Flotation Performance of Biodiesel as Collector for Beneficiation of Low-Grade Graphite Ore

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Abstract. In pursuit of sustainable mineral processing, this study explores the use of biodiesel as a collector for the flotation of low-grade graphite ore. The flotation efficacy of a novel biodiesel collector for the selective recovery of graphite from low-grade ore with 89.5% ash content and 8.7% fixed carbon, using a Taguchi experimental design, was studied. Flotation performance was optimized by evaluating the influence of grind time, depressant sodium silicate dosage, and collector biodiesel dosage on weight recovery, ash content, and fixed carbon recovery. The optimal test configuration was observed at a grind time of 25 minutes, sodium silicate dosage of 1.67 kg/t, and biodiesel dosage of 2.32 kg/t. Under these conditions, the flotation process achieved a concentrate with 11.21% weight recovery, 2.74% ash content, and 94.42% fixed carbon. FTIR and GC analysis of the biodiesel identified its functional groups and the composition of its fatty acid methyl esters, respectively. Regression analysis revealed strong correlations between process variables and responses, with high R^2 values indicating a good fit for most models and illustrated the interaction of variables influencing weight recovery, ash, and fixed carbon. Beyond its performance, this study highlights biodiesel's role as a sustainable and effective alternative to conventional, non-renewable collectors for graphite flotation, offering both efficacy and significant environmental advantages.

Introduction

Graphite is a critical industrial mineral owing to its high electrical and thermal conductivity, chemical inertness, and lubricity, making it essential in applications from refractories and foundries to lithium-ion batteries and advanced composites [1]. Surging global demand, especially from the energy storage and electric vehicle sectors, has intensified the industry to process increasingly low-grade ores with high ash and low fixed carbon. Froth flotation remains the key beneficiation method, as graphite's natural hydrophobicity enables efficient separation from hydrophilic gangue minerals like silica, alumina, and clay. Traditionally, petroleum-derived collectors such as diesel and kerosene are used, but their toxicity, poor biodegradability, and environmental risks necessitate greener alternatives [2]. Biodiesel, produced from renewable oils and fats as fatty acid methyl esters (FAMEs), has emerged as a promising eco-friendly collector, combining strong surface activity with better biocompatibility and lower toxicity [3, 4]. Studies indicate that biodiesel often competes or outperforms hydrocarbons in recovery, grade, and selectivity while reducing ecological impacts and improving safety. As sustainability becomes a priority in mineral processing, systematic evaluations of biodiesel for real ore systems are critical. This study investigates the flotation performance of a biodiesel collector for low-grade, high-ash Indian graphite ore and optimizes process parameters using Taguchi design.

Materials and Methods

Ore Sample Preparation and characterization. A low-grade graphite ore sample from eastern India, containing 89.5% ash and 8.7% fixed carbon, was used for the study. Mineralogical analysis using XRD and optical microscopy confirmed the presence of graphite along with gangue minerals such as quartz, biotite, muscovite, and kaolinite [5]. The sample was subjected to grinding to achieve the desired degree of liberation, with grind times varied according to the experimental design.

Biocollector preparation and characterization. Biodiesel was synthesized via alkaline transesterification, where triglycerides were reacted with methanol in the presence of sodium hydroxide as a catalyst. The process yielded fatty acid methyl esters (FAMEs), which were subsequently washed, dried, and used as biocollector for flotation experiments. To confirm its chemical composition, the biodiesel was characterized using FTIR for functional group identification and gas chromatography (GC) for profiling individual FAMEs. In the flotation studies, sodium silicate was employed as a depressant, with its dosage optimized through a Taguchi L_9 experimental design.

FTIR and GC analysis of collector biodiesel. The FTIR spectrum of biodiesel confirmed the formation of fatty acid methyl esters, with peaks at 3008 cm^{-1} (=C–H stretch), 2921 and 2852 cm^{-1} (–CH_2 stretch), 1742 cm^{-1} (C=O ester), 1464 and 1375 cm^{-1} (–CH_2/–CH_3 bending), 1158 cm^{-1} (–C–O–C stretch), and 720 cm^{-1} (long-chain $(CH_2)_n$) [6, 7]. These features confirm biodiesel's amphiphilic nature, where hydrophobic alkyl chains interact with graphite surfaces, while polar ester groups remain in the aqueous phase, aiding flotation. GC analysis further showed biodiesel to be rich in oleic (46.6%) and palmitic (32.2%) acid methyl esters, where oleic acid enhances hydrophobicity and palmitic acid ensures stability, together supporting its role as an effective graphite collector.

Taguchi L_9 experimental Design. A Taguchi L_9 orthogonal array was employed to study the effect of three process variables namely grind time (25, 30 and 35 min), sodium silicate dosage (1.67, 2.00 and 2.33 kg/t), and biodiesel collector dosage (2.32, 2.90 and 3.48 kg/t). The responses evaluated were weight recovery, ash content, and fixed carbon recovery of the graphite concentrate.

Flotation results and discussion. The flotation results (Fig.1) showed weight recovery ranging from 10.06% to 12.05%, with the maximum recovery achieved in Run 6 (30 min grind, 2.33 kg/t SS, 2.32 kg/t biodiesel). Higher biodiesel dosages generally improved recovery, as increased collector availability enhanced graphite surface hydrophobicity and particle-bubble attachment. Ash content varied between 2.74% and 5.00%, the latter observed at higher biodiesel dosages due to partial entrainment of gangue minerals. Fixed carbon content remained relatively consistent between 91.53 to 94.42%, with Run 1 (25 min grind, 1.67 kg/t SS, 2.32 kg/t biodiesel) offering the most optimized outcome 11.21% recovery, the lowest ash (2.74%), and the highest fixed carbon (94.42%). These conditions highlight the balance between concentrate purity and recovery efficiency.

Based on flotation results, regression models were developed in MINITAB to study the effects of grind time, sodium silicate, and biodiesel dosage. The models showed strong predictive accuracy with high R^2 values (98–99%), confirming their reliability. Weight recovery was strongly influenced by grind time and collector dosage, while ash content was moderately influenced, with lower predictive power compared to other responses. Fixed carbon recovery exhibited the best model fit, highlighting the significant role of biodiesel in improving concentrate quality.

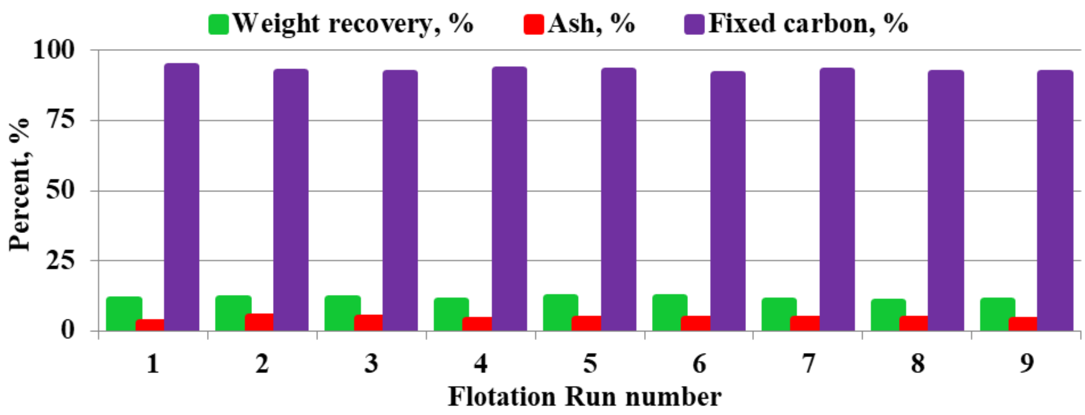

Fig. 1 Flotation Results using Taguchi L_9 design

The regression equations are given as,

Weight recovery, % = -0.367 A + 8.07 B + 6.26 C + 0.079 A*B + 0.022 A*C - 3.24 B*C. (1)

Ash, % = 0.054 A + 1.23 B + 0.5 C - 0.017 A*B - 0.008 A*C + 0.08 B*C. (2)

Fixed carbon, % = 1.23 A + 18.4 B + 44.1 C + 0.30 A*B - 0.718 A*C - 11.15 B*C. (3)

Where, A, B and C represent grind time (min), sodium silicate dosage (kg/t) and biodiesel dosage (kg/t) respectively.

These regression equations (1-3) illustrate the interactions among process parameters and their impact on graphite flotation efficiency. The accuracy of these models was further validated through actual versus predicted plots derived from the experimental data, as illustrated in Fig. 2.

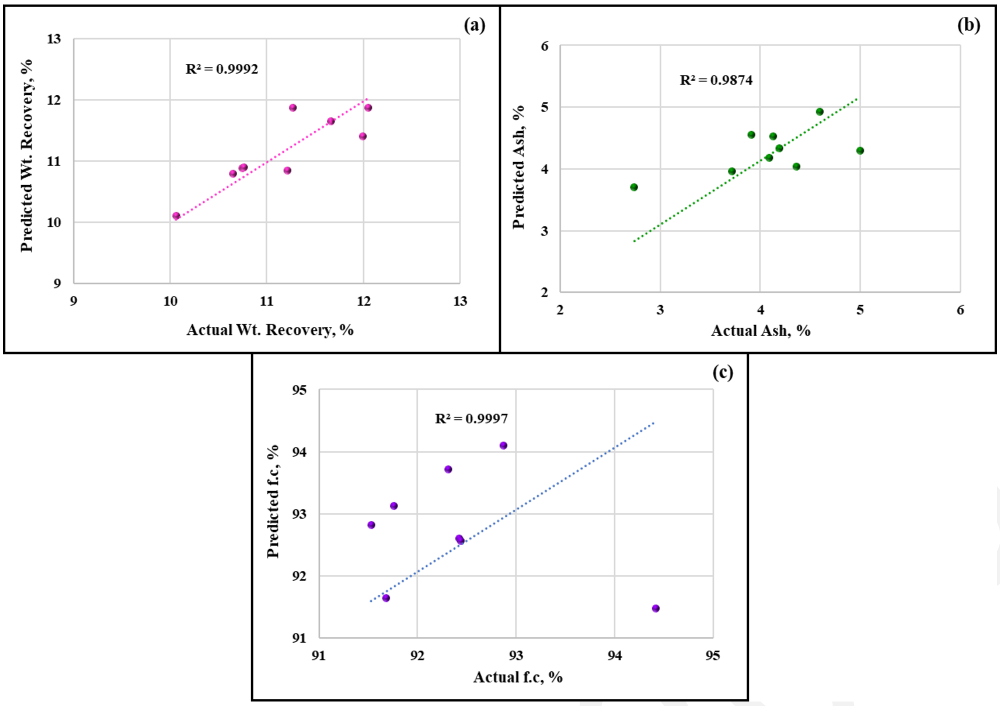

Fig. 2 Actual vs. predicted plot for responses (a) weight recovery (b) ash (c) fixed carbon

Conclusions

The study shows that biodiesel could be used as a sustainable and efficient collector for the flotation of low-grade graphite ore, yielding a concentrate with high fixed carbon recovery (94.42%), low ash (2.74%), and with weight recovery of 11.21% under optimized conditions of 25 min grind, 1.67 kg/t SS, and 2.32 kg/t biodiesel. Beyond its performance, biodiesel offers environmental advantages, being biocompatible and renewable, making it a viable replacement for conventional hydrocarbon oils such as diesel and kerosene.

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