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RESPONSE SURFACE MODELLING AND OPTIMISATION OF ILMENITE FLOTATION USING A MODIFIED LIGNOSULPHONATE REAGENT

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Abstract. Selective flotation of low-grade ilmenite ores is complicated by the similarity in surface properties between ilmenite and silicate gangue, while reagent performance depends jointly on several interacting process variables. The feed ore contained 6.8 wt.% TiO₂ and consisted predominantly of silicate gangue, confirming its low-grade character and the need for selective flotation. This study presents a response surface methodology (RSM) model for ilmenite flotation using a single modified lignosulphonate collector, LS-O2, obtained by permanganate oxidation. A three-factor, three-level Box-Behnken design was used to relate pulp pH, reagent dosage, and temperature to ilmenite recovery and the TiO₂ grade of the concentrate. Second-order polynomial models for both responses were statistically significant ($p < 0.001$), with no significant lack of fit, and explained 99.6% and 98.4% of the variance in recovery and TiO₂ grade, respectively. Reagent dosage was the dominant factor for both responses, followed by pH, while temperature had a comparatively minor effect. For TiO₂ grade, the linear temperature term and temperature-related interactions were not significant, although the quadratic temperature term remained significant. Joint optimisation using a composite desirability function identified an optimum at pH 7.31, an LS-O2 dosage of 0.331 kg/t, and a temperature of 28°C, predicting 76.6% recovery and a TiO₂ grade of 21.4%. Model predictions were verified at five independent operating points, including

a boundary combination and an extrapolated condition, with deviations not exceeding 2.52%.

Keywords: ilmenite, froth flotation, modified lignosulphonate, response surface methodology, Box-Behnken design, process optimisation

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МОДИФИКАЦИЯЛАНҒАН ЛИГНОСУЛЬФОНАТТЫ РЕАГЕНТТІ ҚОЛДАНУ АРҚЫЛЫ ИЛЬМЕНИТ ФЛОТАЦИЯСЫНЫҢ ЖАУАП БЕТІН МОДЕЛЬДЕУ ЖӘНЕ ОҢТАЙЛАНДЫРУ

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Аннотация. Төмен сұрыпты ильменит кендерін селективті флотациялау ильменит пен силикатты бос жыныстың беттік қасиеттерінің ұқсастығымен күрделенеді, ал реагенттің тиімділігі бірнеше өзара әрекеттесетін процесс айнаымалыларына бірлесіп тәуелді. Бастапқы кен құрамында 6,8 масс.% TiO₂ болды және ол негізінен силикатты бос жыныстан тұрды, бұл оның төмен сұрыпты кенге жататынын және селективті флотацияны қолдану қажеттігін көрсетеді. Бұл зерттеуде перманганатпен тотықтыру арқылы алынған бір ғана модификацияланған лигносульфонат жинағышы LS-O2 қолданылған ильменит флотациясына арналған жауап беті әдіснамасының (RSM) моделі ұсынылған. Пульпаның рН мәні, реагент шығыны және температура мен ильмениттің алынуы және концентраттағы TiO₂ мөлшері арасындағы байланысты анықтау үшін үш факторлы, үш деңгейлі Бокс-Бенкен жоспары қолданылды. Екі жауап үшін де екінші ретті полиномдық модельдер статистикалық тұрғыдан маңызды болды ($p < 0,001$), елеулі сәйкессіздік

байқалмады және сәйкесінше алыну мен TiO_2 мөлшері дисперсиясының 99,6% және 98,4%-ын түсіндірді. Реагент шығыны екі жауап үшін де басым фактор болды, одан кейін рН тұрды, ал температура салыстырмалы түрде аз әсер етті. TiO_2 мөлшері үшін температураның сызықтық мүшесі және температурамен байланысты өзара әрекеттесулер маңызды болмады, дегенмен температураның квадраттық мүшесі маңызды болып қалды. Құрамдас қалаулылық функциясын қолдану арқылы жүргізілген бірлескен оңтайландыру рН 7,31, LS-O2 шығыны 0,331 кг/т және температура 28°C болғанда оңтайлы жағдайды анықтады, бұл кезде алыну 76,6% және TiO_2 мөлшері 21,4% болады деп болжанды. Модель болжамдары шекаралық комбинацияны және экстраполяцияланған жағдайды қоса алғанда, бес тәуелсіз жұмыс нүктесінде тексерілді, бұл ретте ауытқулар 2,52%-дан аспады.

Түйінсөздер: ильменит, көбікті флотация, модификацияланған лигносульфонат, жауап беті әдіснамасы, Бокс-Бенкен жоспары, үдерісті оңтайландыру

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МОДЕЛИРОВАНИЕ ПОВЕРХНОСТИ ОТКЛИКА И ОПТИМИЗАЦИЯ ФЛОТАЦИИ ИЛЬМЕНИТА С ИСПОЛЬЗОВАНИЕМ МОДИФИЦИРОВАННОГО ЛИГНОСУЛЬФОНАТНОГО РЕАГЕНТА

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Аннотация. Селективная флотация низкосортных ильменитовых руд затрудняется сходством поверхностных свойств ильменита и силикатной пустой породы, при этом эффективность реагентов зависит одновременно от нескольких взаимодействующих переменных процесса. Исходная руда содержала 6,8 мас.% TiO_2 и была представлена преимущественно силикатной пустой породой, что подтверждает её низкое содержание ценного компонента и необходимость применения селективной флотации. В настоящем исследовании представлена модель методологии поверхности отклика (RSM) для флотации ильменита с использованием одного модифицированного лигносульфонатного собирателя LS-O2, полученного путем окисления перманганатом. Для установления зависимости извлечения ильменита и содержания TiO_2 в концентрате от рН пульпы, расхода

реагента и температуры использовали трёхфакторный трёхуровневый план Бокса-Бенкена. Полиномиальные модели второго порядка для обоих откликов были статистически значимыми ($p < 0,001$), не демонстрировали значимого отклонения от модели и объясняли 99,6% и 98,4% дисперсии в извлечении и содержании TiO_2 , соответственно. Расход реагента был доминирующим фактором для обоих откликов, за ней следовал pH, тогда как температура оказывала сравнительно незначительное влияние. Для содержания TiO_2 линейный температурный член и температурные взаимодействия не были значимыми, хотя квадратичный температурный член оставался значимым. Совместная оптимизация с использованием составной функции желательности определила оптимум при pH 7,31, расходе LS-O2 0,331 кг/т и температуре 28°C, прогнозируя извлечение 76,6% и содержание TiO_2 21,4%. Прогнозы модели были проверены в пяти независимых рабочих точках, включая граничное сочетание и экстраполированное условие, при этом отклонения не превышали 2,52%.

Ключевые слова: ильменит, пенная флотация, модифицированный лигносульфонат, методология поверхности отклика, план Бокса-Бенкена, оптимизация процесса

Introduction. Ilmenite (FeTiO_3) is the principal industrial titanium-bearing mineral and an important feedstock for the production of TiO_2 pigments and titanium metal (El Khalloufi et al., 2021; Maldybayev et al., 2024). The gradual depletion of readily beneficiated ilmenite resources and the growing reliance on complex, low-grade titanium-bearing ores have increased the need for more efficient mineral-processing technologies (Maldybayev et al., 2024; Liu et al., 2025). Such ores typically contain fine-grained ilmenite associated with quartz, feldspar, chlorite, pyroxene, and other silicate gangue minerals, which complicates selective separation and the production of high-grade titanium concentrates (Mehdilo and Irannajad, 2021; Yang et al., 2021). Froth flotation of these ores is particularly challenging because ilmenite has relatively low natural floatability and a chemically heterogeneous surface. In addition, fine silicate gangue particles may form slime coatings on the ilmenite surface or heteroaggregates with ilmenite particles, thereby reducing collector adsorption and flotation selectivity (Yang et al., 2021; Zhai et al., 2020). Therefore, achieving both high recovery and high concentrate grade requires careful control of the reagent system and operating conditions to ensure selective separation of ilmenite from silicate gangue.

Reagents based on natural polymers, particularly lignosulphonates, are attracting increasing interest as potentially sustainable depressants and auxiliary reagents in mineral flotation. Lignosulphonates are readily available and relatively inexpensive lignin derivatives produced during the sulphite pulping of wood. They contain sulphonate, carboxyl and phenolic hydroxyl groups, which confer an anionic character and promote their interaction with selected mineral surfaces (Vidal et al., 2021; Quiroz et al., 2022; Chique et al., 2023). Their adsorption properties, surface activity, molecular charge, and mineral selectivity can be regulated through chemical modification, including sulphomethylation, as well as through control of molecular weight and

degree of sulphonation. These approaches enable the properties of lignosulphonate-based reagents to be adjusted for specific flotation separations (Vidal et al., 2021; Wu et al., 2025). Oxidative modification of lignosulphonate increases its carboxyl-group content and anionic charge density, thereby altering its adsorption and complexation behaviour towards mineral surfaces (Ganguly et al., 2024). Recent flotation studies have shown that oxidised sodium lignosulphonate can produce stronger and more selective depression than its unmodified precursor in oxide-mineral systems (Yao et al., 2026). The depressing action of this reagent, however, depends on the operating conditions. Ilmenite recovery and concentrate grade are simultaneously influenced by pulp pH, reagent dosage, temperature, and other process variables, which may interact rather than act independently. Therefore, favourable flotation conditions cannot be reliably identified by varying only one variable at a time.

Traditional one-factor-at-a-time optimisation is still widely used in preliminary reagent-development studies. In this approach, each variable is evaluated whilst all other variables are held constant, which does not permit interactions between process variables to be reliably identified or quantified. In multivariable systems, the sequential variation of individual factors can also be labour-intensive and experimentally inefficient and may fail to identify the most favourable combination of operating conditions (Szpisják-Gulyás et al., 2023; Ahmadi et al., 2021). Statistically designed experiments, particularly response surface methodology (RSM) implemented using Box-Behnken or central composite designs, address these limitations by varying several factors systematically within a structured experimental matrix (Arancibia-Bravo et al., 2022; Elganidi et al., 2022; Durga Prasad and Meikap, 2025). The resulting data are typically fitted to a second-order polynomial model, enabling the estimation of linear and quadratic effects as well as pairwise interactions, while requiring substantially fewer experimental runs than a corresponding full three-level factorial design.

Despite growing interest in polymer-based flotation reagents, the application of lignosulphonates for the selective flotation of ilmenite remains understudied. In recent studies of organic collectors and polymeric depressants for ilmenite, reagent dosage and pulp pH have typically been evaluated in separate sets of experiments, whilst interactions between process variables have rarely been quantified (Chen et al., 2024; Fan et al., 2024; Wang et al., 2024). To the best of our knowledge, no published study has reported a systematic multivariable optimisation of ilmenite flotation using lignosulphonates. As a result, the combined effects of pH, reagent dosage, and conditioning temperature remain largely unclear. Accordingly, an experimental and modelling study is needed to investigate the flotation process as a multivariable system and quantify the effects of these operating variables on ilmenite recovery and concentrate grade.

This study addresses this research gap by applying a single lignosulphonate modified by permanganate oxidation to a representative ilmenite ore. Using a three-factor Box-Behnken design, response-surface models are developed for ilmenite recovery and the TiO_2 grade of the concentrate as functions of pulp pH, reagent dosage, and temperature.

The specific objectives of this study are to construct and validate second-order regression models for ilmenite recovery and the TiO_2 grade of the concentrate, identify

statistically significant main effects and interactions using ANOVA, determine operating conditions that jointly optimise both responses using a composite desirability function, and assess the predictive performance of the models at independent validation points, including a boundary condition and an extrapolated reagent dosage beyond the design range.

Materials and Methods. *Ore sample and reagents.* The ilmenite ore was crushed and dry-ground to a target D_{85} of 70-80 μm . Its chemical composition was determined by ICP-OES and cross-checked by XRF, while the particle-size distribution was measured by laser diffraction. A modified lignosulphonate, LS-O2, prepared by oxidising technical-grade lignosulphonate with permanganate (0.02M KMnO_4 , pH 5, 65°C), was used as the collector throughout the flotation tests. Pine oil was used as the frother at a fixed dosage of 50 g/t. No other flotation reagents were used; HCl and NaOH were added solely to adjust the pulp pH.

Flotation testing procedure. Flotation tests were carried out in a laboratory mechanical flotation cell with a working volume of 1 dm^3 at a fixed impeller speed and airflow rate, while the pulp temperature was controlled using a thermostatically controlled water jacket. For each run, a fresh portion of ground ore was mixed with water to prepare a pulp containing 20 wt.% solids, and the pulp pH was adjusted to the specified level using dilute HCl or NaOH under agitation. LS-O2 was then added at the prescribed dosage and conditioned with the pulp for 3 min, followed by the addition of pine oil as the frother at a dosage of 50 g/t and a further 1 min of conditioning. Air was subsequently introduced, and the froth product was collected manually for 5 min. The concentrate and tailings were filtered, dried, weighed, and assayed for TiO_2 . Ilmenite recovery was calculated from the product masses and TiO_2 assays, while the TiO_2 grade of the concentrate was determined directly from the analytical results, as described below. Pulp pH, LS-O2 dosage, and temperature were varied according to the experimental design (Section “*Design of experiments*”), while all other conditions were held constant across runs.

Design of experiments. Process variables were investigated using a three-factor, three-level Box-Behnken design (BBD), which enabled the estimation of a full second-order response-surface model, including two-factor interactions, with fewer runs than a full factorial design. The three factors were pulp pH (A, 6.5-8.5), LS-O2 dosage (B, 0.15-0.45 kg/t), and pulp temperature (C, 20-40°C); all other operating conditions were kept constant, as described in the section “*Flotation testing procedure*”. The experimental design comprised 15 runs, including three replicate runs at the centre point, which were used to estimate pure experimental error and perform a lack-of-fit test. The runs were conducted in randomised order.

Ilmenite recovery, expressed on a TiO_2 basis, η (%), was calculated using the following mass-balance equation:

$$\eta = \frac{C \cdot c}{F \cdot f} \times 100$$

where C is the dry mass of the concentrate;

c is the TiO_2 grade of the concentrate;

F is the dry mass of the feed;

and f is the TiO_2 grade of the feed.

The TiO_2 grade of the concentrate, c (%), was used as the second response variable.

Model development and statistical analysis. For each response, a second-order polynomial model was fitted using ordinary least-squares regression at the coded factor levels (-1, 0, and +1):

$$Y = \beta_0 + \sum_{i=1}^3 \beta_i X_i + \sum_{i=1}^3 \beta_{ii} X_i^2 + \sum_{i < j} \beta_{ij} X_i X_j$$

where Y is the predicted response;

β_0 is the intercept;

β_i , β_{ii} , and β_{ij} are the coefficients of the linear, quadratic, and two-factor interaction terms, respectively;

X_i and X_j are the coded process variables;

and $i, j = 1, 2, 3$.

Model significance and adequacy were assessed by analysis of variance (ANOVA) at the 95% confidence level. The model sum of squares was partitioned into linear, quadratic, and interaction contributions, which were evaluated using F-tests and the corresponding p-values. Replicate runs at the centre point were used to estimate pure experimental error and perform a lack-of-fit test. Model quality was further characterised using the coefficient of determination (R^2), adjusted R^2 , and the coefficient of variation. All regression fitting and ANOVA were performed in Python using the statsmodels package.

Results and Discussion. *Characterisation of the feed ore.* The chemical composition of the ilmenite ore used in this study is shown in Figure 1a. The ore consists predominantly of silicate gangue, with SiO_2 accounting for 71.5 wt.%, while the target oxide, TiO_2 , is present at only 6.8 wt.%. The total iron oxide content ($\text{Fe}_2\text{O}_3 + \text{FeO}$) is 14.9 wt.%, which is consistent with the presence of ilmenite (FeTiO_3) and associated iron-bearing phases, while the moderate Al_2O_3 content (4.3 wt.%) points to an aluminosilicate contribution to the gangue assemblage. Overall, the composition is characteristic of a low-grade, silicate-rich ilmenite ore, for which selective flotation reagents are required to achieve an economically meaningful upgrade.

Particle-size analysis of the ground feed (Figure 1b) shows a unimodal, well-defined distribution, with the maximum of the volume-frequency curve located at 50-60 μm and negligible over-grinding, as indicated by the smooth, single-peaked frequency curve. The cumulative undersize curve reaches 85% at approximately 74 μm ($D_{85} \approx 74 \mu\text{m}$), confirming that the target grind was achieved and that the feed is suitable for flotation without excessive fines generation. Such fines could otherwise impair selectivity through non-selective slime coating.

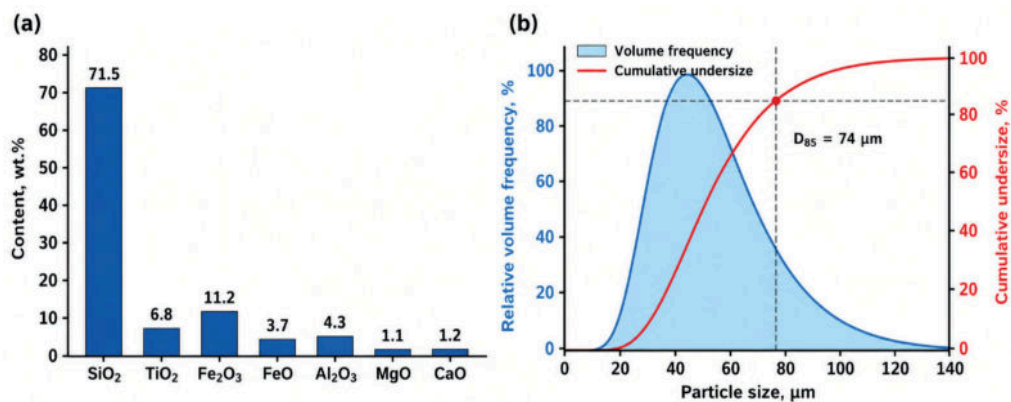


Figure 1. (a) Oxide composition of the feed ilmenite ore; (b) particle size distribution of the ground feed, showing the cumulative undersize curve and D_{85} .

Experimental design and response data. A three-factor, three-level Box-Behnken design (BBD) was used to relate the flotation response of the ilmenite-quartz system to pH (A), LS-O2 dosage (B), and pulp temperature (C). The factor ranges shown in Table 1 were selected to cover the region of favourable flotation performance identified in preliminary single-factor tests, while allowing the combined effects of the three factors to be evaluated.

Table 1. Factors and levels used in the Box-Behnken design.

Factor	Symbol	Low (-1)	Centre (0)	High (+1)
pH of pulp	A	6.5	7.5	8.5
LS-O2 dosage, kg/t	B	0.15	0.30	0.45
Temperature, °C	C	20	30	40

The experimental design comprised 15 runs, including three replicate runs at the centre point (runs 13-15), which were used to estimate pure experimental error and perform a lack-of-fit test. The resulting ilmenite recovery (η) and TiO₂ grade of the concentrate are presented in Table 2.

Table 2. Box-Behnken design matrix and calculated responses.

Run	pH	LS-O2 dosage, kg/t	T, °C	Recovery, %	TiO ₂ , %
1	6.5	0.15	30	70.54	18.32
2	8.5	0.15	30	70.27	18.73
3	6.5	0.45	30	73.22	20.24
4	8.5	0.45	30	71.54	18.98
5	6.5	0.30	20	74.47	20.77
6	8.5	0.30	20	72.41	19.99
7	6.5	0.30	40	73.14	20.13
8	8.5	0.30	40	72.64	19.88
9	7.5	0.15	20	70.99	18.94

Run	pH	LS-O ₂ dosage, kg/t	T, °C	Recovery, %	TiO ₂ , %
10	7.5	0.45	20	73.86	20.09
11	7.5	0.15	40	71.41	18.97
12	7.5	0.45	40	72.58	20.35
13 (centre)	7.5	0.30	30	76.38	21.29
14 (centre)	7.5	0.30	30	76.24	21.14
15 (centre)	7.5	0.30	30	76.82	21.48

Recovery ranged from 70.3% to 76.8%, whilst the TiO₂ grade ranged from 18.3% to 21.5%. Both responses were consistently higher at or near the centre of the design space, providing an initial indication that the optimum was located within the investigated factor ranges rather than at their boundaries.

Development and statistical adequacy of the regression models. Second-order polynomial models were fitted to both responses using least-squares regression. In terms of the coded variables (A, B, C- [-1, 1]), the fitted equations were:

$$\eta (\%) = 76.48 - 0.564A + 0.999B - 0.245C - 2.066A^2 - 3.021B^2 - 1.249C^2 - 0.353AB + 0.390AC - 0.425BC$$

$$TiO_2 (\%) = 21.30 - 0.235A + 0.588B - 0.058C - 0.815A^2 - 1.420B^2 - 0.295C^2 - 0.418AB + 0.133AC + 0.058BC$$

Analysis of variance (Tables 3 and 4) confirmed that both models were statistically significant at the 95% confidence level (η : F=144.7, p<0.0001; TiO₂: F=34.8, p=0.0006).

Table 3. ANOVA for the recovery (η) model.

Source	SS	df	MS	F-value	p-value
Model	61.951	9	6.883	144.66	< 0.0001
Linear (A, B, C)	11.003	3	3.668	77.07	0.0001
Square (A ² , B ² , C ²)	49.121	3	16.374	344.09	< 0.0001
Interaction (AB, AC, BC)	1.828	3	0.609	12.80	0.0088
Residual	0.238	5	0.048	-	-
Lack of fit	0.055	3	0.018	0.20	0.8897 (ns)
Pure error	0.183	2	0.092	-	-
Total (corrected)	62.189	14	-	-	-

Table 4. ANOVA for the TiO₂ grade model.

Source	SS	df	MS	F-value	p-value
Model	13.381	9	1.487	34.82	0.0006
Linear (A, B, C)	3.229	3	1.076	25.21	0.0019
Square (A ² , B ² , C ²)	9.370	3	3.123	73.14	0.0001
Interaction (AB, AC, BC)	0.781	3	0.260	6.09	0.0401
Residual	0.214	5	0.043	-	-
Lack of fit	0.155	3	0.052	1.78	0.3788 (ns)
Pure error	0.058	2	0.029	-	-
Total (corrected)	13.594	14	-	-	-

For both responses, the quadratic terms accounted for the largest share of the model sum of squares (SS) – 79% for η and 70% for the TiO_2 grade. This indicates pronounced curvature and the presence of an interior optimum within the investigated design space, rather than a monotonic trend towards its boundaries. The lack-of-fit test was not statistically significant for either model (η : $p=0.890$; TiO_2 grade: $p=0.379$), supporting the adequacy of the quadratic models within the investigated factor ranges.

The coefficients of determination for both models were high, and the low coefficients of variation indicated good experimental precision. Analysis of the significance of individual terms (Table 5) showed that all linear, quadratic, and interaction terms were significant for recovery ($p<0.05$). For the TiO_2 grade, however, the linear temperature term (C) and the interaction terms AC and BC were not statistically significant ($p>0.25$), whereas the quadratic temperature term (C^2) remained significant.

Table 5. Regression coefficients of the response-surface models in coded variables.

Term	Coef. (η)	p-value (η)	Coef. (TiO_2 grade)	p-value (TiO_2 grade)
Intercept	76.480	< 0.0001	21.303	< 0.0001
A - pH	-0.564	0.0008 ***	-0.235	0.0236 **
B - Dosage	0.999	< 0.0001 ***	0.588	0.0005 ***
C - Temperature	-0.245	0.0246 **	-0.058	0.4669 ns
A^2	-2.066	< 0.0001 ***	-0.815	0.0006 ***
B^2	-3.021	< 0.0001 ***	-1.420	< 0.0001 ***
C^2	-1.249	0.0001 ***	-0.295	0.0405 **
AB	-0.353	0.0232 **	-0.418	0.0099 ***
AC	0.390	0.0159 **	0.133	0.2559 ns
BC	-0.425	0.0114 **	0.058	0.6019 ns

Significance: *** $p<0.01$; ** $p<0.05$; ns - not significant.

The relative importance of the model terms is illustrated by the Pareto chart of standardised effects (Figure 2).

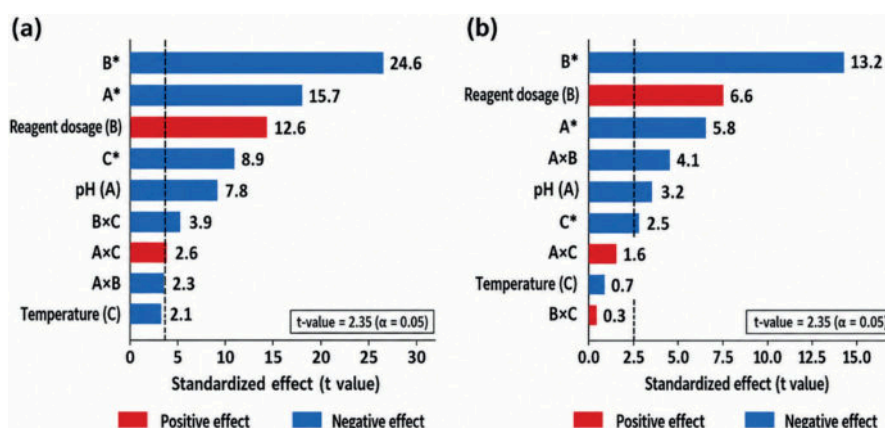


Figure 2. Pareto charts of standardised effects for (a) ilmenite recovery and (b) TiO_2 grade. The dashed line indicates the significance threshold at $\alpha=0.05$.

For both responses, the quadratic dosage term (B^2) had the largest standardised effect, followed by the quadratic pH term (A^2) and the linear dosage term (B). The linear temperature term and most interactions involving temperature ranked among the weaker effects, particularly in the model for TiO_2 grade, in agreement with the ANOVA results. This ordering indicated that reagent dosage was the principal operating variable affecting both ilmenite recovery and concentrate grade. The curved response to dosage may be associated with incomplete surface coverage at low LS-O2 dosages, whereas excessive dosage may promote less selective adsorption on silicate gangue.

Response surface analysis. The three-dimensional response surface and the corresponding contour plot for ilmenite recovery as functions of pH and reagent dosage at the model-predicted optimum temperature are shown in Figures 3 and 4. The surface has a distinct dome shape with a single interior maximum, consistent with the significance of the quadratic terms discussed above. Recovery decreases more steeply along the dosage axis than along the pH axis, which agrees with the larger absolute value of the B^2 coefficient relative to A^2 (Table 5) and with the Pareto ranking shown in Figure 2a. The slight rotation of the contour ellipses in Figure 4 reflects the negative AB interaction, indicating that the pH value yielding maximum recovery shifts slightly with reagent dosage rather than remaining fixed.

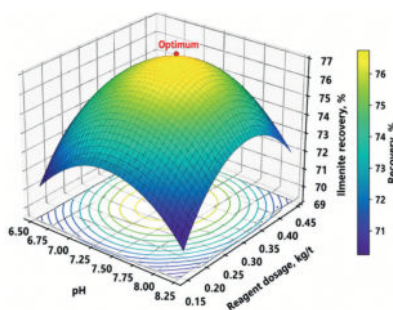


Figure 3. Response surface for ilmenite recovery as a function of pH and reagent dosage at the model-predicted optimum temperature (28°C).

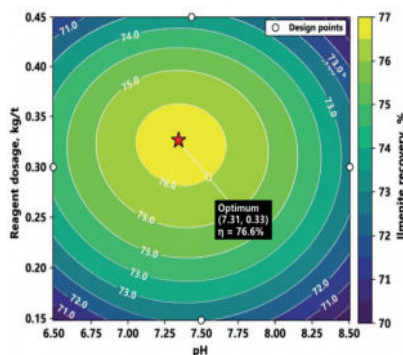


Figure 4. Contour plot of ilmenite recovery as a function of pH and reagent dosage, with design points (circles) and the optimum (star).

The response surface for the TiO_2 grade (Figure 5), evaluated at the centre-point temperature, exhibits a similar dome-shaped dependence on pH and reagent dosage, with the maximum occurring in approximately the same region of the design space as the recovery maximum. The linear temperature term and its interactions with pH and reagent dosage were not statistically significant, whereas the quadratic temperature term remained significant, indicating a weak nonlinear effect of temperature on TiO_2 grade.

The consistency between the two response surfaces is encouraging from a process-design perspective, as it indicates that recovery and concentrate grade are not strongly conflicting objectives within the investigated range. A common operating region can therefore provide favourable values for both responses, supporting the joint optimisation carried out in the following section.

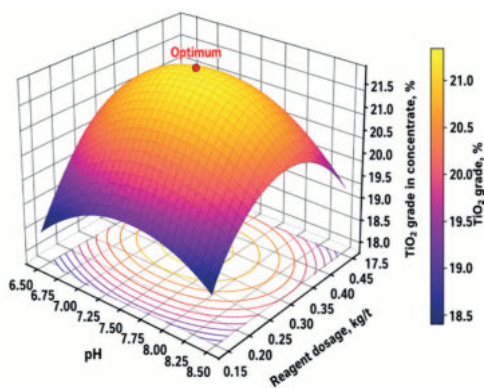


Figure 5. Response surface for the TiO_2 grade of the concentrate as a function of pH and reagent dosage at the centre-point temperature (30°C).

The agreement between the fitted models and the experimental design data is demonstrated by the parity plots in Figure 6. For both responses, the predicted and experimental values lie close to the 1:1 line, with R^2 values of 0.996 and 0.984, respectively, and no obvious systematic deviation from the line.

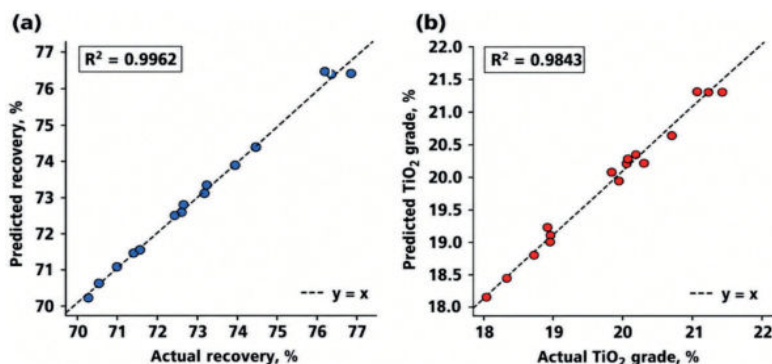


Figure 6. Predicted versus actual values for (a) ilmenite recovery and (b) TiO_2 grade (Box-Behnken design data).

Process optimisation. The two responses were combined using a composite desirability function defined as the geometric mean of the individual desirabilities, and the resulting desirability surface as a function of pH and reagent dosage at the model-predicted optimum temperature is shown in Figure 7. A single, clearly defined maximum was identified at pH 7.31, an LS-O2 dosage of 0.331 kg/t, and a temperature of 28°C, corresponding to a composite desirability of $D=0.872$ and predicted values of $\eta=76.64\%$ and TiO_2 grade= 21.40% (Table 6). The high-desirability region ($D>0.85$) was confined to the vicinity of the optimum, indicating that close control of pH and reagent dosage is required to maintain favourable values for both responses. The narrowing of the contours towards the boundaries of the design space shows that operation outside the identified region, particularly at low reagent dosage and extreme pH values, leads to a substantial decrease in desirability.

The multivariable optimum identified in this study differs both quantitatively and methodologically from that obtained by sequential one-factor-at-a-time variation of the same reagent system (pH $\rightarrow$ dosage $\rightarrow$ temperature). Joint optimisation using RSM predicted slightly higher ilmenite recovery and concentrate grade at a marginally lower pH and temperature but a higher reagent dosage. These differences highlight the advantage of accounting for factor interactions rather than optimising each variable separately.

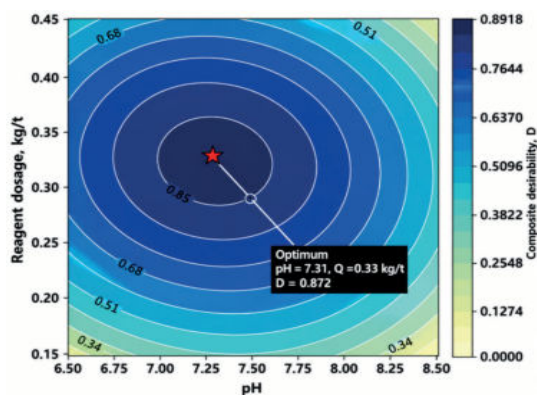


Figure 7. Contour plot of composite desirability as a function of pH and reagent dosage at the model-predicted optimum temperature (28°C).

Table 6. Model-predicted optimal flotation conditions obtained using the composite desirability function.

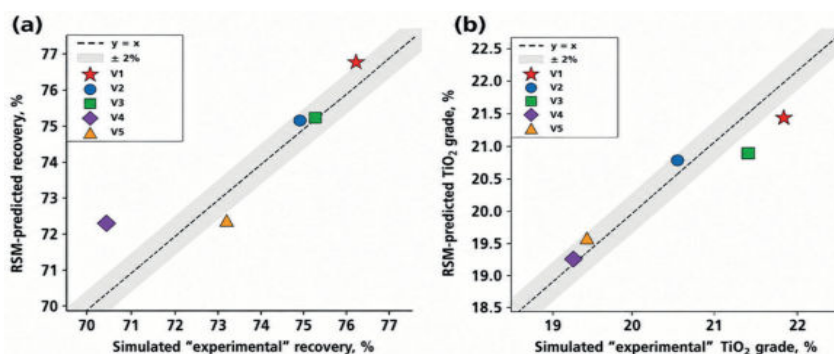
Parameter	Value
pH	7.31
LS-O2 dosage, kg/t	0.331
Temperature, °C	28
Predicted recovery, %	76.64
Predicted TiO_2 grade, %	21.40
Composite desirability, D	0.872

Model verification. To assess the predictive reliability of the fitted models outside the immediate neighbourhood of the design points used for their construction, five additional operating points were evaluated: the predicted optimum (V1), two interior points not coinciding with design or centre points (V2, V3), a corner combination lying outside the original BBD point set (V4), and one point deliberately placed beyond the upper dosage boundary of the design region to test extrapolation behaviour (V5). The corresponding conditions, model predictions and verification results are given in Table 7 and Figure 8.

Table 7. Verification results.

Run	pH	LS-O2 dosage, kg/t	T, °C	Predicted recovery, %	Experimental recovery, %	d _η , %	Predicted TiO ₂ grade, %	Experimental TiO ₂ grade, %	d _{TiO₂} , %
V1 (optimum)	7.31	0.331	28.0	76.64	76.09	0.72	21.40	21.84	2.00
V2	7.00	0.250	25.0	75.35	75.21	0.20	20.79	20.65	0.71
V3	8.00	0.350	35.0	75.21	74.99	0.30	20.89	21.43	2.52
V4 (corner)	6.50	0.200	35.0	72.25	70.58	2.36	19.23	19.28	0.26
V5 (extrapolation)	7.50	0.500	30.0	72.44	73.11	0.92	19.56	19.45	0.58

Agreement between the model predictions and the verification results was good across all five points, with relative deviations ranging from 0.20% to 2.52% and a mean absolute deviation of 0.90% for recovery and 1.21% for TiO₂ grade – well within the $\pm 5\%$ band typically considered acceptable for RSM-based process models (Figure 8). The largest deviation (2.36% for recovery, corner point V₄) occurred at the factor combination furthest from the design centre and not included in the original Box-Behnken matrix, which is the expected behaviour for a second-order polynomial model, whose predictive accuracy is highest near the centre of the design space and decreases progressively toward its boundaries and beyond. Notably, even the deliberately extrapolated point (V₅, dosage=0.50 kg/t, outside the upper design bound of 0.45 kg/t) showed a deviation of less than 1%, suggesting that the fitted quadratic surface remains reasonably well-behaved in the immediate vicinity of the design region.

Figure 8. Predicted versus verification values for (a) ilmenite recovery and (b) TiO₂ grade.

The obtained results confirm that the regression models provide a statistically adequate and practically reliable description of the flotation response of the ilmenite ore to pH, LS-O2 dosage and temperature over the investigated range.

Conclusion. A response surface methodology (RSM) based on a three-factor Box-Behnken design was applied to model and optimise the flotation of an ilmenite ore using a single modified lignosulphonate reagent, LS-O2. Second-order polynomial models were developed to relate pulp pH, reagent dosage, and temperature to ilmenite recovery and the TiO_2 grade of the concentrate. ANOVA confirmed that both models were statistically significant, with non-significant lack of fit and high coefficients of determination ($R^2=0.996$ for recovery and $R^2=0.984$ for TiO_2 grade).

Reagent dosage was identified as the dominant factor governing both responses, followed by pulp pH, while temperature had a comparatively minor effect. For TiO_2 grade, the linear temperature term and temperature-related interactions were not statistically significant, although the quadratic temperature term remained significant. Joint optimisation of both responses using a composite desirability function identified an optimum at pH 7.31, an LS-O2 dosage of 0.331 kg/t, and a temperature of 28°C, predicting an ilmenite recovery of 76.6% and a TiO_2 grade of 21.4%. Verification at five independent operating points, including a boundary combination and an extrapolated dosage beyond the design range, supported the predictive reliability of the models, with relative deviations ranging from 0.20% to 2.52% and mean absolute deviations of 0.90% for recovery and 1.21% for TiO_2 grade.

The results show that a statistically validated quadratic RSM model can adequately describe the combined and interactive effects of pH, reagent dosage, and temperature on ilmenite flotation using a single lignosulphonate-based reagent.

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