International Journal of Science and Research Archive
eISSN: 2582-8185
Cross Ref DOI: 10.30574/ijsra
Journal homepage: https://ijsra.net/

(Research Article)

Overview of flotation studies on low grade limestone for sustainable industrial potentiality: A case study

Thippeswamy DR [1,*], Venkataiah C [1], Sharath Kumar P [2] and Basavaraj Hatti [1]

[1] *Department of Applied Geology, PG Centre, Nandihalli-Sandur, VSK University, Ballari-583119, Karnataka, India.*
[2] *Department of Mineral Processing, PG Centre, Nandihalli-Sandur, VSK University, Ballari-583119, Karnataka, India.*

International Journal of Science and Research Archive, 2024, 13(01), 3248–3266

Publication history: Received on 16 September 2024; revised on 27 October 2024; accepted on 29 October 2024

Article DOI: https://doi.org/10.30574/ijsra.2024.13.1.2059

Abstract

In the present investigation, we used low-grade limestone from the Joldhal region of the Shimoga schist belt in Karnataka to upgrade the CaO and minimize the gangue components for its effective industrial utilization. These samples, having SiO_2-10.40%, 42.50% CaO, and 5.20% Al_2O_3. We used D-12 Denver laboratory flotation equipment for this study. In optimization studies, we used variables 0.8, 1.0, and 1.2 kg/t collector [sodium oleate] dosage and 0.5, 0.6 and 0.7 kg/t depressants [sodium silicate]. In the detailed experimental studies, we used an optimum dosage of 1.2 kg/t collector and 0.7 kg/t depressants. Froth concentrate was collected in cumulative flotation time frames of 15, 30, and 60 seconds, remaining considered as tailings. The collected product and tailings were filtered, dried, and weighed, and the obtained assays were analyzed. It was observed that the optimum result was achieved in a -200# size class with a CaO percentage increase from 42.50% to 52.83% and silica reduction from 10.50% to 2.01% in 15-second products. For the obtained test results, product performance computations such as weight percentage recovery, CaO grade recovery, grade ratio concentration, and weight distribution ratio were performed. When processing this sample, direct flotation proved to be a more effective treatment. The obtained concentrate can be directly utilized in the cement, iron, and steel industries. The results of this study will probably extend the life of raw material resources for future requirements, which will have vital implications for rapidly expanding industries.

Keywords: Limestone; Joldhal Formation; CaO; flotation; Collectors; Calcite; Concentrate

1. Introduction

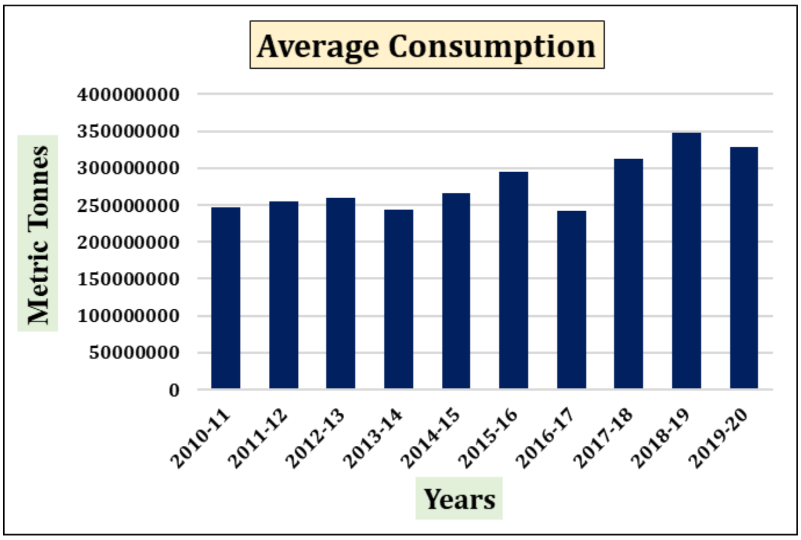
Limestone, a sedimentary litho unit primarily composed of calcite and other minerals, has diverse industrial applications in cement, iron and steel, plastic, paper, chemical, sugar, and other metallurgical industries [1-4] For iron and cement making, limestone with a minimum content of 44% CaO, 4% MgO, and 6% SiO_2. A steel-making sample with 53% CaO, 1.5% MgO, and 1.5% SiO_2 might be used. The present limestone sample having SiO_2-10.40%, 42.50% CaO, and 5.20% Al_2O_3. The depletion of suitable-quality limestone for various sectors has been rapid in recent decades due to overuse brought on by urban and developmental activities that are gradually reducing limestone resources, as depicted in Fig.1. To mitigate this technological problem, there is a need for innovative process optimizations to effectively use lower-grade limestone or explore alternative materials in industrial processes. To conserve the available limestone resources, low-grade ore must be beneficiated using proper methods to make it industrially usable [5].

There is an abundance of low-grade ores containing industrial minerals around the world, hence, it is inevitable to utilize low-grade limestone with proper beneficiation to meet the present specifications of the requirement [6]. Approximately India's available limestone deposits will only survive for 40 years at the current pace of usage. Increasing the value of calcium oxide will contribute to the vision of a sustainable world. Hence, we used flotation techniques to enhance the CaO grade. Froth flotation is the cheapest, most popular, and most extensive scientific innovation technique used in the

* Corresponding author: Thippeswamy DR

mineral processing industry for recovering valuable minerals from their ores [7-9]. This applicability has recently expanded to new fields like effluent treatment [10]. It is based on the difference in the surface hydrophobicity of solid particles [6]. An Indian limestone beneficiation plant employs various processes, including crushing, screening, scrubbing, grinding, and classification. It includes the development of commercially viable techniques for lowering the mass of gangue minerals [11]. In the context of flotation, the process involves treating a mixture of finely ground ore particles with water and chemicals to create a slurry. The hydrophobic particles are then carried to the surface of the flotation cell, forming a froth that can be skimmed off as concentrate. Which is hydrophilic and sinks to the bottom as tailings. Direct flotation (float carbonate minerals) as well as reverse flotation (float gangue minerals) were adopted to reduce SiO_2% and enhance CaO grade [12]. Either cationic or anionic collectors can be used to accomplish the task of lifting calcite or silica [12]. Many previous studies recommended that sodium oleate be considered an effective collector [13-15]. Flotation-related tests have generally been evaluated by two parameters, like mineral recovery and concentrate grade [16]. The limestone available in the study region is crystalline with schistose nature; it can be further utilized in cement, steel, and other minor industries.

This study investigates the beneficiation of low-grade limestone using flotation techniques. Other techniques are not encouraging good yields. Hence, in this study, we used flotation techniques. Since previous workers on beneficiation studies on limestone ore had not taken cognizance of the mineralogy of the ore prior to their investigations, this study provides a beneficiation approach based on the first ore mineralogical examination. Aim of the studies. (i) To conduct flotation studies for the limestone sample; (ii) To demonstrate chemical and mineralogical characteristics of feed and flotation products; (iii) To develop a flow sheet to treat the limestone for CaO grade enrichment; (iv) To enhance the utilization of marginal-grade ore and sustainable development for future mineral resources. The present investigation examines the impact of process variables on the processing of high silica-containing limestone to produce a concentrate with less than 3% silica for the cement and iron-making industries.



(Source; Indian mineral year book 2010-2018)

Figure 1 Illustrate the average consumption of limestone by different industries from 2010-2020 in India

1.1. Description of the study site

The study area is located in the central part of Karnataka, India, and in administrative terms, it covers the three districts of Davanagere, Shivamogga, and Chikmagalur. It is well connected in all seasons by a road network. The collected sample location is situated in part of the Joldhal Formation of the Shimoga schist belt. The area lies between longitude 75°48'00" and 75°59'24" E latitude 13°45'00" and 14°00'00" N, as illustrated in Fig.2. The Survey of India Toposheet No. 48 O/13 covers the research area. However, the study region enjoys a semi-arid climate in the middle temperate zone, with an annual precipitation of about 500 to 750 mm and a temperature of 38°C in summer and 16°C in winter, respectively. The terrain in the area is mainly undulating and hilly, with an elevation of 275 m to 335 m above mean sea level. The highest peak in the study area is Rangayyanagiri Betta, which is around 940 m above mean sea level. A few isolated granite batholiths were exposed in this area. Lithologically, the region is composed of granite gneiss, quartz chlorite schist, limestone, dolomites, carbonaceous phyllites, magnesian limestone, and manganiferous iron phyllites. This region has a great history of prolonged limestone mining activities, which provided the raw material for Vishweshwaraiah Iron and Steel Limited (VISL) Bhadravathi.

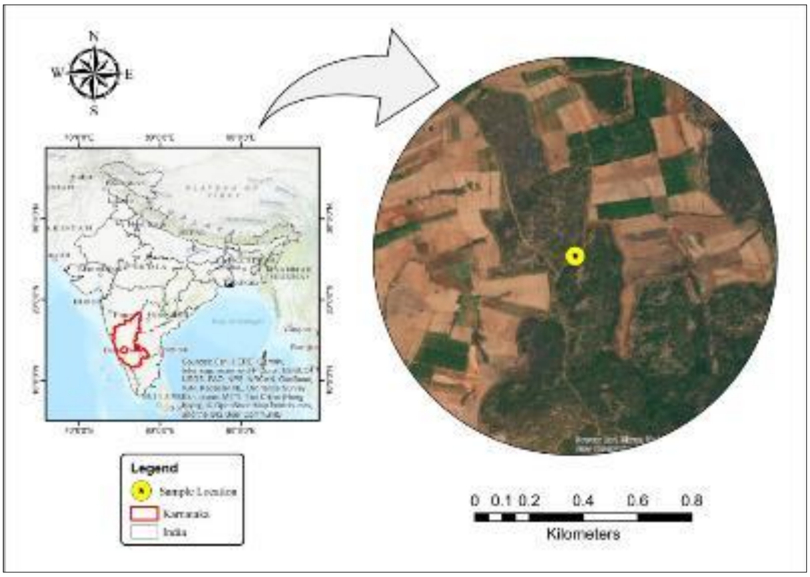


Figure 2 Depicts location map of study area with sample location

2. Materials and Methods

Low grade limestone samples were used in this investigation, procured from the vicinity of Joldhal Formation. For this sample, preliminary petrographic, X-ray fluorescence, and X-ray diffraction studies were investigated. The selected sample was subjected to crushing and ground into a -200# size fraction. The experiment was carried out using a D-12 Denver flotation laboratory machine. The reagents sodium oleate [collector], sodium silicate [depressant], and NaOH were used for maintaining the pH. The sample was conditioned with pre-determined amounts of sodium oleate (0.8, 1.0, and 1.2 kg/t) and sodium silicate (0.5, 0.6, and 0.7 kg/t) as a silica depressant. The natural pH was maintained. In the single flotation test, we procured three different time (15, 30, and 60-second) intervals of concentrate product. The obtained product was chemically investigated through the X-ray fluorescence method.

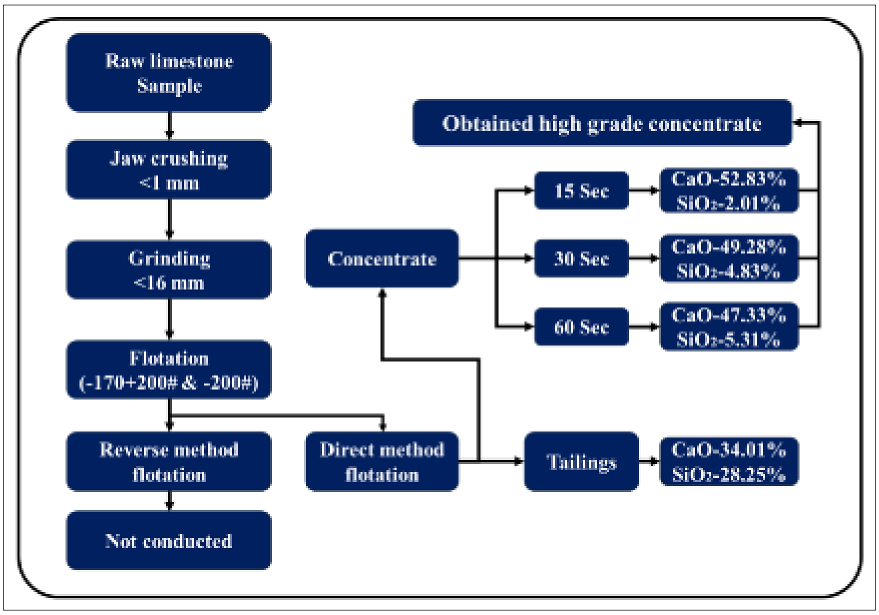


Figure 3 Depicts the methodology flow chart for Flotation studies

2.1. Feed characterization

The characterization of the feed sample prior to beneficiation is critical to developing the beneficiation approach. It includes sample chemistry (Table 1), identification of valuable and gangue minerals (major, minor, and trace minerals), textures of mineral phases, and liberation size. The limestone sample appeared greyish banded, crystalline, and coarse- to fine-grained in nature [1]. The polished, thin section of limestone sample was analyzed using plane-polarized transmitted light under an optical petrological microscope (Olympus). It shows a rhombohedral cleavage pattern, anisotropy, and a very coarse grain texture and variable size, with anhedral to subhedral calcite grains that are heterogeneously distributed throughout the sample [6,17], and its higher-order interference colour ranges from light pink to greenish. The minerals present in the limestone are calcite, quartz, muscovite mica, and chlorite. Chlorite minerals show the crenulation structures depicted in Fig.4, b. The principal gangue minerals in the sample are quartz, mica, and chlorite minerals. Clay also occurs at the intergranular boundaries of calcite at the microlevel. Calcite, which is liberated from the gangue mineral, is about 200# in size. Mineral particle liberation varies based on the grain size of the limestone and the physical quality of the impurities. For the best screening results, a thorough petrographic examination is required.

2.2. Chemical characterization

The limestone deposits in the study area are metamorphic calc-schist varieties of limestone. These limestones, SiO_2-10.40%, 42.50% CaO, and 5.20% Al_2O_3, are not suitable for cement and iron making due to their low grade, but they can be used for blending or after beneficiation. Therefore, in this study, calc-schist limestone has been subjected to detailed beneficiation studies to upgrade them as mineable reserves. The oxide percentages of different constituents of representative samples chosen for study are given below in Table 1.

Table 1 Chemical composition of feed Sample

Radicals	SiO_2	TiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	MnO	Na_2O	K_2O	P_2O_5	LOI
%	10.40	0.24	5.20	3.26	42.50	2.33	0.82	0.49	0.66	0.0248	34.03

2.3. Mineralogical Characterization

Limestone deposits from the study area are medium-grained and consist of calcite quartz and chlorite mica as their constituents Fig.5. It is very difficult to distinguish the two minerals Megascopically. However, clastic grains of quartz show irregular boundaries; occasionally, quartz is medium-grained and mostly subrounded to rounded in shape. Chlorite occurs in crenulation cleaves Fig.4, a. Detailed mineralogical studies of limestone samples have been made to choose a suitable method for beneficiation. The X-ray diffraction method is an efficient, accurate, and reliable technique to identify a number of mineral phases that can be identified in samples [18]. XRD was used to characterize the feed and flotation products in order to determine mineral components and relative abundance by using Model Rigako, Smartlab, Japan. Monochromatized Cu Kα radiation with a Ni filter was used to identify the mineral phases in the corresponding fractions.

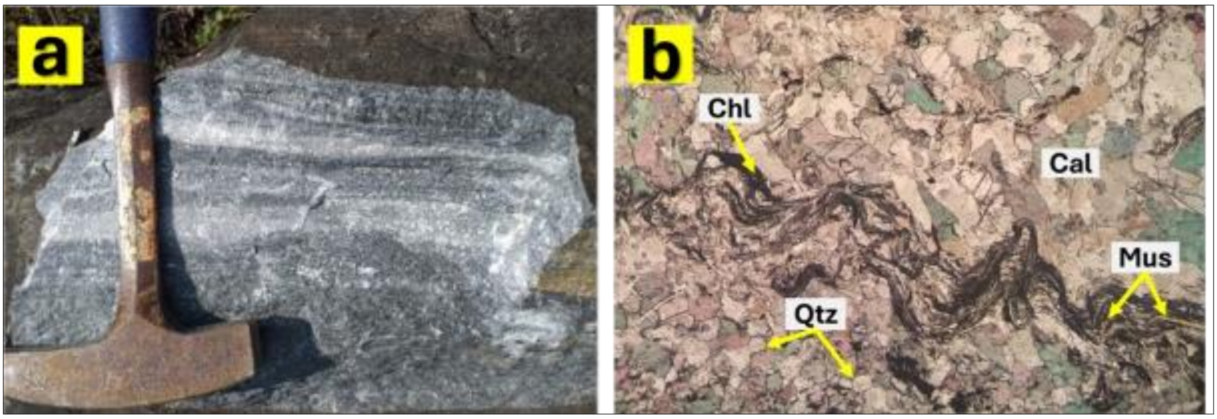


Figure 4 a) Megascopic specimen of limestone sample, b) Both coarse- and fine-grained calcite crystals; note chlorite impurities in both coarse- and fine-grained crystals shows.

The diffraction pattern was indexed using interplanar spacing (d-value), peak position (2θ degrees), and peak intensity. During the XRD studies, the limestone sample showed major calcite mineral phases along with quartz, chlorite, mica, feldspar, and kaolinite gangue minerals. The XRD patterns of the feed sample are provided in Fig.5; the pattern clearly displays a main reflection at 3.01 Å. The main peak is typical of calcite and can be attributed to the 104-plane other peak at position d spacings of 14.15Å, 9.93Å, 7.04Å, 3.34Å, and 3.19Å and indicates a small amount of chlorite, mica, kaolinite, quartz, and feldspar. The minerals are identified by the standard JCPDS data file, and the present limestone sample XRD pattern matches the reported JCPDS Card No. 01-086-2335.

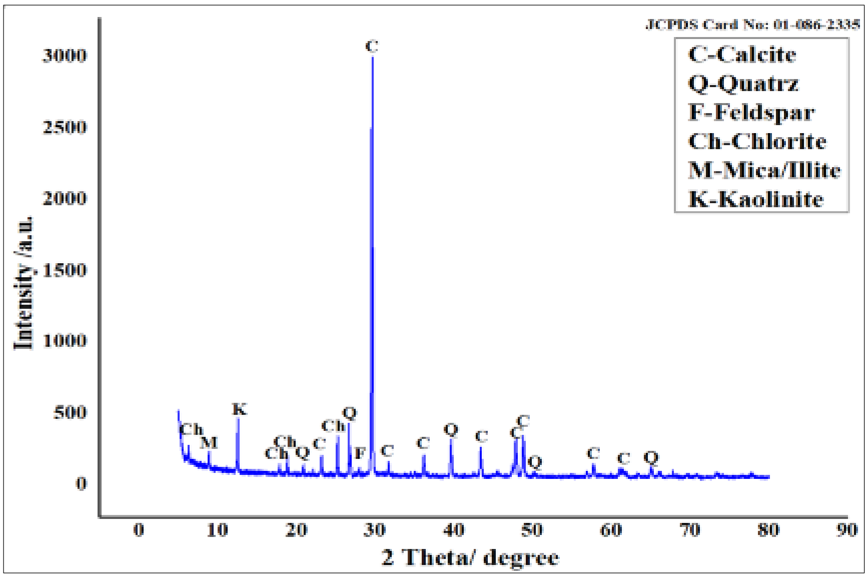


Figure 5 X-ray diffraction pattern of feed limestone sample

2.4. Flotation Experiment

Based on the optimum conditions obtained for the depressant and collector dosage, detailed experimental studies were carried out on a selected low-grade limestone sample. A representative 6 kg limestone sample was crushed using a laboratory scale jaw and ground in a stainless-steel rod mill for 10 minutes. A flotation test was conducted for two different sizes of class (-170+200# and -200#). A 250 g sample of powder has been taken for a test carried out in a D-12 Denver laboratory flotation cell having a capacity of 1000 ml [16]. The chemical reagent of sodium oleate emulsion to float calcite minerals, sodium silicate, was used as a depressant for the silicate minerals (SiO_2). The pH of the pulp was adjusted using NaOH solutions, and the depressant dosage of sodium silicate was also kept constant at 0.7 kg/t. The conditioning time for sodium silicate was 5 minutes, and for sodium oleate, it was 2 minutes. Collector dosages of sodium oleate varied from 0.8, 1.0, and 1.2 kg/t. The frother was added during the last minute of the conditioning period, and air was introduced to the pulp at the end of conditioning. The froth product, collected as the top layer, was removed to a tray. Subsequently, three concentrates were collected with cumulative flotation time frames of 15, 30, and 60 sec. The froth was collected manually by using scraper blades; after the collected material was left over, the cell was considered tailings. The water level in the cell was readjusted to the original volume after collecting different time intervals of froth. The collected product in the form of concentrate and tailing was dried, and detailed chemical analysis was carried out by using XRF to determine the CaO and SiO2 percentages in the concentrate and the CaO recovery

3. Results

3.1. Optimization studies

Optimization is the manual decision-making process to minimize the cost, effectiveness, and value of the product [19], and these techniques are crucial for the sustainable and efficient extraction of valuable minerals from ores. Higher-level operational optimization systems are integrated into every unit's operation to ensure that the operational indices quality, efficiency, and consumption during the production phase fall within their target ranges and to provide the controllers with setpoints [20-21]. These studies contribute to increased resource recovery, reduced environmental impact, and improved economic viability in the mining industry. Adjustment of operating conditions based on optimization models to maximize the recovery of valuable minerals. Optimization may involve balancing factors such as recovery rates, concentrate quality, and processing costs. For limestone beneficiation, flotation studies can be conducted to optimize the reagent dosage. This involves investigating the effects of variable-dosage flotation reagents, pulp conditions, and other parameters on the recovery and grade of limestone. These studies aim to maximize the effectiveness of sodium silicate while addressing factors such as cost, environmental impact, and end product quality.

3.2. Optimization study of Sodium silicate (Depressants):

Utilizing sodium silicate allowed for the removal of silica particles and improved the CaO grade in the concentrate. We performed sodium silicate (depressant) optimization trials using various dosages, such as 0.5, 0.6, and 0.7 kg/t, while maintaining other variables constant. Optimization studies for sodium silicate can be conducted to enhance its performance and efficiency towards depressing the silica percent in concentrate products. The analytical results of the experiments demonstrate that sodium silicate, at 0.7 kg/t, has the highest silica depression. The ideal value is therefore 0.7 kg/t of sodium silicate. The nature of these studies will depend on the specific application, as sodium silicate is used in various diverse industries, such as processing industries. The results are tabulated in Tables 2, 3, 4 and 5, and a graphical representation is illustrated in Fig.6 (a, b, and c). In the Fig., we have observed the CaO and SiO_2 comparisons of different depressant dosages.

Table 2 Optimization trials of variable Sodium silicate (Depressants) dosage

Details	**Trial-1**	**Trial-2**	**Trial-3**
Feed (gm)	0.250	0.250	0.250
Grinding Mill Size	300×175 mm	300×175 mm	300×175 mm
RPM	48-50	48-50	48-50
Cell Capacity (Kg)	1	1	1
Sodium oleate (kg/t)	1.2	1.2	1.2
Sodium silicate(kg/t)	0.5	0.6	0.7
Conditioning Time(min)	10	10	10
Flotation Time(m)	1	1	1
Pulp Density (%)	20	20	20
Impeller speed(r.p.m.)	1400	1400	1400
pH maintained	9.0	9.0	9.0
pH modifier	Sodium Hydroxide	Sodium Hydroxide	Sodium Hydroxide

Table 3 Optimization test result effect of depressants (Sodium silicate) dosage of 0.5 kg/t

Size Fraction	**Reagent dosage**		**Product**	**Weight**	**CaO**	**SiO_2**
	Sodium silicate	**Sodium Oleate**				
-170 +200#	0.5 kg/t	1.2 kg/t	Feed	100	42.50	10.40
			Concentrate	63.28	45.17	8.04
			Tailings	36.72	40.23	12.68
			Material recovery	100		
-200#	0.5 kg/t	1.0 kg/t	Feed	100	42.50	10.40
			Concentrate	66.47	46.15	7.19
			Tailings	33.53	37.22	14.35
			Material recovery	100		

Table 4 Optimization test result effect of depressants (Sodium silicate) dosage of 0.6 kg/t

Size Fraction	Reagent dosage		Product	Weight	CaO	SiO_2
	Sodium silicate	Sodium Oleate				
-170 +200#	0.6 kg/t	1.2 kg/t	Feed	100	42.50	10.40
			Concentrate	66.35	46.30	6.02
			Tailings	33.65	39.12	15.38
			Material recovery	100		
-200#	0.6 kg/t	1.2 kg/t	Feed	100	42.50	10.40
			Concentrate	69.31	47.65	4.89
			Tailings	30.69	37.26	18.82
			Material recovery	100		

Table 5 Optimization test result effect of depressants (Sodium silicate) dosage of 0.7 kg/t

Size Fraction	Reagent dosage		Product	Weight	CaO	SiO_2
	Sodium silicate	Sodium Oleate				
-170 +200#	0.7 kg/t	1.2 kg/t	Feed	100	42.50	10.40
			Concentrate	69.19	50.71	4.02
			Tailings	30.81	30.48	19.78
			Material recovery	100		
-200#	0.7 kg/t	1.2 kg/t	Feed	100	42.50	10.40
			Concentrate	70.04	52.62	3.46
			Tailings	29.96	29.63	20.77
			Material recovery	100		

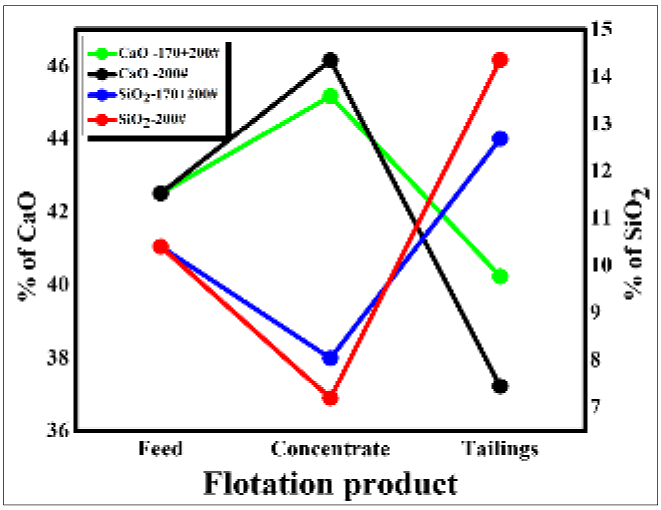


Figure 6 a Graphical illustrated of grade enhanced dosage 0.5 kg/t of sodium silicate (depressants)

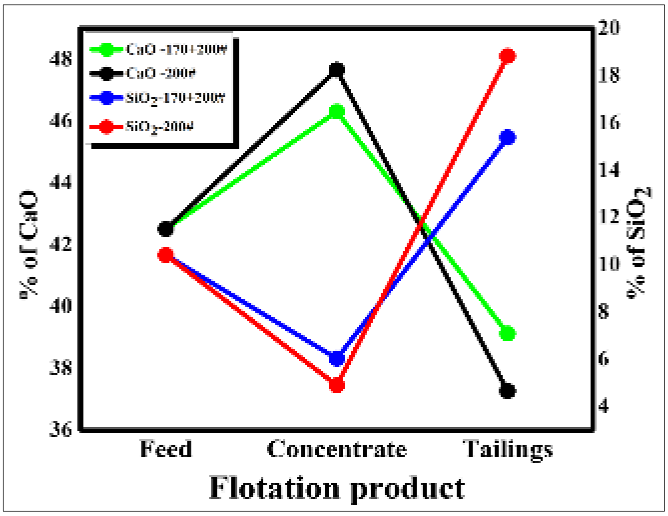


Figure 6 b Graphical illustrated of grade enhanced dosage 0.6 kg/t of sodium silicate (depressants)

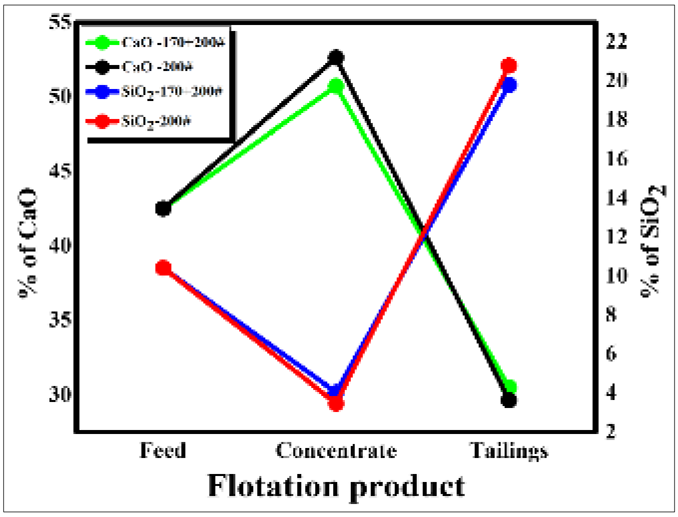


Figure 6 c Graphical illustrated of grade enhanced dosage 0.7 kg/t of sodium silicate (depressants)

3.2.1. Optimization study of Sodium oleate (Collector):

These experiments were carried out to identify the amount of sodium oleate needed to make the calcite float without floating much silica. Sodium oleate is the sodium salt of oleic acid; it is commonly used as a collector in the flotation of calcite minerals, including fluorite, apatite, scheelite, and other minerals. The flotation process involves selectively separating valuable minerals from gangue (worthless material) based on differences in their surface properties, and its performance may vary depending on the mineralogical composition of the ore. The use of sodium oleate in ore beneficiation processes aims to increase the recovery of valuable minerals and improve the overall efficiency of mineral processing operations. Ongoing research and optimization studies are conducted to improve the efficiency of sodium oleate in specific mineral processing applications. The trials were carried out with varied sodium oleate dosage variations of 0.8, 1.0, and 1.2 kg/t in order to determine the sodium oleate's optimal condition. Based on experimental research, a 1.2 kg/t dose results in the maximum enrichment of CaO. The results are tabulated in tables 6, 7, 8 and 9, and a graphical representation is illustrated in Fig.7 (a, b and c).

Table 6 Optimization trials of variable Sodium oleate (Collector) dosage

Details	Trial-1	Trial-2	Trial-3
Feed (gm)	0.250	0.250	0.250
Grinding Mill Size	300×175 mm	300×175 mm	300×175 mm
RPM	48-50	48-50	48-50
Cell Capacity (Kg)	1	1	1
Sodium oleate (kg/t)	0.8	1.0	1.2
Sodium silicate(kg/t)	0.7	0.7	0.7
Conditioning Time(min)	10	10	10
Flotation Time(m)	1	1	1
Pulp Density (%)	20	20	20
Impeller speed(r.p.m.)	1400	1400	1400
pH maintained	9.0	9.0	9.0
pH modifier	Sodium Hydroxide	Sodium Hydroxide	Sodium Hydroxide

Table 7 Optimization test result effect of collector (Sodium oleate) dosage of 0.8 kg/t

Size Fraction	Reagent dosage		Product	Weight	CaO	SiO_2
	Sodium silicate	Sodium Oleate				
-170 +200#	0.7 kg/t	0.8 kg/t	Feed	100	42.50	10.40
			Concentrate	65.36	47.82	8.01
			Tailings	34.64	39.43	14.28
			Material recovery	100		
-200#	0.7 kg/t	0.8 kg/t	Feed	100	42.50	10.40
			Concentrate	66.83	47.82	7.18
			Tailings	33.17	37.	15.32
			Material recovery	100		

Table 8 Optimization test result effect of collector (Sodium oleate) dosage of 1.0 kg/t

Size Fraction	Reagent dosage		Product	Weight	CaO	SiO_2
	Sodium silicate	Sodium Oleate				
-170 +200#	0.7 kg/t	1.0 kg/t	Feed	100	42.50	10.40
			Concentrate	67.05	48.43	6.01
			Tailings	32.95	36.44	16.75
			Material recovery	100		
-200#	0.7 kg/t	1.0 kg/t	Feed	100	42.50	10.40
			Concentrate	68.21	49.95	5.76

			Tailings	31.79	35.78	18.32
			Material recovery	100		

Table 9 Optimization test result effect of collector (Sodium oleate) dosage of 1.2 kg/t

Size Fraction	Reagent dosage		Product	Weight	CaO	SiO_2
	Sodium silicate	Sodium Oleate				
-170 +200#	0.7 kg/t	1.2 kg/t	Feed	100	42.50	10.40
			Concentrate	69.19	50.71	4.74
			Tailings	30.81	30.48	19.27
			Material recovery	100		
-200#	0.7 kg/t	1.2 kg/t	Feed	100	42.50	10.40
			Concentrate	70.68	51.32	3.46
			Tailings	29.32	29.63	23.80
			Material recovery	100		

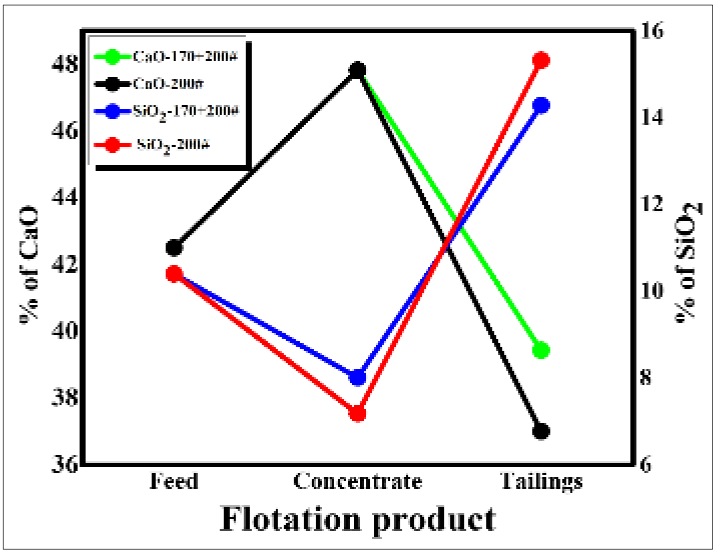


Figure 7a Graphical illustrated of grade enhanced of dosage 0.8 kg/t of sodium oleate (collector)

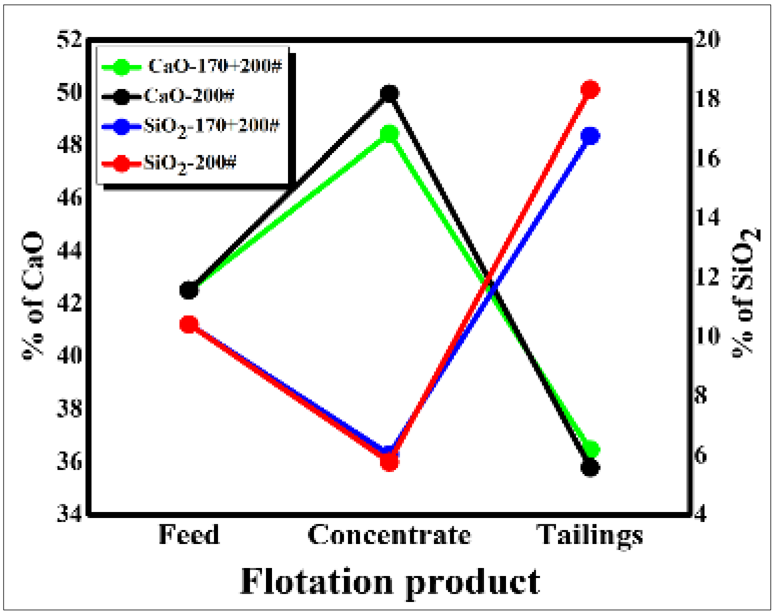


Figure 7b Graphical illustrated of grade enhanced of dosage 1.0 kg/t of sodium oleate (collector)

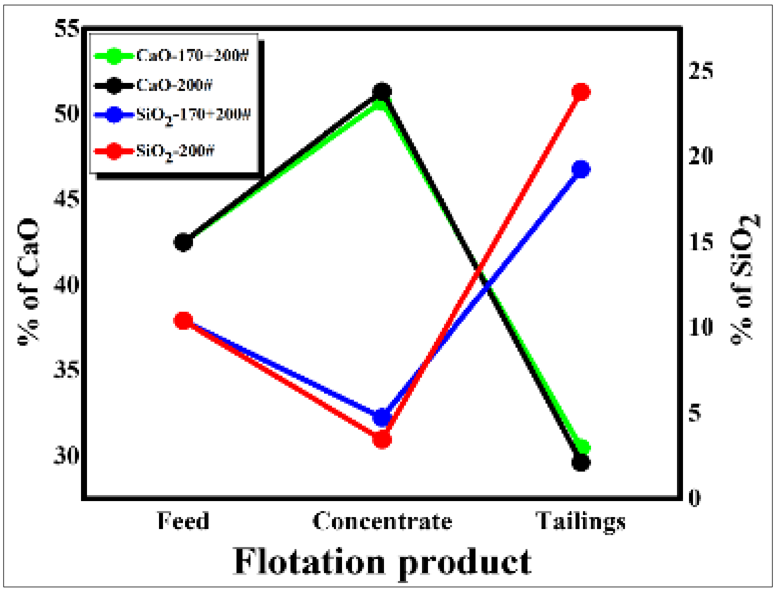


Figure 7c Graphical illustrated of grade enhanced of dosage 1.2 kg/t of sodium oleate (collector)

From these results, a set of optimum conditions for the process and system parameters expected to produce a high-quality product with a reasonable recovery will be selected. In the optimized flotation experiments, we used 0.7 kg/t sodium silicate as a depressant and 1.2 kg/t sodium oleate as a collector. Collector and depressant dose values will be dependent on the gangue percentage in the feed materials. In this sample, there is a low gangue component; hence, we used a low depressant and collector. The difference is attributed to a lack of mineral (calcite) liberation in the -170+200# class.

Table 10 Flotation test results of (-170+200#) & (-200#) under optimized conditions (0.7 kg/t sodium silicate, 1.2 kg/t sodium oleate

MOG (Mesh of Grind)	Reagent dosage		Flotation product	Flotation Time in Sec	Assay in percentage		
	Sodium silicate	Sodium oleate			Weight	CaO	SiO_2
-170+200#	0.7	1.2	Feed		100	42.50	10.40
			Concentrate	15	27.02	49.25	3.09
				30	24.78	47.22	5.93
				60	17.08	46.34	8.35
			Tailings		31.12	36.25	14.03
			Material recovery		100		
-200#	0.7	1.2	Feed		100	42.50	10.40
			Concentrate	15	29.05	52.83	2.01
				30	23.54	49.28	4.83
				60	19.38	47.33	5.31
			Tailings		28.03	34.01	28.25
			Material recovery		100		

4. Performance computations

Performance to enhance the optimization of the beneficiation process to recover and grade valuable components while minimizing mineral loss. The findings will contribute to scientific understanding and industrial advancement, balancing economic considerations with environmental sustainability. Flotation enhances limestone's market value. Calculations reveal froth flotation efficiency and effectiveness, evaluating performance based on key parameters. Techniques and instrumentation aid in real-time monitoring and control, though efficiency cannot be expressed universally. Here is some key performance calculations commonly used in flotation.

4.1. Weight % recovered

Weight percent recovered is very essential in flotation. It has been calculated by the percentage of the substance that has been recovered. Moreover, the material weight recovered is 72% of the remaining 28% of tailings. As a result, the plant is expected to produce 7.2 tonnes of concentrate for every 10 tonnes of feed. 15, 30, and 60-second time intervals produce 2.9, 2.3, and 1.9 tonnes, respectively. The following equation was used to calculate the weight percentage of the material.

$$\text{Weight \% recovered} = \frac{C\times(F-T)}{F\times(C-T)} \times 100 \qquad \text{…………..1)}$$

Where,

R is the recovery of the valuable mineral.
C is the concentration of the valuable mineral in the concentrate.
F is the concentration of the valuable mineral in the feed.
T is the concentration of the valuable mineral in the tailings.

4.2. CaO grade recovery

The recovery of calcium oxides (CaO) in flotation is mainly dependent on the collection time of the process. Sometime grade recovery is dependent on the particle size. Decreased Grade and weight of the product in the presence of very coarse particles [22]. It's essential to find the right balance to allow sufficient time for effective flotation. Grade depends on mineralogy, flotation reagents, pH level, pulp density, particle size, temperature, water quality, equipment, and cell design. In this experiment, 85% of CaO was recovered, and grades achieved 36.11%, 27.29%, and 21.58% in a cumulative flotation time of 15, 30, and 60 sec, respectively. The grade recovery for this test is graphically illustrated in Fig.8.

$$\text{CaO grade recovery} = \frac{\text{Assay weight} \times \text{CaO grade in Concentrate}}{\text{CaO grade in Feed} \times 100} \times 100 \text{ ………. 2)}$$

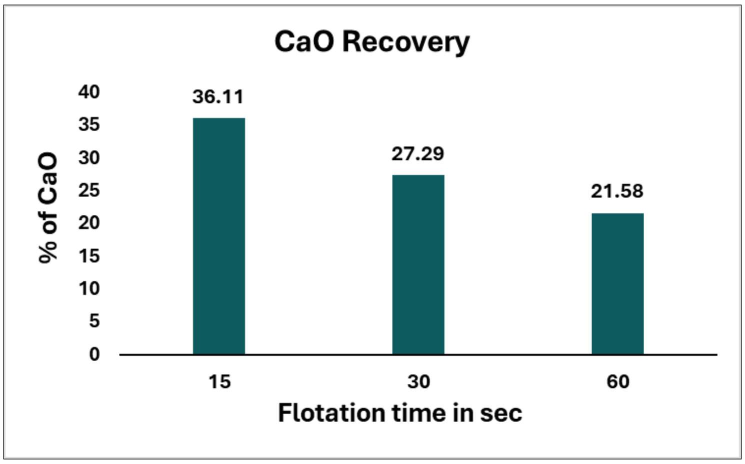


Figure 8 CaO grade recovery of different flotation time

4.3. Grade concentration ratio

The grade concentration ratio is a critical parameter in mineral processing to reflect the efficiency of the valuable minerals. A higher concentration ratio generally signifies a more effective and economically viable processing operation. It provides a quantitative assessment of how effectively the beneficiation process has increased the concentration of valuable components. However, achieving a high concentration ratio must be balanced with considerations such as recovery rates, processing costs, and the overall economics of the mineral deposit. The grade concentration ratio of different flotation times is illustrated in Fig.9. The grade concentration ratio is calculated by the following equation.

$$\text{CaO grade concentration ratio} = \frac{\text{Concentrate grade}}{\text{Feed grade}} \text{……………. 3)}$$

The grade concentration ratio is typically expressed as a numerical value or percentage. A ratio greater than 1 indicates a grade enrichment of the valuable minerals in the concentrate compared to the feed grade. In this case, the 15, 30, and 60-second concentrate is 1.24, 1.15, and 1.11 times more compared to the original feed material.

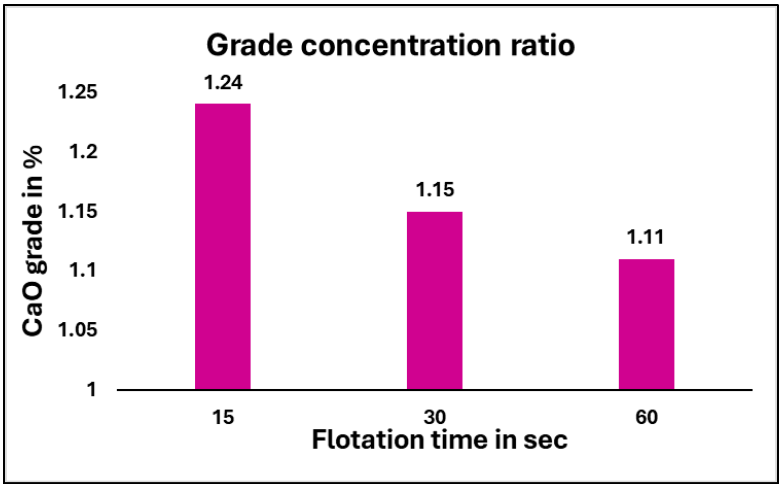


Figure 9 Illustrate the Grade concentration ratio of different flotation time in sec

4.4. Weight distribution ratio (WDR)

The "Weight Distribution Ratio" in the context of flotation processes refers to the distribution of weight or mass between the concentrate and tailings in a flotation separation. A high WDR suggests that a significant proportion of the valuable minerals are reporting to the concentrate, and the separation is effective. Conversely, a low WDR may indicate poor selectivity and incomplete separation of valuable minerals from the gangue (undesirable minerals). WDR distribution decreased, and feed particle size became coarser [22]. Optimizing the flotation process conditions, such as the type and dosage of reagents, pulp density, pH, and other factors, can influence the WDR and improve the overall efficiency of the ore flotation process. It is a key parameter that reflects the efficiency of the separation process and is also an important parameter in flotation studies because it can provide quantitative support for the mining operation. In this flotation process, the weight distribution ratio was calculated for two size classes. In the -200# size class, we found the best result in weight distribution as compared to the -170+200# size class. In the -200# class of 15 seconds, the product was distributed as CaO-33.09% and SiO_2-5.13%, and the gangue distribution in tailings was SiO_2-76.02% and CaO-22.51% by weight obtained. The weight distribution of both CaO and SiO_2 in two different size classes is provided in Tables 11, 12, 13, and 14. The WDR is calculated using the following equation.

$$\text{Weight in } \% = \frac{\text{induvidual weight}}{\text{total weight}} \times 100 \ \ldots\ldots\ldots\ 4)$$

$$\text{Distribution ratio} = \frac{\text{Individual weight}}{\text{Total weight}} \times 100 \ \ldots\ldots 5)$$

4.4.1. WDR for CaO (Calcium oxide)

Table 11 CaO distribution of -170+200# size class

Mesh size	Flotation Time in sec	Weight in gram	Weight in %	Assay % of CaO	Units	Distribution in %
-170+200#	15	67	26.8	49.25	1319.9	29.93
	30	62	24.8	47.22	1171.056	26.55
	60	41	16.4	46.34	759.976	17.22
	Tailings	80	32	36.25	1160	26.30
Total		250	100		4410.932	100

Table 12 CaO distribution of -200# size class

Mesh size	Flotation Time in sec	Weight in gram	Weight in %	Assay % of CaO	Units	Distribution in %
-200#	15	71	28.4	52.83	1500.372	33.09
	30	56	22.4	49.28	1103.872	24.36
	60	48	19.2	47.33	908.736	20.04
	Tailings	75	30.0	34.01	1020.3	22.51
Total		250	100		4533.28	100

4.4.2. WDR For SiO2 (Silicon dioxide)

Table 13 SiO_2 distribution of -170+200# size class

Mesh size	Flotation Time in sec	Weight in gram	Weight in %	Assay % of SiO_2	Units	Distribution in %
-170+200	15	67	26.8	3.09	82.812	10.16
	30	62	24.8	5.93	147.064	18.02
	60	41	16.4	8.35	136.94	16.79
	Tailings	80	32	14.03	448.96	55.03
Total		250	100		815.776	100

Table 14 SiO_2 distribution of -200# size class

Mesh size	Flotation Time in sec	Weight in gram	Weight in %	Assay % of SiO_2	Units	Distribution in %
-200	15	71	28.4	2.01	57.084	5.13
	30	56	22.4	4.83	108.192	9.71
	60	48	19.2	5.31	101.952	9.14
	Tailings	75	30.0	28.25	847.5	76.02
Total		250	100		1114.728	100

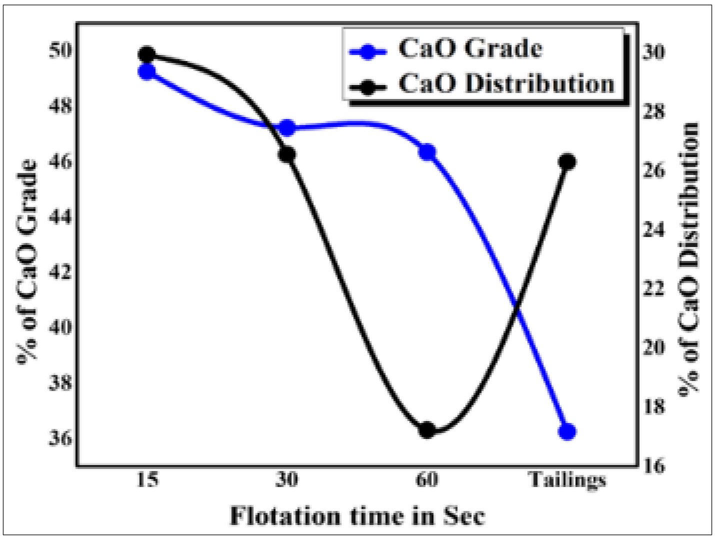


Figure 10 CaO Grade and weight distribution in -170+200# size class

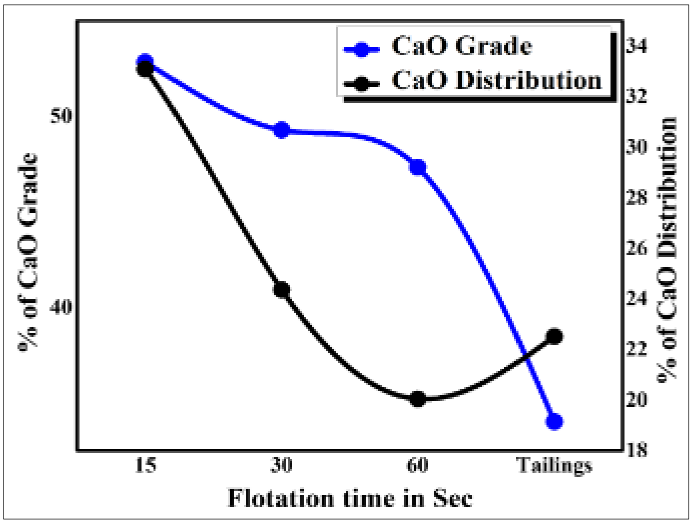


Figure 11 CaO Grade and weight distribution in -200# size class

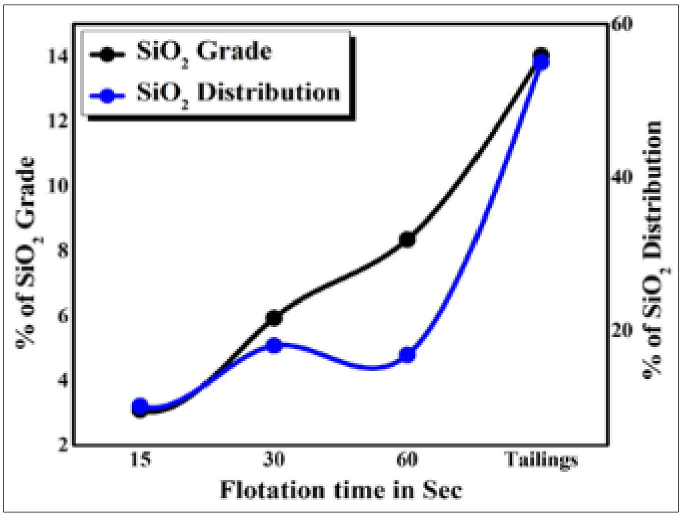


Figure 12 SiO_2 Grade and weight distribution in -170+200# size class

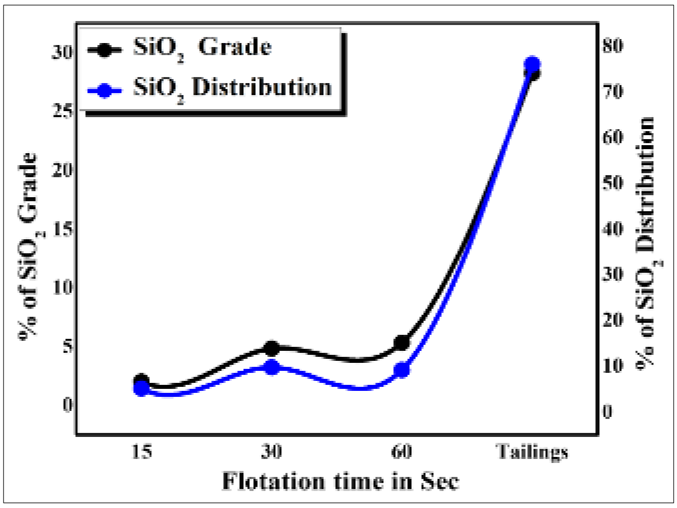


Figure 13 SiO_2 Grade and weight distribution in -200# size class

5. Discussion

The excessive use of high-grade limestone is causing significant environmental issues. Therefore, it has become very essential to highlight the current problems regarding low grades of industrial minerals. This study explores limestone flotation performance to optimise beneficiation processes, improve recovery and grade, minimize mineral loss, and contribute to scientific understanding and industrial advancement. Three froth concentrates were collected at 15, 30, and 60 seconds. The total flotation time was 105 seconds. The beneficiation by flotation technique was found to be more effective in the present study. The two size ranges, viz., -170 +200# and -200#, have both encouraging results, but -200# is the best size. Reagents 0.7 kg/t sodium silicate as depressant and 1.2 kg/t sodium oleate as collector in the optimized experiments. In this study, results indicate that CaO values increased from 42.50% to 52.83% with an 82% recovery. CaO percentage decreased when we increased the collected concentrate time interval by 15 sec (52.83%), 30 sec (49.58%), and 60 sec (47.33%), respectively. Experimental data are presented in Table. 10 and graphically represented in Fig.10, 11, 12, and 13, respectively. Recovery and distribution of CaO and SiO_2 in the concentrate increased in the initial collected product. For 15, 30, and 60 seconds, respectively, the SiO_2 weight distribution in the concentrate exhibits 5.13%, 9.71%, and 9.14%. 76.02% SiO_2 was found in tailings, and the remaining 23.08% was distributed in three concentrates. This implies that the -200# size test findings yield the best results for CaO and SiO_2% in general, recovery, and weight. The product necessary for iron and cement production could be produced by reducing the silica content in the concentrate to 2.01% with an 83% CaO-grade recovery. The better result could be achieved at the first concentrate product of the lowest time (15 sec). The CaO recovery in the concentrate could be increased by using a lower collector dosage. Sustainable point of view in this study broad insight into raw material life extending for a few more years without shortage. This investigation promotes a country having the strength to create raw materials without depending on other countries.

6. Conclusion

The present study investigates the use of flotation techniques to enhance efficiency, economic feasibility, and environmental sustainability. This research mainly targets small, low-grade deposits and their effective utilization for future sustainability. The research findings of the above studies are highlighted below.

- Petrological examination of the samples revealed the presence of quartz, feldspar, chlorite, and mica as the major silicate gangue minerals. Although the majority of these silicates are liberated from the selected feed size (-200#) material, some amounts of silicates are still locked within the calcite grains as inclusions that appear in the concentrate.
- Sodium oleate (NaOL) was used as a collector cum frother, and it has been found to have excellent performance in terms of solubility, selectivity, utilization, froth stability.
- The optimized concentrate's composition is CaO 52.83% and SiO_2 2.01%, indicating a significant increase in CaO content and a reduction in SiO_2 compared to the initial composition.
- The tests clearly suggest that using a flotation technique to achieve limestone concentrates assaying around CaO 52.83% and less than 3% SiO_2 with a CaO recovery of 82% and 70% weight recovery is achieved at a collector dosage of 1.2 kg/t.
- Successful concentration refinement was indicated by the study's 10.33% rise in CaO concentration and 8.39% decrease in SiO_2.
- In comparison to the feed materials, the CaO concentrations increased by 1.24, 1.15, and 1.11 times.
- This product, necessary for cement and iron making, could be produced by reducing the concentrate's SiO_2 content from 10.40% to 2.01%.
- The flotation test result shows a significant drop in froth concentration with increased froth collection timing, suggesting a correlation between froth grade recovery, material recovery weight percentage, and froth collection timing.
- The research effectively and efficiently improved a calcium oxide separation flotation technique, with an emphasis on raising CaO content and lowering SiO_2.

The research provides insightful information for future investigation and advancement in mineral processing, specifically with regard to the use of low-grade resources and the enhancement of flotation techniques. The investigation results in a finding that low-grade deposits in the mineral processing industries could be efficiently used by extending flotation techniques and optimizing collector dosages for CaO value addition.

Compliance with ethical standards

Acknowledgments

We would like to express our sincere thanks to Dept. of Mineral processing, Vijayanagara Sri Krishnadevaraya University, Ballari for providing lab facilities for carried out beneficiation studies. I Grateful to the Department of Applied Geology, Kuvempu University, Shankaraghatta for preparation of thin section and their moral support.

Funding

The research was not financed by any specific agency in the public or commercial sectors.

Disclosure of conflict of interest

The authors declare that they have no known competing personal relationships or financial interests that could have appeared to influence the work reported in this paper.

Author Contributions

Thippeswamy D.R: Research design, Implementation, Conceptualization, Data handling & Curation, Data analysis & Interpretation, Writing & Editing original manuscript, Venkataiah. C: Supervision, Review & Editing original manuscript, Sharath Kumar. P: Data analysis and interpretation of results, and Basavaraj Hatti: Conceptualization and designing of the research article. The idea was developed with collaboration from all authors, who also discussed their recommendations and findings. The final version of the manuscript has been read and approved by all authors.

References

[1] Ravi, B. P., Krishna, S. J. G., Patil, M. R., Rudrappa, C., Kumar, P. S., & Rampur, V. (2017). Beneficiation of Low-Grade Limestone from Madukkarai, Coimbatore District, Tamil Nadu, India. International Journal of Mineral Processing and Extractive Metallurgy, 2, 1-6. https://doi.org/10.11648/j.ijmpem.20170201.11

[2] Dey, S., Sahu, L., Chaurasia, B., & Nayak, B. (2020). Prospects of utilization of waste dumped low-grade limestone for iron making: A case study. International Journal of Mining Science and Technology, 30(3), 367-372. https://doi.org/10.1016/j.ijmst.2020.03.011

[3] Rao, K. V., & Sengupta, S. K. (1997). Beneficiation of a limestone sample: a case study at NML.

[4] Sd, M. K. V., & Vasumathi, N. (2023). Beneficiation of low-grade limestone by flotation. Materials Today: Proceedings, 72, 181-186. https://doi.org/10.1016/j.matpr.2022.07.041

[5] Chitra, S. V. (2011). A brief account of cement industry in India and the norms for suitability of limestone deposits for cement manufacture. Asian Journal of Research in Chemistry, 4(4), 516-523.

[6] Jena, M. S., Sahu, P., Dash, P., & Mohanty, J. K. (2013). Beneficiation of limestone plant rejects for value addition. Journal of hazardous materials, 262, 218-227. https://doi.org/10.1016/j.jhazmat.2013.08.048

[7] Masami Tsunekawa et al. (2009). Removal of Trace Impurity from Limestone Using Flotation Tsunekawa, M., Honma, Y., Yoo, K., Hiroyoshi, N., & Ito, M. (2009). Removal of Trace Impurity from limestone using flotation techniques. Materials transactions, 50(1), 171-176.Techniques. MATERIALS TRANSACTIONS, 171-176. https://doi.org/10.2320/matertrans.M-MRA2008839

[8] Mondal, S., Acharjee, A., Mandal, U., & Saha, B. (2021). Froth flotation process and its application. Vietnam Journal of Chemistry, 59(4), 417-425.

[9] Houot, R. (1982). Beneficiation of phosphatic ores through flotation: Review of industrial applications and potential developments. International Journal of Mineral Processing, 9(4), 353-384. https://doi.org/10.1016/0301-7516(82)90041-2

[10] Matis, K. A., Gallios, G. P., & Kydros, K. A. (1993). Separation of fines by flotation techniques. Separations Technology, 3(2), 76-90. https://doi.org/10.1016/0956-9618(93)80007-E

[11] Filippov, L. O., Duverger, A., Filippova, I. V., Kasaini, H., & Thiry, J. (2012). Selective flotation of silicates and Ca-bearing minerals: The role of non-ionic reagent on cationic flotation. Minerals Engineering, 36, 314-323. https://doi.org/10.1016/j.mineng.2012.07.013

[12] Rao, D. S., Vijaya, K. T., Bhaskar, R. G., & Prabhakar, S. (2011). Effect of the particle size on flotation performance of a siliceous limestone sample. Journal of Mining and Metallurgy A: Mining, 47(1), 37-49.

[13] Pugh, R., & Stenius, P. (1985). Solution chemistry studies and flotation behaviour of apatite, calcite and fluorite minerals with sodium oleate collector. International journal of mineral processing, 15(3), 193-218. https://doi.org/10.1016/0301-7516(85)90035-3

[14] Rao, K. H., Antti, B. M., & Forssberg, E. (1989). Flotation of phosphatic material containing carbonatic gangue using sodium oleate as collector and sodium silicate as modifier. International Journal of Mineral Processing, 26(1-2), 123-140. https://doi.org/10.1016/0301-7516(89)90047-1

[15] Prasad Rao, P. D., & Kunwar, R. K. (1997). Washing studies with a limestone sample for reduction of insoluble: a case study.

[16] Yianatos, J. B., Moys, M. H., Contreras, F., & Villanueva, A. (2008). Froth recovery of industrial flotation cells. Minerals Engineering, 21(12-14), 817-825. https://doi.org/10.1016/j.mineng.2007.12.012

[17] Panda, D. K., Sharma, N. K., & Gotecha, S. K. (2006). Mineral Beneficiation Potentialities Archaean Limestone for Cement Manufacture.

[18] Gunatilaka, H. A., & Till, R. (1971). A precise and accurate method for the quantitative determination of carbonate minerals by X-ray diffraction using a spiking technique. Mineralogical magazine, 38(296), 481-487. https://doi.org/10.1180/minmag.1971.038.296.10

[19] Ding, J., Yang, C., & Chai, T. (2017). Recent progress on data-based optimization for mineral processing plants. Engineering, 3(2), 183-187. https://doi.org/10.1016/J.ENG.2017.02.015

[20] Jäschke, J., & Skogestad, S. (2011). NCO tracking and self-optimizing control in the context of real-time optimization. Journal of Process Control, 21(10), 1407-1416. https://doi.org/10.1016/j.jprocont.2011.07.001

[21] Würth, L., Hannemann, R., & Marquardt, W. (2011). A two-layer architecture for economically optimal process control and operation. Journal of Process Control, 21(3), 311-321. https://doi.org/10.1016/j.jprocont.2010.12.008

[22] Awatey, B., Skinner, W., & Zanin, M. (2013). Effect of particle size distribution on recovery of coarse chalcopyrite and galena in Denver flotation cell. Canadian Metallurgical Quarterly, 52(4), 465-472. https://doi.org/10.1179/1879139513Y.0000000085