96.1	17.5	96.2	20
	High Grade Copper System								High Grade Cu System				
21	62.6	19	0.6	0.075	0.015	0.06	0.13	6.31	23.5	95.4	22.1	96.8	21
22	80.6	19	0.6	0.15	0.015	0.06	0.13	6.31	23.8	96.5	23.5	96.8	22
23	62.6	33	0.6	0.075	0.045	0.12	0.13	6.31	18.8	98.4	19.0	98.3	23
24	80.6	33	0.6	0.15	0.045	0.06	0.24	6.31	20.8	96.7	20.4	97.8	24
25	62.6	19	1.2	0.15	0.045	0.12	0.24	6.31	21.1	97.3	22.1	97.3	25
26	80.6	19	1.2	0.075	0.045	0.12	0.24	6.31	24.9	97.5	23.5	97.3	26
27	62.6	33	1.2	0.15	0.015	0.06	0.24	6.31	19.9	97.9	19.0	97.8	27
28	80.6	33	1.2	0.075	0.015	0.12	0.13	6.31	21.4	98.4	20.4	98.3	28
29*	71.6	26	0.9	0.10	0.03	0.08	0.185	6.31	20.8	98.5	21.2	97.5	29
30*	71.6	26	0.9	0.10	0.03	0.08	0.185	6.31	19.4	98.6	21.2	97.5	30
31*	71.6	26	0.9	0.10	0.03	0.08	0.185	6.31	20.5	97.6	21.2	97.5	31
32*	71.6	26	0.9	0.10	0.03	0.08	0.189	6.31	20.0	97.4	21.2	97.5	32

* Replicate Runs

CONCLUSIONS

Only one meaningful mathematical relationship between the variables and the responses was developed. This model can be used to estimate the copper content of the concentrate with a confidence of 95% when the ore grade is between 1.44 and 6.31% and the other variables are within the range investigated. The resulting mathematical model is:

Grade of concentrate % Cu = 15.21 + 0.081 (% minus 200 mesh in feed) -0.22 (% solids in pulp) + 0.95 (% copper in feed).

Reagent additions within the ranges investigated had no significant effect on the concentrate grade.

ACKNOWLEDGEMENTS

The contribution of other members of the Extraction Metallurgy Division staff to this work is gratefully acknowledged. The regression analysis was calculated by Mr. F.J. Kelly of the Hydrometallurgy Section. The chemical analysis was done in the Chemical Analysis Section under the direction of Mr. J.C. Ingles, and principally by Mr. R.J. Guest. Acknowledgement is also made of the work of Mr. L.E. Carter who, as laboratory technician, assisted with the experimental work.

REFERENCES

1. C.E. Makepiece, "A Review of Statistical Design of Experiments in Metallurgical Engineering", CIM Bulletin, 60, No. 666, 1173-1177 (1967)

2. F.J. Kelly, W.R. Honeywell and W.A. Gow, "The Regression of Ore Treatment Test Results. Part 1: The Development and Assessment of First-order Regression Equations", Mines Branch. Investigation Report IR 68-9, Department of Energy Mines and Resources, Ottawa, (1968).

3. O.L. Davies, (Editor), "The Design and Analysis of Industrial Experiments", Second Edition, Hafner, New York (1963).

APPENDIX

TABLE A

Regression Results for Low-Grade Copper Ore: Concentrate Grade

(a) Empirical Model

Conc Grade (% Cu) = $24.86 - 0.22X_8 - 23.33X_4$

Response Mean = 16.7% Deviation in Response = $\pm$ 1.9

Note: Included terms are significant and variation in the response due to each is greater than that due to experimental error at a confidence level of 75%.

(b) Standard Error of Estimate For Response Mean

Source	Confidence Level of 75%	
	Standard Error	Interval
Empirical Model	$\pm$ 1.18	$\pm$ 1.41
System or experimental Error	$\pm$ 1.10	$\pm$ 1.31

(c) Variation In Response Due To Significant Terms

Variables	Pct. of Variation	Coefficient
X2	47.9	- 0.2173539
X4	18.0	- 23.32799
Total	65.9	
Constant Term in Empirical Model		24.85599

(d) Variance Tests

Source	Deg. Freedom	F-Calculated
Regression Variation / Residual Variation	2, 17	4.70*
Lack-fit Variation / Exp. Error Variation	14, 3	1.19

*Indicates Statistical Significance at a Confidence Level of 95%

(e) Overall Variation In Response

Source	Amount (%)
Significant Independent Variables	65.9
Unexplained Sources or Lack-of-fit	28.9
System or Experimental Error	5.2
Total	100.0

Note: A detailed explanation of these tables is given elsewhere (2).

APPENDIX

TABLE B

Regression Results for High-Grade Copper Ore: Concentrate Grade

(a) Empirical Model

$$\text{Conc Grade (\% Cu)} = 19.44 + 0.106\, X_1 - 0.22 X_2$$

Response Mean = 21.2 % Deviation in Response = ± 1.9

Note: Included terms are significant and variation in the response due to each is greater than that due to experimental error at a confidence level of 75%.

(b) Standard Error of Estimate For Response Mean

Source	Confidence Level of 75%	
	Standard Error	Interval
Empirical Model	± 1.17	± 1.44
System or experimental Error	± 0.61	± 0.75

(c) Variation In Response Due To Significant Terms

Variables	Pct. of Variation	Coefficient
X1	18.7	0.1055556
X2	49.6	-0.2214286
Total	68.3	
Constant Term in Empirical Model		19.441032

(d) Variance Tests

Source	Deg. Freedom	F-Calculated
Regression Variation / Residual Variation	2, 9	9.60 *
Lack-fit Variation / Exp. Error Variation	6, 3	4.94

*Indicates Statistical Significance at a Confidence Level of 95%

(e) Overall Variation In Response

Source	Amount (%)
Significant Independent Variables	68.3
Unexplained Sources or Lack-of-fit	28.8
System or Experimental Error	2.9
Total	100.0

APPENDIX

TABLE C

Regression Results for Overall Copper System: Concentrate Grade

(a) Empirical Model

Conc Grade (% Cu) = $15.21 + 0.081X_1 - 0.22X_2 + 0.95X_8$

Response Mean = 18.4%   Deviation in Response = $\pm$ 2.9%

Note: Included terms are significant and variation in the response due to each is greater than that due to experimental error at a confidence level of 95%.

(b) Standard Error of Estimate For Response Mean

Source	Confidence Level of 95%	
	Standard Error	Interval
Empirical Model	$\pm$ 1.25	$\pm$ 2.56
System or experimental Error	$\pm$ 0.89	$\pm$ 1.82

(c) Variation In Response Due To Significant Terms

Variables	Pct. of Variation	Coefficient
X1	4.2	0.08062344
X2	19.1	-0.2201742
X8	59.9	0.9487196
Total	83.2	
Constant Term in Empirical Model		15.207136

(d) Variance Tests

Source	Deg. Freedom	F-Calculated
Regression Variation / Residual Variation	3, 28	17.29*
Lack-fit Variation / Exp. Error Variation	22, 6	2.25

*Indicates Statistical Significance at a Confidence Level of 95%

(e) Overall Variation In Response

Source	Amount (%)
Significant Independent Variables	83.2
Unexplained Sources or Lack-of-fit	15.0
System or Experimental Error	1.8
Total	100.0

APPENDIX

TABLE D

Regression Results for Low-Grade Copper Ore: Copper Recovery

(a) Empirical Model

$$\text{Recovery (\% Cu)} = 92.85 + 0.025 X_1 + 0.071 X_2 + 16.7 X_5$$

Response Mean = 96.96% Deviation in Response = ± 0.70

Note: Included terms are significant and variation in the response due to each is greater than that due to experimental error at a confidence level of 75%.

(b) Standard Error of Estimate For Response Mean

Source	Confidence Level of 75%	
	Standard Error	Interval
Empirical Model	± 0.50	± 0.60
System or experimental Error	± 0.13	± 0.15

(c) Variation In Response Due To Significant Terms

Variables	Pct. of Variation	Coefficient
X1	8.2	0.02510003
X2	38.0	0.07058042
X5	10.8	16.666667
Total	57.0	
Constant Term in Empirical Model		92.84737

(d) Variance Tests

Source	Deg. Freedom	F-Calculated
Regression Variation / Residual Variation	3, 16	7.07 *
Lack-fit Variation / Exp. Error Variation	13, 3	19.17 *

*Indicates Statistical Significance at a Confidence Level of 95%

(e) Overall Variation In Response

Source	Amount (%)
Significant Independent Variables	57.0
Unexplained Sources or Lack-of-fit	42.5
System or Experimental Error	0.5
Total	100.0

APPENDIX

TABLE E

Regression Results for High-Grade Copper Ore: Copper Recovery

(a) Empirical Model

Recovery (% Cu) = $93.27 + 0.084X_2 + 1.08X_3 + 12.56\,X_6$

Response Mean = 97.52% Deviation in Response = ± 0.96%

Note: Included terms are significant and variation in the response due to each is greater than that due to experimental error at a confidence level of 75%.

(b) Standard Error of Estimate For Response Mean

Source	Confidence Level of 75%	
	Standard Error	Interval
Empirical Model	± 0.74	± 0.91
System or experimental Error	± 0.61	± 0.76

(c) Variation In Response Due To Significant Terms

Variables	Pct. of Variation	Coefficient
X2	27.3	0.08392857
X3	13.2	1.080392
X6	16.5	12.55882
Total	57.0	
Constant Term in Empirical Model		93.273739

(d) Variance Tests

Source	Deg. Freedom	F-Calculated
Regression Variation / Residual Variation	3, 8	3.54
Lack-fit Variation / Exp. Error Variation	5, 3	1.71

*Indicates Statistical Significance at a Confidence Level of 95%

(e) Overall Variation In Response

Source	Amount (%)
Significant Independent Variables	57.0
Unexplained Sources or Lack-of-fit	31.8
System or Experimental Error	11.2
Total	100.0

APPENDIX

TABLE F

Regression Results for Overall Copper System: Copper Recovery

(a) Empirical Model

Recovery (% Cu) = $94.08 + 0.076\ X_2 + 8.74\ X_6 + 0.11X_8$

Response Mean = 97.17% Deviation in Response = $\pm$ 0.84%

Note: Included terms are significant and variation in the response due to each is greater than that due to experimental error at a confidence level of 95%.

(b) Standard Error of Estimate For Response Mean

Source	Confidence Level of 95%	
	Standard Error	Interval
Empirical Model	$\pm$ 0.64	$\pm$ 1.31
System or experimental Error	$\pm$ 0.44	$\pm$ 0.91

(c) Variation In Response Due To Significant Terms

Variables	Pct. of Variation	Coefficient
X2	29.7	0.07614061
X6	7.4	8.738892
X8	10.5	0.1111580
Total	47.6	
Constant Term in Empirical Model		94.078233

(d) Variance Tests

Source	Deg. Freedom	F-Calculated
Regression Variation / Residual Variation	3, 28	4.23 *
Lack-fit Variation / Exp. Error Variation	22, 6	2.38

*Indicates Statistical Significance at a Confidence Level of 95%

(e) Overall Variation In Response

Source	Amount (%)
Significant Independent Variables	47.6
Unexplained Sources or Lack-of-fit	47.0
System or Experimental Error	5.4
Total	100.0