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A COMPREHENSIVE INVESTIGATION OF PLANT SEED OIL AS GREEN COLLECTOR FOR ENHANCED FLOTATION OF NON-COKING COAL

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ABSTRACT

The shift toward sustainable industrial practices has led to a growing interest in bio-based reagents that reduce environmental impact. This study explores the application of oil extracted from custard apple (*Annona squamosa*) seeds, rich in fatty acids, as a green collector in non-coking coal flotation. The oil was obtained through solvent extraction using n-hexane and evaluated for its flotation efficiency. Using an optimized collector dosage of 12.75 kg/t and 1.33 kg/t of methyl isobutyl carbinol as frother, the process yielded a coal concentrate with 60.92% recovery, 11.16% ash content, and 54.53% fixed carbon. The gross calorific value of the cleaned coal surpassed 6700 kcal/kg, qualifying it as G2-grade coal, suitable for thermal power plants. These findings confirm the collector's effectiveness in improving separation performance and product quality. The use of custard apple seed oil, a renewable and biodegradable resource, presents a promising eco-friendly alternative to petroleum-based reagents. The work demonstrates a significant advancement in sustainable mineral processing and supports the development of green technologies in the coal industry.

Keywords: Bio-Collector, Coal Flotation, Custard Apple Seed Oil, Non-Coking Coal, Sustainable Reagent, Ash Reduction, Fixed Carbon Recovery.

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INTRODUCTION

India depends heavily on coal for its energy needs, with non-coking coal accounting for nearly 55% of the total coal consumption. Non-coking coal is crucial for energy production but typically requires beneficiation to boost its calorific value and reduce environmental impact. According to recent reports, more than 75% of India's thermal coal has an ash content exceeding 30%, contributing to particulate matter, SO_2 emissions, and lower boiler efficiency. The high ash level leads to greater emissions and a drop in gross calorific value (GCV). Such high ash levels contribute to increased emissions of particulate matter, sulphur dioxide, and other combustion-related pollutants.[1] Amid growing concerns over energy security, environmental sustainability, and climate change, India urgently needs to adopt cleaner coal practices and advanced clean coal technologies (CCT) for thermal power generation. [2] These technologies are crucial for reducing greenhouse gas emissions and ensuring the long-term viability of coal-based energy. Thermal power plants using non-coking coal are designed to operate within specific quality parameters, and any deviation can lower efficiency and output. [3] Therefore, coal beneficiation to enhance and stabilize quality has become essential for maintaining performance and meeting industry demands. Research has increasingly focused on practical ways to reduce ash-forming minerals in non-coking coal, aiming to improve its utilization and lessen environmental impact. This area of research continues to evolve with innovative solutions addressing the challenges of non-coking coal use. [4,5] Froth flotation is a widely used method to reduce ash content in fine coal. Coal's natural hydrophobicity enables it to separate easily from hydrophilic gangue minerals during flotation. This property allows for efficient recovery using frothers and non-polar collectors like diesel and kerosene[6-12], which enhance surface hydrophobicity and promote bubble attachment. However, the common use of petroleum-based collectors such as diesel and kerosene raises

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environmental concerns due to their non-biodegradable nature and carbon footprint. This has prompted a search for eco-friendly flotation reagents aligned with Sustainable Development Goal (SDG) 12 – Responsible Consumption and Production, which promotes the use of renewable resources and sustainable industrial practices. Higher-rank coals, being more hydrophobic, float more easily, while low-rank coals show poorer floatability due to surface chemistry. This highlights the importance of thoroughly characterizing each coal type and adjusting flotation parameters accordingly. In particular, non-coking coals often perform poorly in flotation due to the presence of hydrophilic functional groups on their surfaces, which impede attachment to air bubbles.[13,14] Recent studies have demonstrated that this limitation can be overcome using plant-derived oils as bio-collectors, which alter surface chemistry while being biodegradable and sustainable.[15,16] Recent advances reveal that even low-rank, difficult-to-float non-coking coals can be effectively separated using specialized reagents or surface chemistry modification.[17] Notably, non-traditional flotation agents, particularly plant-derived oils, have shown promise in improving the recovery of high-ash non-coking coal. These bio-collectors, derived from renewable biomass, offer an eco-friendly alternative to conventional reagents, supporting green technology and circular economy goals. This study explores the use of plant seed-based oil (CAO) as a bio-collector for non-coking coal flotation. It evaluates the oil's properties, its flotation performance, and its potential to reduce the environmental impact of coal beneficiation while enhancing coal quality. By integrating renewable biomass-derived reagents into coal processing, this work advances clean coal technologies and supports circular economy principles crucial for transitioning toward sustainable energy systems.

EXPERIMENTAL

Materials and Methods

Custard apple seeds were sourced from a local greengrocer in India, and the oil was extracted using the Soxhlet extraction method with *n*-hexane as the solvent. This bio-based oil served as the collector in the flotation of non-coking coal. Methyl Iso-Butyl Carbinol (MIBC) was employed as the frother during the flotation process. The non-coking coal sample was obtained from an operational coal mine in Jharkhand, India, and was subsequently ground to the desired particle size using a ball mill.

Soxhlet Extraction Method

The oil from custard apple seeds was extracted using the Soxhlet method with n-hexane as the solvent. This method is preferred because of its ability to perform continuous extraction, where the solvent is continuously cycled through the sample, allowing for efficient extraction over a period of time. This method is also advantageous because the solvent is recycled, which is both environmentally and economically beneficial. The seeds were washed, dried, and blended to the required size before being loaded into the extractor. After extracting the oil (CAO), the solvent was removed using a rotary evaporator.

Characterization Studies

A comprehensive characterization of the oil extracted from non-edible seeds was carried out using a combination of analytical methods, including Fourier Transform Infrared Spectroscopy (FTIR) and Gas Chromatography Mass Spectrometry (GC-MS). FTIR was employed to qualitatively identify and evaluate the functional groups present in CAO, while GC-MS provided a quantitative analysis of the individual free fatty acids, enabling precise determination of the CAO's compositional profile.

Flotation Studies

Non-coking coal sample was subjected to laboratory-scale flotation tests, varying key parameters such as grind time, collector CAO dosage, MIBC dosage, and aeration rate. Experiments were conducted using a Denver D12 flotation cell, with ball mill output processed in a 2L cell at 800 RPM. CAO was used as the collector and MIBC as the frother in the rougher stage. After conditioning, aeration introduced fine air bubbles into the coal slurry, enabling coal fines made hydrophobic by CAO to attach to the bubbles. The resulting froth, enriched with coal fines, was skimmed off, while gangue settled as primary tailings (PT). The rougher concentrate was cleaned in five additional stages (Fig.-1) to remove ash-forming minerals, yielding a final concentrate (FC) with high carbon recovery. All flotation products were dried and analyzed for ash content and fixed carbon.

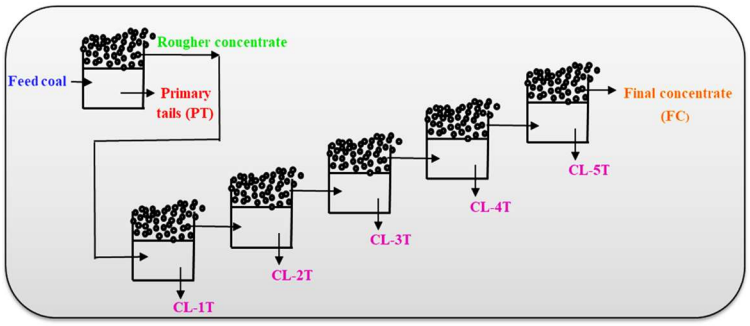

Fig.-1: Schematic Representation of Various Stages of the Flotation Process

RESULTS AND DISCUSSION

FTIR Analysis of Collector CAO

The FTIR spectrum of CAO (Fig.-2), evaluated for its role as a green collector in coal flotation, reveals the presence of key functional groups that contribute to its surface-active behavior. Distinct absorption bands observed at 2921 cm^{-1} and 2854 cm^{-1} are attributed to the asymmetric and symmetric stretching vibrations of $–CH_2$ and $–CH_3$ groups, respectively, which are characteristic of long-chain aliphatic hydrocarbons typically found in fatty acids. A strong peak at 1743 cm^{-1} is attributed to the stretching vibration of carbonyl (C=O) groups. The band observed at 1464 cm^{-1} is associated with the bending vibration of methylene groups, further supporting the existence of saturated hydrocarbon chains. Additionally, the peak at 1162 cm^{-1} arises from C–O stretching vibrations, typically found in esters or alcohols.[18] These spectral features confirm that CAO contains long-chain fatty acids, imparting both hydrophobicity and surface activity. These properties are essential for enhancing coal particle floatability, thereby validating the suitability of CAO as a sustainable and effective bio-collector in the flotation of non-coking coal.

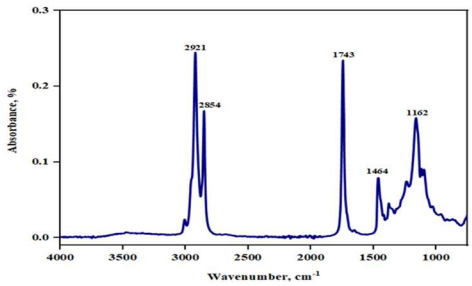

Fig.-2: FTIR Spectrum of Collector CAO

GC-MS Analysis

GC-MS analysis of collector CAO reveals a complex blend of fatty acids crucial to its role as a bio-based flotation agent (Fig.-3). Major components include oleic, linoleic, linolenic, stearic, and palmitic acids, along with smaller amounts of myristic, arachidic, and lignoceric acids. This mix of saturated and unsaturated fatty acids provides a balance of hydrophobicity and surface activity, enhancing coal floatability. FTIR data support these findings, showing characteristic peaks for fatty acid esters C–H stretching at 2921 and 2854 cm^{-1}, a strong ester carbonyl at 1743 cm^{-1}, and C–O stretching at 1162 cm^{-1}. These features confirm CAO's richness in surface-active compounds, highlighting its effectiveness as a sustainable collector for coal flotation.

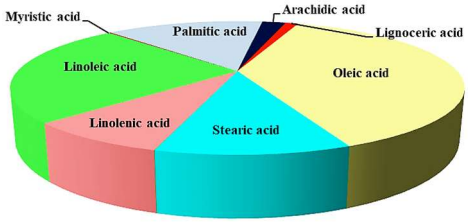

Fig.-3: GC-MS Analysis of Collector CAO

Flotation Results

Flotation tests were conducted under controlled conditions, with variations in grind time, collector dosage, frother dosage, and airflow rates for optimizing the non-coking coal flotation process. This section details the beneficiation process by systematically evaluating the effect of variation of grind time, collector and frother dosages, airflow rates, and multi-stage cleaning on flotation efficiency. Additionally, the impact of implementing a five-stage cleaning process on the rougher concentrate was assessed. The effects of these parameters on flotation performance provide valuable insights for enhancing the overall efficiency of non-coking coal beneficiation.

Optimization of Grind Time

The run-of-mine coal sample was ground in a ball mill for 20, 30, and 40 minutes to achieve size reduction and mineral liberation. The milled products were then subjected to flotation using the bio-collector CAO to assess the effect of grind time on flotation performance.

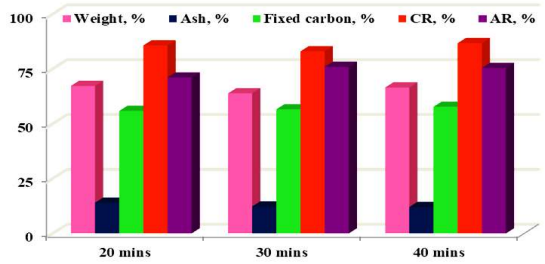

Fig.-4: Effect of Grind Time on Non-Coking Coal Flotation

Figure-4 shows the results after five cleaning stages, presenting weight recovery, ash content, fixed carbon, combustible recovery (CR), and ash rejection (AR) for each grind time. Among the three, the 40-minute grind yielded the best results, with the lowest ash content (11.99%), highest fixed carbon (57.75%), and highest combustible recovery (86.75%). In comparison, the 20- and 30-minute grinds showed ash contents of 13.96% and 12.23%, fixed carbon values of 55.83% and 56.59%, and combustible recoveries of 85.71% and 83.03%, respectively. Therefore, 40 minutes was selected as the optimal grind time. The multi-stage cleaning process further improved the coal concentrate's quality and carbon recovery. Final tests were conducted at the optimized 40-minute grind using roughing followed by five cleaning stages, progressively enhancing concentrate purity. The flotation performance of CAO was evaluated based on recovery rates and concentrate quality in terms of fixed carbon and ash content at each stage.

Influence of collector CAO

The effect of collector CAO on the flotation efficiency of non-coking coal was investigated to enhance recovery. Rougher flotation was conducted on 300 g of coal at 800 RPM impeller speed, using 5 ml of CAO without any frother, and an aeration rate of 2.5 lpm. Froth was collected, and the resulting rougher concentrate was cleaned and analyzed for ash content and fixed carbon. Both flotation concentrates and tailings were evaluated for weight recovery%, ash%, fixed carbon%, combustible recovery%, and ash rejection%. The study assessed whether CAO effectively induced coal hydrophobicity. As shown in Fig.-5, the primary tailings showed about 21% weight recovery with 70% ash, while cleaner tailings reduced to 7–1% with 59–25% ash. The final concentrate yielded 57% with 11.38% ash and 52.34% fixed carbon, up from 15.25% in the primary tailings. Combustible recovery and ash rejection in the final concentrate were 79.95% and 79.07%, respectively. Increased collector dosage improved yield, fixed carbon, and combustible recovery, though excessive CAO reduced froth stability and negatively affected valuable mineral recovery. Hence, frother was introduced to improve froth stability.

Influence of Airflow Rate

The influence of airflow rate along with CAO dosage and MIBC dosage on the flotation efficiency of non-coking coal was studied for enhanced recovery. The rougher flotation was carried out with 4.5ml of the collector CAO, 0.45ml of frother at a 1.5lpm aeration rate. The froth was collected, and this rougher concentrate was further cleaned, and the final concentrate was analyzed for its ash content and fixed carbon.

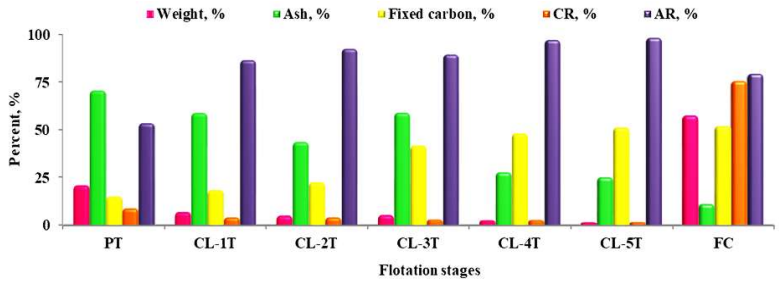

Fig.-5: Effect of Collector CAO on Flotation

The flotation concentrate and tailing products were analyzed to determine weight recovery%, ash%, fixed carbon%, combustible recovery%, and ash rejection%. In order to evaluate the effectiveness of the airflow rate, the collector CAO was reduced to 4.5ml, and a frother of 0.45ml was introduced to stabilize the froth. The results, as depicted in Fig.-6, shows that the weight recovery with ash content of the primary tailings amounted to approximately 16% with 80%. Furthermore, the cleaner tailings decreased significantly to around 9-2% with 70-3%, whereas the final concentrate was 62% with ash of 11.52%. The fixed carbon in the primary tailings was 12.02% and increased to 53.92% in the final concentrate. The combustible recovery in the final concentrate was 80.15% with the ash rejection of 77.75%.

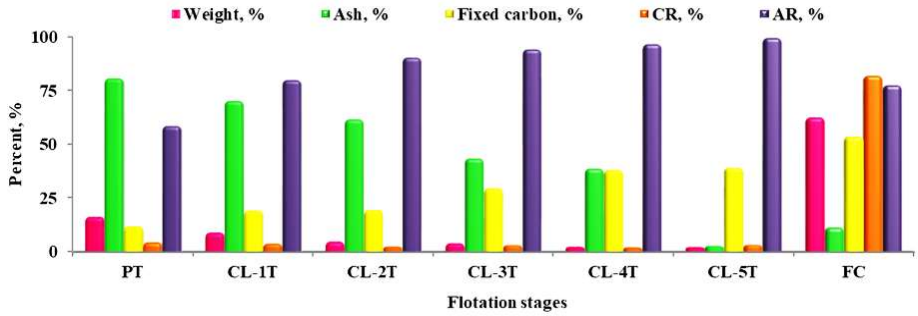

Fig.-6: Effect of Airflow Rate on Flotation

Influence of Frother MIBC

The study examined the effect of frother on non-coking coal flotation efficiency to enhance recovery. Rougher flotation was performed by adding 4.5 ml CAO, 0.5 ml MIBC, and 1.5 lpm aeration. The resulting froth was collected, cleaned, and analyzed for ash content and fixed carbon. Recovery parameters, including weight recovery, ash content, fixed carbon, combustible recovery, and ash rejection, were evaluated for both concentrate and tailings. The collector CAO and airflow rate were kept constant to evaluate the effectiveness of the frother. As shown in Fig.-7, primary tailings had about 18% weight with 84% ash, which reduced to 7–3% and 75–29% in cleaner tailings.

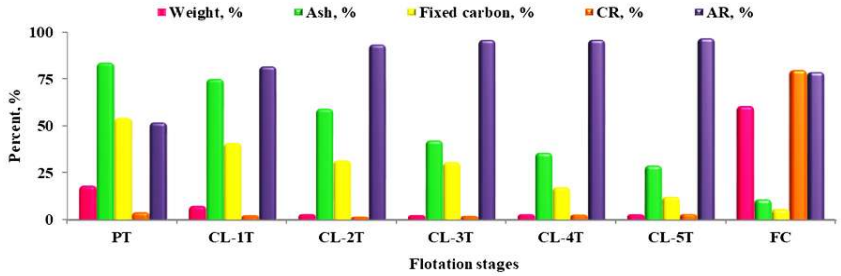

Fig.-7: Effect of Frother MIBC on Flotation

The final concentrate accounted for ~60% weight with 11.16% ash. Increasing frother led to higher ash in primary tailings, indicating better rejection of ash-forming minerals. Fixed carbon rose from 5.99% in primary tailings to 54.53% in the final concentrate. Combustible recovery reached 82.19% with 79.72% ash rejection. Figure-7 also shows the lowest fixed carbon (5.99%) and highest ash (84.09%) in primary tailings, with minimal combustible recovery (4.35%) and peak ash rejection (79.79%), reflecting optimal

clean coal yield. These results confirm that the combination of 12.75 kg/t collector and 1.33 kg/t frother yielded the best performance.

CONCLUSION

This study demonstrates the feasibility of using custard apple (*Annona squamosa*) seed oil (CAO), rich in fatty acids and extracted using n-hexane, as a sustainable bio-collector in the flotation of non-coking coal. At an optimized dosage of 12.75 kg/t CAO with 1.33 kg/t MIBC as frother, the process achieved a clean coal concentrate with 60.92% weight recovery, 11.16% ash, and 54.53% fixed carbon. The high gross calorific value (>6700 kcal/kg) qualifies the product as G2 grade, suitable for thermal power generation. The results signify improved combustion efficiency and lower environmental impact due to reduced ash and enhanced fixed carbon content. The successful substitution of petroleum-based collectors with biodegradable, renewable CAO underscores its role in advancing sustainable mineral processing. These findings align with SDG 12: Responsible Consumption and Production, promoting eco-friendly practices in energy-intensive sectors. Further studies are recommended to explore scalability, cost-effectiveness, and broader applicability of plant-derived collectors in industrial coal beneficiation.

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CONFLICT OF INTERESTS

The authors declare that there is no conflict of interest.

AUTHOR CONTRIBUTIONS

The present work is related to *SDG-12: Responsible Consumption and Production*. All the authors contributed significantly to this manuscript, participated in reviewing/editing and approved the final draft for publication. The research profile of the authors can be verified from their ORCID ids, given below:

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