Factorial design and statistical analysis of Flotation Experiments

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INTRODUCTION :

'Trial and error' and 'one-variable-at-a-time' type of conventional experiments are inadequate to study a complex process like flotation, with results dependent on a number of interacting variables. Recent studies (1 – 3) suggest that factorial experiments, evolved by Sir Ronald Fisher in late 1920's, offer several advantages in flotation investigations involving quantitative factors. However, factorial design did not become popular in flotation studies, and so far it has not been tried as an alternative to the 'trial and error' method. Therefore, the study of graphite fines flotation is presented in this paper to illustrate the advantages of factorial design over the conventional 'trial and error' approach.

Experimental design :

A 'trial and error' experiment involving kerosene, sodium silicate and sodium carbonate (9.5 pH), has been designed to find the effective reagent combination to float graphite fines (Table 1).

It may be observed that in this 'trial and error' experiment the effectiveness of kerosene will be determined by comparing the results of the trials 1 and 2. If the results of the latter are better than those of the former, kerosene will be considered effective and hence, it will be used in the subsequent trials. If, on the other hand, the results of trial 1 are found to be as good as or better than those of trial 2, then kerosene will be considered ineffective and, therefore, it will be discontinued in the subsequent trials. By this 'trial and error' method, the effective reagent combination to float graphite fines will be determined. However, this 'trial and error' experimental design suffers from the following drawbacks :

Table—1 : 'Trial and error' Experimental Design :

Trial No.	Trial conditions
1	Pine oil
2	Pine oil and Kerosene
3	Pine oil, kerosene and sodium silicate (if kerosene is found effective)
	Or Pine oil and Sodium Silicate (if kerosene is found ineffective)
4	Pine oil, kerosene, sod. silicate and sod. carbonate (9.5 pH) (if both kerosene and sodium silicate are found effective)
	Or Pine oil, kerosene and sodium carbonate (9.5 pH) (if kerosene is effective and sod. silicate is ineffective)
	Or Pine oil, sod.silicate and sod. carbonate (9.5 pH) (if kerosene is ineffective and sod. silicate is effective)
	Or Pine oil and sodium carbonate (9.5 pH) (if both kerosene and sodium silicate are found ineffective)

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1. Of the eight trial conditions only four will be selected for the tests and, hence, the experiment fails to generate complete data.
2. The effectiveness of each reagent will be determined only under one set of conditions. Therefore, to ensure the reliability of the inferences, duplicate or triplicate tests have to be conducted. These additional tests will offset the economy of the original experimental design. Further, the duplicate and triplicate tests conducted under identical trial conditions are comparable to the repetitions of the meaurements with the same yard-stick to ensure the reliability.
3. The treatment combination for the third trial can be decided only after evaluating the effectiveness of kerosene. Similarly, the conditions for the fourth trial can be decided only after assessing the effectiveness of sodium silicate. It is obvious that the investigator has to waste much time waiting for the chemical analysis report.

The 'trial and error' experimental design can be transformed into a factorial experimental design by including eight trials with all the possible conditions as shown in the following table :

Table – 2 : 2^3 Factorial Experimental Design :

Trial No.	Trial symbol	Treatment combination
1	$a_0\, b_0\, c_0$	Pine oil
2	$a_+\, b_0\, c_0$	Pine oil and kerosene
3	$a_0\, b_+\, c_0$	Pine oil and sodium silicate
4	$a_+\, b_+\, c_0$	Pine oil, kerosene and sodium sillcate
5	$a_0\, b_0\, c_+$	Pine oil and sodium carbonate (9.5 pH)
6	$a_+\, b_0\, c_+$	Pine oil, kerosene and sodium carbonate (9.5 pH)
7	$a_0\, b_+\, c_+$	Pine oil, sodium silicate and sodium carbonate (9.5 pH)
8	$a_+ b_+\, c_+$	Pine oil, kerosene, sodium silicate and sodium carbonate (9.5 pH)

It may be noticed that in both types of experimental design each reagent (qualitative factor) assumes or is assigned only two values or levels viz, 'reagent not added' ('o') and 'reagent added' ('+'). For the sake of convenience the 'o' levels of kerosene, sodium silicate and sodium carbonate are respectively denoted by a_0, b_0 and c_0; and their '+' levels are represented by a_+, b_+ and c_+. Using these symbols the eight treatment combinations have been formed as shown in Table 2. The trial symbols can also be used to represent the results of the respective flotation trials. The experimental design will be referred to as 2^3 factorial experimental design because it involves three 2-level factors. It is needless to mention that the expression 2^3 indicates that the experimental design includes eight trials. This (eight trials of the 2^3) experiment can be regrouped into two 2^2 factorial experimental designs as follows :

$a_0\, b_0\, c_0$, $a_+ b_0\, c_0$, $a_0\, b_+ c_0$ and $a_+ b_+ c_0$ – 2^2 Factorial design at neutral pH;

$a_0\, b_0\, c_+$, $a_+ b_0\, c_+$, $a_0\, b_+ c_+$ and $a_+ b_+ c_+$ – 2^2 Factorial design at 9.5 pH.

The following are the principal advantages of the factorial design :

1. The experimental design includes eight trials with all the possible treatment combinations and, hence, complete data will be obtained.
2. The four trials of the 2^2 factorial design can be visualized as the four corners of a square, with its four sides defining the boundaries of the experimental area. Similarly, the eight trials of the 2^3 factorial experimental design can be represented as the eight corners of a cube, whose six faces define the experimental space. It is evident that the factorial experiments can generate large data from minimum number of trials, and that the data will be amenable for mathematical analysis.
3. There is an implied replication in the factorial experimental design and, therefore, duplicate and triplicate tests need not be conducted to verify the results of the original trials.

4. Since the conditions for all the eight trials are decided in advance, the investigator can complete the flotation tests without loosing time waiting for the chemical analysis report. Further, the eight flotation tests can be conducted in a random order to eliminate human bias and chance experimental errors. After completing the tests, the chemical analysis of the flotation products can also be carried out in a random order.

In view of the above mentioned advantages, it is proposed to conduct the flotation tests as per the 2^3 factorial experimental design.

Results :

The ore selected for the present tests comprises graphite with quartz, felspars and altered felspars as the principal gangue minerals. The bluk sample has been crushed and dry ground to a fine size (about 60%—76μm), and the representative samples (of 500 g each) have been floated without further grinding. However, prior to flotation the feed samples have been soaked in water (of neutral pH) in the case of the c_0 trials, and of 9.5 pH in the case of c_+ trials for 30 minutes to ensure proper wetting of the particle surfaces.

Each trial comprises rougher and cleaner flotations consecutively carried out in 2 litre and 4 litre flotation cells respectively. Rougher flotations have been carried out after adding the reagents in the following order : sodium carbonate (in the four c_+ trials adequate doses to raise the pH of water to 9.5), sodum silicate (in the four b_+ trials; 0.5 g/ 500 g), kerosene (in the four a_+ trials; adequate doses in two stages) and pine oil (in all the eight trials; adequate doses in two stages). Except sodium carbonate (to raise the pH of water to 9.5 in the four c_+ trials), further doses of reagents have not been employed for cleaner flotations.

The flotation tests as well as the chemical analysis of the flotation products have been conducted as per the random numbers to minimize the experimental errors. The results of the 2^3 factorial experiments are presented in Table 3. The eight total heads are not identical, but they do not deviate much from their average. This indicates that the experimental errors are negligible, and that the results of the eight trials can be accepted for analysis.

Table 3 : Results of the 2^3 factorial experiment

Trial symbol	Weight per cent			Carbon per cent			Recovery (%)	Total (% C)
	Clean. conc.	Clean tail.	Rough. tail.	Clean. conc.	Clean. tail.	Rough. tail.		
$a_0\ b_0\ c_0$	27.3	35.3	37.3	83.4	48.1	10.7	52.1	43.74
$a_+ b_0\ c_0$	29.3	36.8	33.9	86.3	40.3	11.1	57.6	43.88
$a_0\ b_+ c_0$	34.4	31.2	34.4	84.6	35.2	11.3	66.2	43.97
$a_+ b_+ c_0$	39.8	28.3	31.9	85.4	25.3	9.5	76.9	44.18
$a_0\ b_0\ c_+$	31.1	31.1	37.9	87.5	38.3	12.9	61.8	44.01
$a_+ b_0\ c_+$	39.9	22.8	37.3	80.8	32.3	11.2	73.6	43.78
$a_0\ b_+ c_+$	30.4	31.8	37.8	86.6	43.5	10.4	59.7	44.09
$a_+ b_+ c_+$	44.8	21.0	34.2	82.3	20.5	7·8	84.1	43.84
Average	34.6	29.8	35.6	84.6	35.4	10.6	66.5	43.94

Statistical Analysis :

In the present factorial experiment each reagent has been assigned two discrete values viz. 'reagent not added' ('o') and 'reagent added' ('+'). Consequently, the mathematical analysis has to be limited to the estimation of the effects and interactions of the three reagents (qualitative factors) involed in the experiment. Such an analysis is called the statistical analysis. The effects and the interactions have been estimated as follows :

By subtracting the results of the four 'a_0' ('kerosene not added') trials from those of the corresponding four 'a_+' ('kerosene added') trials, the following four effects of kerosene are obtained and their average is called the Main Effect of Kerosene :

$a_+b_0\,c_0 - a_0\,b_0\,c_0 =$
Effect of kerosene at $b_0\,c_0$ = A1

$a_+b_+c_0 - a_0\,b_+c_0 =$
Effect of kerosene at $b_+\,c_0$ = A2

$a_+\,b_0\,c_+ - a_0\,b_0\,c_+ =$
Effect of kerosene at $b_0\,c_+$ = A3

$a_+b_+c_+ - a_0\,b_+c_+ =$
Effect of kerosene at b_+c_+ = A4

$\frac{1}{4}$ (A1 + A2 + A3 + A4) =
<u>Main Effect of Kerosene = A</u>

The four effects - A1, A2, A3 and A4 - are obtained owing to the implied replications in the factorial experiment. These four estimates give the effects of kerosene under four different conditions ($b_0\,c_0$, b_+c_0 , $b_0\,c_+$ and b_+c_+) and, therefore, their average (A), called the Main Effect, will be regarded as the reliable effect of kerosene.

By subtracting the results of the four 'b_0' trials from those of the corresponding four 'b_+' trials, four estimates of the effects of sodium silicate (i. e., B1, B2, B3 and B4) have been obtained, and from these four estimates the Main Effect (B) of sodium silicate has been calculated. Similarly, the four effects—C1, C2, C3 and C4 — and the Main Effect (C) of sodium carbonate have been computed from the results of the eight trials.

To evaluate the mutual influences of the three reagents in graphite fines flotation process, the interactions have been estimated as follows :

$\frac{1}{2}$ (A2 — A1) = $\frac{1}{2}$ (B2 — B1) = Kerosene — Sodium silicate interaction at c_0 = AB1

$\frac{1}{2}$ (A4 — A3) = $\frac{1}{2}$ (B4 — B3) = Kerosene — Sodium silicate interaction at c_+ = AB2

$\frac{1}{2}$ (AB1 + AB2) = <u>Kerosene — Sodium silicate interaction = AB</u>

$\frac{1}{2}$ (A3 — A1) = $\frac{1}{2}$ (C2 — C1) = Kerosene — Sodium carbonate interaction at b_0 = AC1

$\frac{1}{2}$ (A4 — A2) = $\frac{1}{2}$ (C4 — C3) = Kerosene — Sodium carbonate interaction at b_+ = AC2

$\frac{1}{2}$ (AC1 + AC2) = <u>Kerosene — Sodium carbonate interaction = AC</u>

$\frac{1}{2}$ (B3 − B1) = $\frac{1}{2}$ (C3 — C1) = Sodium silicate — Sodium carbonate interaction at a_0 = BC1

$\frac{1}{2}$ (B4 — B2) = $\frac{1}{2}$ (C4 — C2) = Sodium silicate − Sodium carbonate interaction at a_+ = BC2

$\frac{1}{2}$ (BC1 + BC2) = Sodium silicate − Sodium carbonate interaction = BC

$\frac{1}{2}$ (AB2 — AB1) = $\frac{1}{2}$ (AC2 − AC1) = $\frac{1}{2}$ (BC2 − BC1)
= Kerosene − Sodium silicate — Sodium carbonate interaction = ABC

The interactions − AB, AC and BC − are called the first order interactions, and they will be regarded as the reliable estimates because each is an average of two estimates. The interaction between three factors i.e., ABC, is called the second order interaction. The following relations are useful in evaluating the interactions of the factors :

AB − ABC = AB1 ; AB + ABC = AB2 ; AC − ABC = AC1 ; AC + ABC = AC2 ;
BC — ABC = BC1 ; and BC + ABC = BC2.

The estimates of Main Effects and the Interactions are given in Table 4.

Table—4 : Effects and Interactions :

	Cleaner Concentrate	
	Recovery (%)	Grade (% C)
Main Effect of Kerosene (A)	13.10	- 1.83
Main Effect of Sodium Silicate (B)	10.45	0.23
Main Effect of Sodium Carbonate (C)	6.60	- 0.63
Kerosene — Sodium Silicate Interaction (AB)	4.45	0.08
Kerosene — Sodium Carbonate Interaction (AC)	5.00	−3.68
Sod. Silicate — Sod. Carbonate Interaction (BC)	—6.25	0.08
Kerosene — Sodium Silicate—Sodium Carbonate Interaction (ABC)	1.85	1.13

Pine oil is an implied factor in the experiment, and its effects and interactions can be obtained by reversing the sign of the effects and interactions of kerosene.

Discussion :

In respects of cleaner concentrate recovery, all the three Main Effects — A, B, and C — are positive and appreciable (see Table 4). It can be known that in the absence of kerosene, sodium silicate and sodium carbonate (i.e., trial condition $a_0\ b_0\ c_0$) minimum recovery will be obtained, and that the addition of all the three reagents (i.e., treatment combination $a_+b_+c_+$) will maximize the recovery of graphite fines. These observations may be explained as follows :

1. The cleavage surfaces of graphite flakes are naturally hydrophobic, whereas their perpendicular end faces are hydrophilic. Consequently, in the absence of kerosene (collector) the air bubbles can attach themselves to the graphite fines only at the cleavage surfaces but not at their perpendicular end faces[5] .

2. In the absence of sodium silicate non-selective flocculation (in the form of slime coatings and hetero flocs) conceals the graphite surfaces and, therefore, low recovries result[6] .

3. In the absence of sodium carbonate (9.5pH), the soluble salts present in the water inhibit the flotation of graphite fines.

4. When all the three reagents are added the right conditions for the flotation of graphite fines will be maintained and, hence maximum recovery will be obtained. Kerosene will render the perpendicular end faces of graphite flakes hydrophobic; sodium silicate affects selective dispersion of the silicate slime and thereby minimizes non-selective flocculation ; and sodium carbonate (9.5pH) precipitates the soluble salts present in the water as insolubles.

With regard to the recovery, Main Effect A > Main Effect B > Main Effect C. It can, therefore, be readily observed that kerosene is the most effective factor, and that sodium silicate is a better modifier than sodium carbonate. The interactions AB and AC clearly indicate that in the absence of sodium silicate and/or sodium carbonate, kerosene — the most effective factor will be least effective. The addition of kerosene alone will not improve the recovery of graphite fines to the desired level because non-selective flocculation and the soluble salts in the water continue to inhibit the flotation of graphite fines.

If we consider both recovery and grade of cleaner concentrate, AB is decidedly better than AC. Since sodium silicate is a better modifier than sodium carbonate, and interaction AB is better than AC, trial $a_+b_+c_0$ gives better results than the trial $a_+b_+c_+$. However, the presence of soluble impurities at the air-water or solid-water or both the interfaces continue to pose problems.

The change from $a_+b_+c_0$ to $a_+b_+c_+$ will bring into play the Main Effect C, first order intercations AC and BC, and the second order interaction ABC. In so far as the recovery is concerned the Main Effect C will be principal contributor because the positive interactions AC and ABC will be nullified by then egative interaction BC. Further, the grade of concentrate will be lowered because of AC. However, the change from $a_0\ b_0\ c_0$ to $a_+b_+c_+$ improves the recovery by 32% and the grade by 1.1% (not appreciable.) This clearly indicates that by maintaining proper conditions, the recovery of graphite fines can be improved appreciably without affecting the grade of concentrate.

The addition of sodium silicate and sodium carbonate (9.5 pH) produces a stable suspension which acts as a physical barrier between the air bubbles and graphite fines. This explains the poor recovery resulting from the interaction BC, particularly in the trial $a_0\ b_+c_+$.

In the absence of sodium silicate, kerosene sodium carbonate combination increases the flotation of mixed flocs, which in turn lowers the grade of concentrate. In the presence of sodium silicate, kerosene and sodium carbonate interact to remove into the final concentrate considerable gangue slime (stable suspension) as a constituent of the froth. Thus the grade of cleaner concentrate will be lowered by the interaction AC.

Conclusions :

The 'trial and error' experimental design can be transformed into a factorial experimental design. The 2^3 factorial experimental design offers several advantages over the 'trial and error' approah. The eight trials of the factorial experiment could generate data equivalent to that of 24 flotation trials, and this large data could be condensed into three Main Effects, three first order interactions and one second order interaction. Statistical analysis enable easy identification of the effective factors and interactions that yield good flotation results. The study clearly establishes that flotation reagents are functionally interdependent, and that the interdependencies or interactions of the reagents can be studied by employing factorial design and statistical analysis.

References

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DISCUSSION :

R. Y. Sane,
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Question 1 : Every time the quantity of pine oil, kersone and sod. silicate and carbonate change, the values of A, A2, A3, A4, etc will change. And it will be again a trial and error method. In factorial Design the quantity of reagents also change simultaneously. Please comment.

Author : I agree with the statement that the changes in the quantities of reagents produce changes in the flotation results i.e., effects and interactions of the factors. However, in the present factorial experiment the quantities of the reagents have not been varied.

So far, factorial design has been applied to flotation experiments involving reagents as quantitative variables and therefore, the tests will be conducted by simultaneously varying the quantities of the reagents. The present study illustrates that factorial design can be applied to flotation studies involving reagents as qualitative factors. Each reagent has been assigned '0' (reagent not added) and '+' (reagent added) as its two levels, and the eight trials have been formed by simultaneously varying the '0' and '+' levels of the three reagents (kerosene, sodium silicate and sodium carbonate).

'Trial and error' flotation experimental design (involving qulitative factors) is an incomplete or a partial factorial design. One variable at a time' type of flotation experimental design (involving quantitative factors) is also an incomplete design. Unike these conventional methods, the factorial design includes all the possible treatment combinations and, therefore, can provide adequate data amenable for mathematical analysis.

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Question 2 : You have suggested 8 tests with different combinations of the reagents. What about the factors i.e. feed size, conditioning time and flotation time ?

Author : The 2 factorial experiment, comprising eight trials, has been designed to find the effective reagents combination but not to find optimal conditions for the flotation of graphite. Therefore, feed size and other factors have not been included in the experimental design. Flotation time, expressed as rate of flotation' is normally regarded as a performance parameter.