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Optimizing Dry Beneficiation of Palygorskite through Air Classification for Improved Grade and Recovery

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Abstract

Palygorskite (PAL), also known as attapulgite, is a clay mineral prized for its nanorod-like silicate structure and fibrous morphology. The traditional PAL purification methods often involve wet gravity separation techniques such as sedimentation and screening, which require significant water usage and pose sustainability challenges, especially in the water-scarce regions. This work introduces a novel, environmentally sustainable dry beneficiation method for PAL. A large PAL sample with 41.7% content and 10% moisture was crushed, ground using a pin mill, and classified into three particle size fractions: $-0.088 \text{ mm} + 0.066 \text{ mm}$, $-0.066 \text{ mm} + 0.044 \text{ mm}$, and -0.044 mm . These fractions were treated with an air classifier. A Box-Behnken experimental design was employed to investigate the effects of particle size, shutter opening, and motor speed on the classification efficiency. The optimal parameters for grade were 400 rpm motor speed, shutter opening of 1 mm, and feed size of $-0.066 \text{ mm} + 0.044 \text{ mm}$. For the recovery, the optimal conditions were 1200 rpm motor speed, shutter opening of 2.5 mm, and feed size of -0.044 mm . The most favorable balance of grade (67.8%) and recovery (53.2%) was achieved with a motor speed of 1200 rpm, shutter opening of 4 mm, and feed size of $-0.066 \text{ mm} + 0.044 \text{ mm}$. The work concludes that air classification significantly enhances the PAL beneficiation process, with a 50% increase in grade, and recommends exploring the low shear grinding techniques for further improvement.

1. Introduction

Palygorskite (PAL), also referred to as attapulgite, is a type of clay mineral that is highly esteemed for its unique fibrous shape and silicate structure, resembling nanorods. The high surface area, significant adsorption capacity, and thermal stability of this material make it essential in various industrial applications including adsorbents, catalysts, and pharmaceuticals [1–8]. Nevertheless, the task of processing and enhancing PAL is arduous because of its intricate composition, and the existence of impurities that can impact its functionality [9,10].

The traditional PAL beneficiation commonly employs the wet gravity separation methods such as sedimentation and screening. While these methods are effective, they impose a significant environmental burden, especially in the regions

with limited water resources [11–13]. The substantial water demands give rise to concerns regarding sustainability, increase operational expenses, and restrict the viability of extensive processing in the arid regions [14]. Therefore, it is crucial to develop alternative beneficiation methods that are more sustainable, and reduce or eliminate the need for water.

The recent studies have focused on altering the chemical composition, and enhancing the functionality of PAL, thereby expanding its potential uses in the areas such as environmental cleanup, catalytic processes, and the chemical industries [6,10,12,15–21]. For example, Yang and others demonstrated the PAL's effectiveness in adsorbing various ionic dyes, showcasing its versatility in environmental cleanup [16].

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Similarly, Chen and others developed a biosurfactant-modified PAL dispersant for oil spill mitigation, highlighting its environmental applications [15]. Cheng and others further explored the modified PAL's benefits in improving poultry health, indicating its potential in the agricultural sectors [17]. Finally, Lu and others investigated the effect of oxalic acid leaching on PAL, advancing its functionality for the industrial applications. These studies underscore the ongoing advancements in enhancing the PAL's utility across various fields [20]. In addition, the emphasis on sustainable processing has led to investigations into the alternative methods of improving the efficiency and sustainability of the PAL processing. These methods include low-energy milling and optimizing air classification parameters [2].

PAL clay contains other non-clay minerals (quartz, calcite, gypsum etc.), which are formed during its formation [9]. To remove the impurities, PAL is processed by many approaches including extrusion, acid activation and sedimentation [2,7,9,22]. It is reported in the literature that the acid activation can eliminate the impurities, increase its area, and pore volume, as well as the number of adsorption sites by disaggregation of its particles [22]. By sedimentation, after dispersing the PAL into slurry, non-clay minerals are settled down at the bottom by the gravitational force. The settled materials can then be removed by wet screening, sedimentation, hydro cyclone, or other wet physical separation methods [9].

The study conducted by Xavier and others examined the impact of acid treatment on PAL clay by using hydrochloric acid (HCl) at different concentrations and durations. The objective of the research work was to augment the clay's adsorption capacity by amplifying its surface area through acid activation. The findings indicated that the elevated acid concentrations (6 Mol/L) had a significant impact on the specific surface area of PAL, as well as causing noticeable alterations in its chemical composition, as verified by the XRF and XRD analyses. The study emphasized the capacity of acid treatment to alter and enhance the adsorption characteristics of PAL [22].

The study conducted by Nefzi and others involved the obtaining of PAL clay from diatomite using a chemical modification procedure. PAL was obtained by subjecting diatomite, a naturally occurring sedimentary rock, to chemical treatment, aimed at modifying its composition and structure. Subsequently, the altered PAL was

examined to assess its capacity to adsorb heavy metals, namely Cu^{2+} and Ni^{2+} , from aqueous solutions. The findings indicated that the synthesized PAL exhibited markedly superior adsorption capabilities in comparison to the untreated diatomite, rendering it an exceptionally efficient substance for the elimination of heavy metals from water [23].

The efficiency of PAL dispersion in the dry processing techniques, such as ball grinding, stone milling, and extrusion is affected by the mechanical forces and the duration of treatment. In their study, Jin and others discovered that extended ball grinding can reduce the size of particles. However, this process can also cause a transformation in the crystalline structure of PAL, converting it into an amorphous state [24]. Moreover, excessive grinding can result in re-aggregation, and cause a significant damage to the rods. Liu and others found that stone milling can partially break apart the PAL crystals, but prolonged milling can cause a significant damage to the rod structure, reducing its effectiveness [25]. According to Wang, extrusion causes a partial separation of the crystal bundles, which enhances the hydration and expansion of particles [26]. However, complete dispersion may not be achieved. These findings emphasize the need for a delicate equilibrium in dry processing to improve the properties of PAL without jeopardizing its structural integrity.

Air classification, a method of processing without the use of liquid, shows a great potential as an alternative approach for the PAL beneficiation. Air classification, which employs an airstream to segregate particles according to their size and density, obviates the necessity for water, rendering it a more ecologically viable alternative [27]. Nevertheless, the utilization of this technique on PAL is relatively new, and necessitates meticulous adjustment of the operational factors including feed particle size, classifier motor speed, and shutter opening in order to optimize both the quality and quantity of the output [28,29].

Sun and others explore the use of hydrocyclones for separating PAL clay, aiming to enhance the efficiency and reduce costs compared to the traditional methods. The work investigates the effects of feed concentration, feeding pressure, and underflow port diameter on the separation performance using a two-stage hydrocyclone separation. The results show that the purity of Palygorskite increased from 45.1% to 64.2%, with a high recovery rate of 95.9% [30]

Air classification provides numerous benefits in the process of the PAL beneficiation. The dry process obviates the necessity of water and the subsequent drying procedures, thereby diminishing the environmental repercussions of the PAL processing [31]. In addition, air classification can be combined with the other dry processing methods such as grinding to improve the overall effectiveness of the beneficiation process. Previous studies have demonstrated that the integration of air classification with the grinding techniques such as pin milling can result in substantial enhancements in both the purity and the recovery of PAL [1].

This work introduces a novel approach to the dry beneficiation of PAL by employing air classification, which offers significant advancements over the traditional wet methods. Unlike the previous research works that primarily focused on the wet gravity separation techniques, this work leverages a Box-Behnken experimental design to systematically investigate the effects of particle size, shutter opening, and classifier motor speed on the efficiency of air classification. The novelty of this research work lies in its focus on optimizing these parameters to achieve a high-quality PAL grade and recovery rate using a dry process, addressing the sustainability challenges associated with the water-intensive methods. The work's innovative approach not only enhances the classification efficiency but also explores alternative grinding techniques that minimize shear forces, presenting a comprehensive solution

to improve both the quality and quantity of the output. This work is crucial in advancing sustainable beneficiation practices, and offers valuable insights for future research and industrial applications in the water-scarce regions.

2. Materials and Methods

2.1. Sample preparation

The raw palygorskite (PAL) with a moisture content of 10% was obtained from Sindh, Pakistan. The Jones Riffle was utilized to perform systematic sampling of the feed material. Afterwards, the feed underwent comminution using a Roll Crusher (FOOTE BROS 212A-213), and was then sieved to obtain a particle size fraction of 1 mm. This allowed for more accurate separations during the laboratory-scale processes.

2.2. Particle size distribution

A systematic sampling of the feed material intended for the roll crusher was conducted to analyze its particle size distribution. Initial observations revealed that the largest particle size in the feed material was smaller than 6.9 mm. In order to provide a clearer understanding of the distribution of particle sizes, a graph was created to illustrate the correlation between the particle size and the corresponding percentages of material that passed through and was retained. Figure 1 displays the particle size distribution curve obtained as a result.

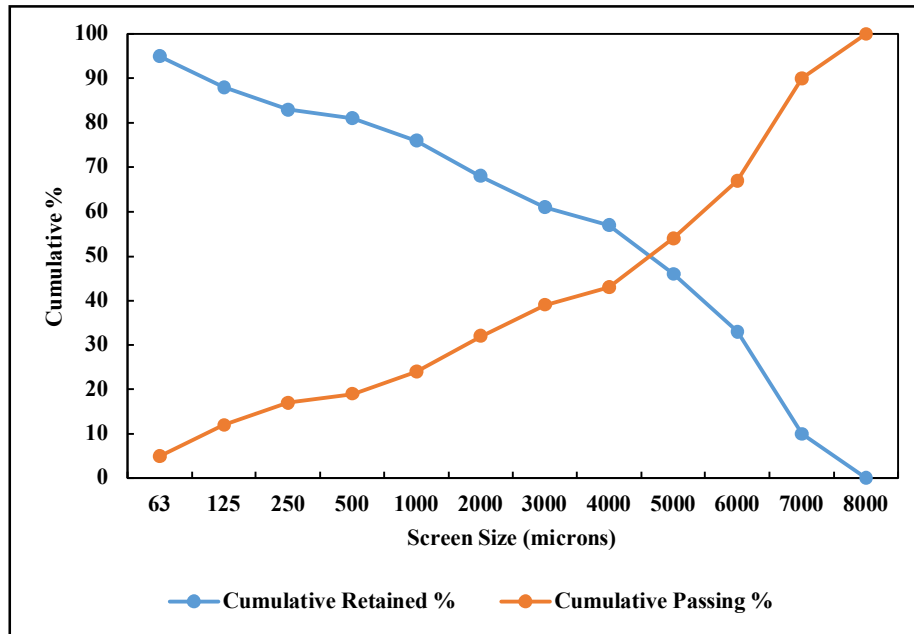


Figure 1. Particle size distribution of the bulk sample as received.

2.3. Characterization of raw feed

A sample was prepared from the raw PAL for detailed characterization. The X-ray diffraction (XRD) analysis was performed at the COMSATS University of Islamabad, Lahore Campus using a Philips PANalytical X'Pert Powder diffractometer. The analysis utilized Cu-K α radiation with a wavelength of 1.5406 Å, covering a 2 θ range from 4° to 70°. The measurements were taken with a step size of 0.02°, and a time per step of 30 seconds.

2.4. Methodology

The bulk sample of PAL was initially subjected to crushing using a roll crusher with a processing capacity of 2 tons per hour. This crushing stage resulted in a product with a maximum particle size of 1 mm. Subsequently, a 30 kg sample of

PAL was ground using a DERBY MRL-16 pin mill. The ground PAL was then sieved to obtain three distinct size fractions: -0.088 mm + 0.066 mm, -0.066 mm + 0.044 mm, and -0.044 mm.

The sieved fractions were further processed using a KINCSTON-98 air classifier to separate the particles into coarse and fine categories. The air classification was conducted under varying conditions, including different feed sizes (-0.088 mm + 0.066 mm, -0.066 mm + 0.044 mm, and -0.044 mm), motor speeds (400, 800, and 1200 rpm), and shutter openings (1, 2.5, and 4 mm). The products obtained from the air classification were then analyzed to assess their individual properties. A flowchart illustrating the experimental design and workflow is provided in Figure 2.

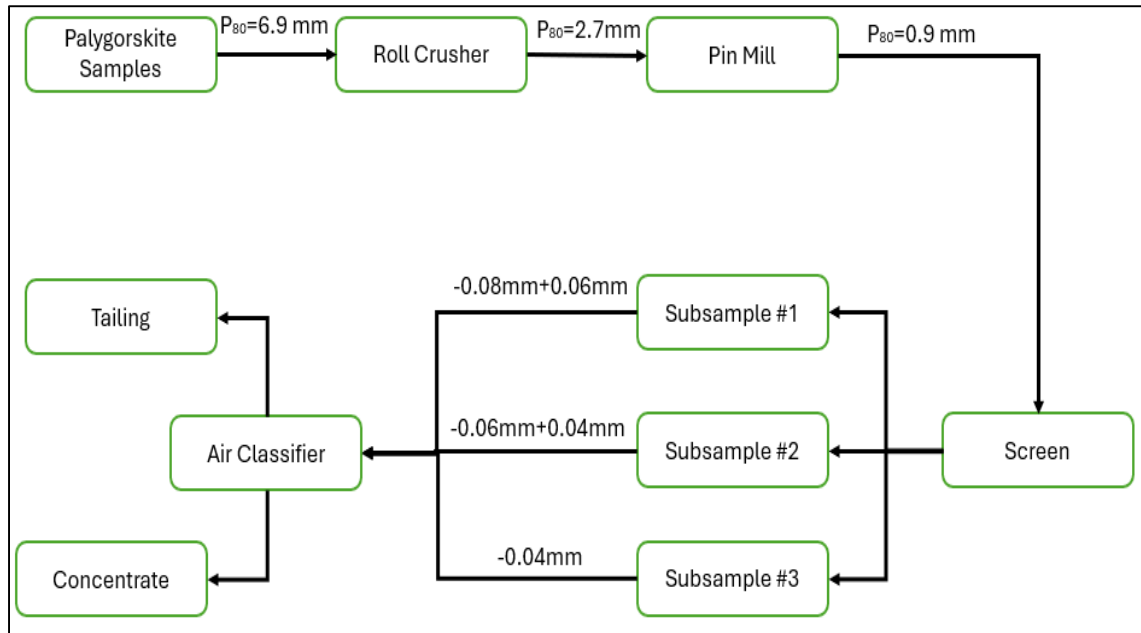


Figure 2. Flowchart of processing the PAL.

2.5. Model development and validation

In this work, three primary factors—feed size, motor speed, and shutter opening—were identified as having significant impacts on the air classification performance. A Box-Behnken experimental design was employed to explore these effects systematically. The experimental design was developed using Minitab-17 software, applying Response Surface Methodology (RSM) to generate the experimental plan efficiently. This design was selected for its ability to minimize the

number of experimental runs, while still capturing the complex interactions between the factors.

2.5.1. Experimental design parameters

Table 1 presents the levels of the three factors (motor speed, feed size, and shutter opening) used in the experimental design [32]. Each factor was tested at three levels (low, center, and high), and a total of 30 experimental runs were conducted to ensure sufficient repeatability and reliability of results.

Table 1. Coded values for the Box-Behnken design.

Variables	Symbols	Coded variable level		
		Low	Centre	High
Speed (rpm)	A	400	800	1200
Feed size (microns)	B	44	66	88
Shutter opening (mm)	C	1	2.5	4

The experimental data was used to develop regression models that describe the relationship between the air classifier's operating parameters, and the resulting recovery, and grade of PAL. The following equations were generated to predict the

recovery and clay content percentages based on the varying experimental conditions. Equations 1 and 2 provide a comprehensive understanding of how each variable influences the overall performance of the air classifier.

$$Recovery\% = 125.1 - 0.0163 \text{ speed} - 49.2 \text{ shutter opening} - 1.477 \text{ size} + 0.02333 \text{ speed} \times \text{size} + 0.498 \text{ shutter opening} \times \text{size} \tag{1}$$

$$Clay\% = -124.0 - 0.0436 \text{ speed} - 21.83 \text{ shutter opening} + 6.57 \text{ size} - 0.04686 \text{ size} \times \text{size} + 0.02338 \text{ speed} \times \text{shutter opening} \tag{2}$$

2.5.2. Model verification and ANOVA

To verify the validity of the model, each experiment was repeated twice to ensure the reliability of the results. ANOVA (Analysis of Variance) was performed to identify the significant factors and interactions. The ANOVA results indicated that the motor speed and shutter opening had the most substantial effect on the recovery, while feed size played a critical role in determining the final grade. The interaction between motor speed and feed size was also found to be significant, particularly in influencing the recovery rates. Residual analysis confirmed that the models adequately fit the data, with residuals displaying normal distribution and homoscedasticity, further validating the model's accuracy.

2.5.3. Significance of factors and interaction effects

The ANOVA revealed that both the motor speed and shutter opening were highly significant ($p < 0.05$) in influencing the recovery, with feed size having a notable effect on the grade. The interaction between feed size and motor speed was particularly important for the fine fractions, where higher motor speeds increased recovery, but with diminishing returns at extreme settings due to particle misclassification. The optimal settings for both the grade and recovery were determined using the desirability function,

achieving a maximum recovery at a motor speed of 1200 rpm, a shutter opening of 2.5 mm, and a feed size of -0.044 mm.

3. Results and Discussions

3.1. XRD analysis

X-Ray Diffraction (XRD) is an essential analytical method used to identify minerals. The product of the pin mill was analyzed using XRD. The XRD analysis of the pin mill product identified the predominant minerals as calcite, quartz, non-tronite, and PAL, with smaller quantities of gypsum and montmorillonite also detected.

Figure 3 shows the XRD pattern of the product obtained from the pin mill. The prominent peaks are of the primary minerals that have been identified. More precisely, the peaks corresponding to quartz are especially prominent at 2θ values approximately equal to 20.8° , 26.6° , and 36.5° , which aligns with the standard peaks for quartz. Furthermore, the presence of calcite is clearly indicated by the prominent peaks observed at 29.4° and 39.5° in the sample.

Non-tronite, a specific type of clay known as smectite, shows distinct peaks at approximately 8.4° and 19.7° , whereas PAL can be identified by peaks at around 34.6° and 42.6° . Gypsum is identified by a prominent peak at approximately 11.6° , while montmorillonite exhibits peaks at around 8.4° and 42.6° .

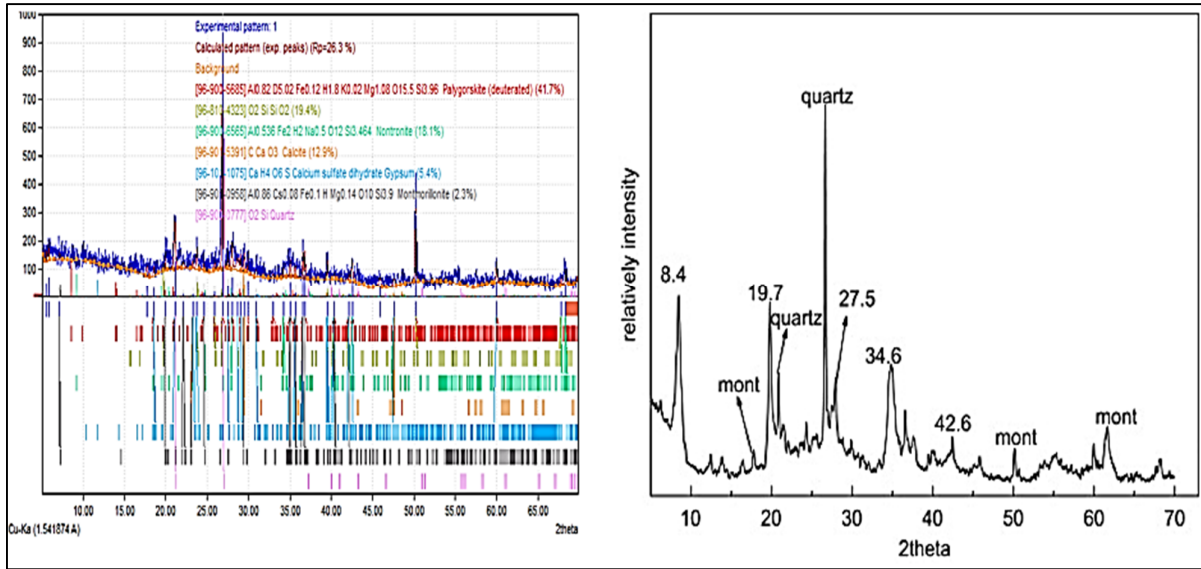


Figure 3. XRD of pin mill product.

Figure 3's XRD pattern unambiguously demonstrates that the pin mill product comprises a blend of these minerals, with quartz and calcite prevailing as the primary phases. The presence of non-tronite, PAL, gypsum, and montmorillonite, even in small amounts, emphasizes the intricate mineral composition of the raw material.

An in-depth characterization is crucial for comprehending the composition of the feed material prior to beneficiation. This enables a comparison between the results obtained and those of the beneficiated Pal, thus facilitating an evaluation of the efficacy of the beneficiation process in eliminating undesirable mineral impurities.

3.2. Effects of variables on air classifier

3.2.1. Effect of feed size on air classification efficiency

The feed size significantly impacts both recovery and grade, as shown in Figure 4. Larger particles such as the 88-micron fraction exhibit more consistent recovery rates (33.03% to 57.94%), which suggests that they are less affected by changes in the classifier settings. This stability arises because larger particles possess greater inertia, allowing them to resist aerodynamic forces and settle more predictably. In contrast, the 66-micron fraction displayed a wider range of recovery values (2.95% to 63.64%),

indicating that the medium-sized particles are more sensitive to variations in motor speed and shutter opening.

Larger particles are less influenced by airflow because their higher mass gives them more stability within the classifier. The aerodynamic forces acting on medium and smaller particles (44 microns) are stronger, making them more prone to staying suspended in the air stream, or being carried out of the classification zone. This is why smaller particles exhibit more variability in the recovery rates, as seen with the 44-micron fraction (2.13% to 55.84%).

The grade percentages also reflect the influence of feed size. Larger particles (88 microns) yield grades between 48.2% and 55.4%, while medium (66 microns) and fine particles (44 microns) achieve higher grades under optimal conditions, reaching up to 65.9% and 65.7%, respectively. This suggests that smaller particles are easier to purify because; they are more readily separated from impurities, provided that classifier settings are fine-tuned.

The higher grade for smaller particles can be attributed to their ability to remain suspended longer, giving the classifier more time to separate the impurities. However, achieving a consistent recovery for these particles requires precise control of airflow, motor speed, and shutter opening.

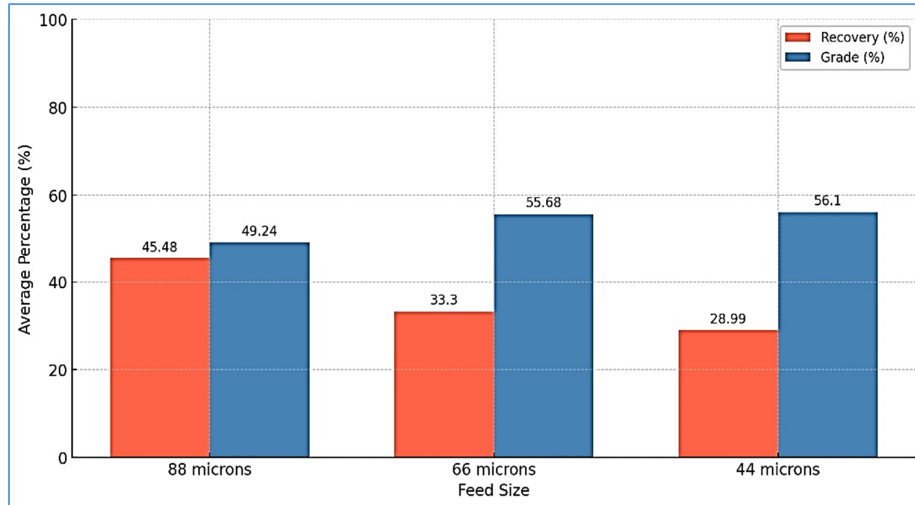


Figure 4. Average recovery and grade percentages by feed size.

3.2.2. Impact of motor speed on product recovery

Motor speed plays a key role in the recovery of particles during air classification. As seen in Figure 5, at the low speed of 400 rpm, the centrifugal force is insufficient to separate larger particles effectively. The 88-micron fraction shows a lower recovery at this speed. As motor speed increases to 800 rpm, the recovery improves across all feed sizes, with medium-sized particles showing the greatest stability. The highest recovery rates were observed at 1200 rpm for the smallest particles (44 microns), as the

increased speed provides enough force to overcome the aerodynamic drag.

At low motor speeds, larger particles do not receive enough centrifugal force to be separated efficiently. The finer particles, on the other hand, benefit from higher speeds that generate a sufficient airflow to carry them into the classifier’s collection zone. However, beyond 1200 rpm, the recovery for larger particles may decrease as excessive force causes them to escape classification, while finer particles continue to be captured more effectively.

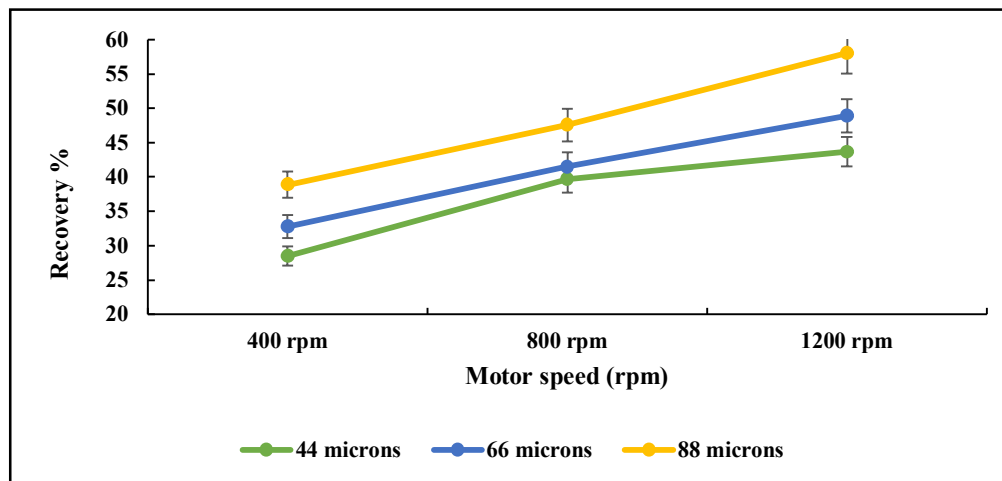


Figure 5. Impact of motor speed on the product recovery.

3.2.3. Influence of shutter opening on particle separation

Shutter opening affects the airflow through the classifier, influencing both the recovery and grade. A narrower shutter opening (1 mm)

restricts airflow, leading to a lower recovery for all feed sizes, particularly fine particles, as presented in Figure 6. As the opening increases to 2.5 mm, the recovery improves significantly, especially for medium-sized particles (66

microns). However, at the widest opening (4 mm), fine particles show a better recovery, while the recovery of larger particles becomes more variable.

A narrow opening restricts the airflow, which hinders the separation of finer particles. These particles require sufficient airflow to be carried

into the correct classification zones. In contrast, a wide shutter opening generates more airflow, improving the recovery for finer particles, but potentially causing larger particles to be swept out of the classification zone due to an excessive airflow velocity.

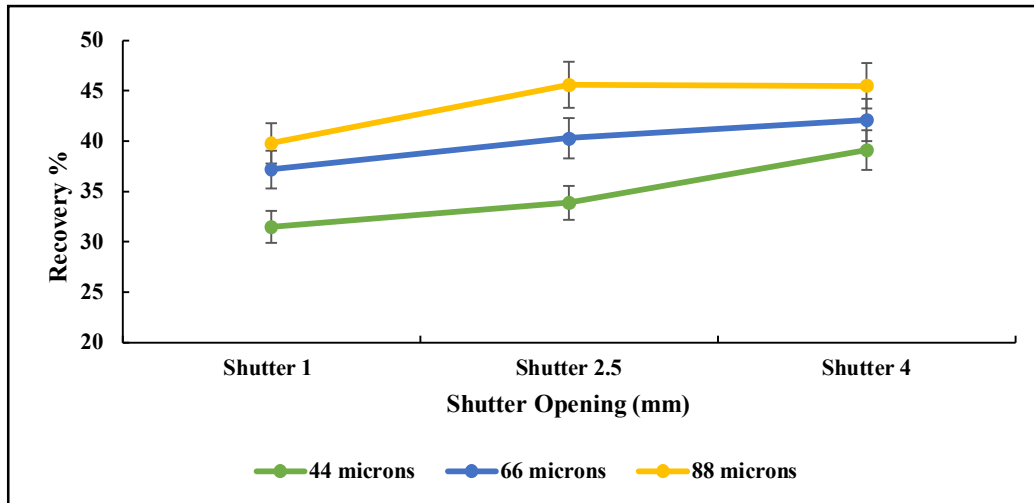


Figure 6. Influence of shutter opening on the particle recovery.

3.3. Significant interaction effects using 3D response plots

The interaction between motor speed and shutter opening was analyzed using 3D response plots (Figure 7), which visualize how these two factors jointly influence recovery and grade. The interaction is particularly significant for finer particles. As motor speed increases, recovery improves, but when combined with wider shutter openings, the benefits diminish due to the excessive force pushing the particles beyond the classifier’s collection zone.

The interaction between motor speed and shutter opening can be explained by the delicate balance required between airflow and centrifugal force. Higher speeds increase the force exerted on the particles, which is necessary for a fine particle separation. However, if the airflow becomes too strong (due to wider shutter openings), it can disrupt the classification of larger particles by sweeping them away before they can settle. This

explains why recovery improves up to a point, beyond which further increases in speed and shutter opening lead to the diminishing returns.

The grade also follows a non-linear interaction between these two factors. Optimal conditions for maximizing grade occur when motor speed is high (around 1200 rpm), and shutter opening is moderate (2.5 mm). Beyond these settings, grade percentages decrease, likely due to the misclassification of particles caused by an excessive airflow.

A high motor speed combined with a moderate shutter opening creates the optimal balance between the particle recovery and the separation from the impurities. However, when either factor exceeds its optimal range, the airflow becomes turbulent, leading to misclassification, particularly for finer particles. This is why grade decrease after a certain point despite higher motor speeds or larger openings.

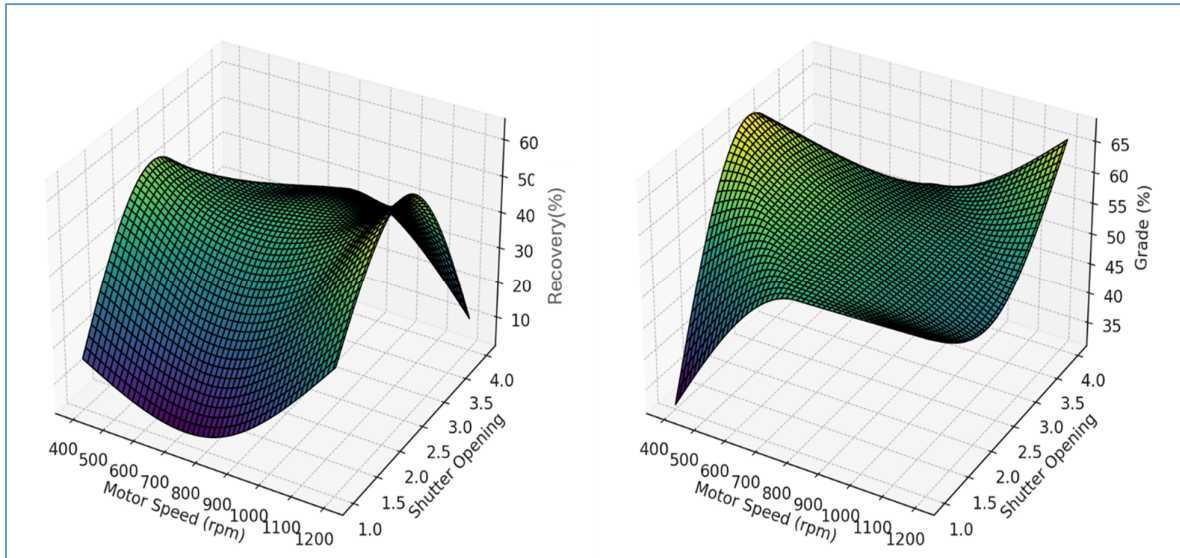


Figure 7. Response surface methodology (RSM) for both Grade (%) and recovery (%) based on motor speed (rpm) and shutter opening (mm).

3.4. Optimization of air classifier parameters

Optimization of motor speed and shutter opening is crucial for maximizing the recovery and the grade simultaneously. The 3D surface plot for the recovery (Figure 7) shows that increasing both motor speed and shutter opening enhances the recovery, particularly for fine particles. However, the grade optimization surface reveals that beyond certain thresholds, further increases in motor speed or shutter opening result in a decline in the product quality.

The optimal conditions for the recovery were found at a motor speed of 1200 rpm and a shutter opening of 2.5 mm. These conditions provide the necessary centrifugal force and airflow to efficiently separate fine particles, while avoiding the misclassification of larger particles. In terms of grade, the best results were achieved at moderate motor speeds and shutter openings, where excessive airflow is avoided, minimizing the loss of finer particles.

4. Conclusions

This work demonstrates the effectiveness of air classification as a dry beneficiation method for purifying PAL, offering a sustainable alternative to the traditional wet processing techniques. The research work provides valuable insights into optimizing the classification process to enhance both the grade and recovery of PAL. In conclusion, this work finds that:

- Finer particles required precise airflow control to achieve optimal recovery and grade, showing a higher sensitivity to the classifier settings.
- Higher motor speeds improved the recovery of fine particles by generating sufficient centrifugal force.
- The optimal conditions were found at 1200 rpm motor speed and a 2.5 mm shutter opening.
- Wider shutter openings enhanced airflow and improved fine particle recovery but resulted in a variability for larger particles due to increased velocity.
- Under these optimized conditions, a recovery rate of 53.2% and a PAL grade of 67.8% were achieved, demonstrating the effectiveness of the dry air classification.

In general, this work opens several avenues for further research:

- Explore the use of low shear grinding techniques to further enhance the grade and recovery of PAL, potentially leading to more efficient beneficiation processes.
- Conduct studies at a pilot scale to validate the laboratory-scale findings and assess the practicality of scaling up the air classification process for industrial applications.
- Examine additional variables and process parameters to refine the air classification method and improve its efficiency and effectiveness in different operational conditions.

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بهینه‌سازی بهره‌وری خشک پالیگورسکیت از طریق طبقه‌بندی هوا برای بهبود درجه و بازیابی

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چکیده:

پالیگورسکیت (PAL)، همچنین به عنوان آتاپولزیت شناخته می‌شود، یک کانی رسی است که به دلیل ساختار سیلیکات نانومیله مانند و مورفولوژی فیبری آن ارزشمند است. روش‌های سنتی تصفیه PAL اغلب شامل تکنیک‌های جداسازی گرانشی مرطوب مانند رسوب‌گذاری و غربال‌گری است که به مصرف آب قابل توجهی نیاز دارد و چالش‌های پایداری را به‌ویژه در مناطق کم‌آب ایجاد می‌کند. این کار یک روش جدید بهره‌برداری خشک سازگار با محیط‌زیست را برای PAL معرفی می‌کند. یک نمونه PAL بزرگ با محتوای ۴۱.۷ درصد و رطوبت ۱۰ درصد خرد شد، با استفاده از آسیاب بین آسیاب شد و به سه بخش اندازه ذرات طبقه‌بندی شد: -۰.۰۸۸ mm + 0.066 mm، -0.066 mm + 0.044 mm، و -۰.۰۴۴ mm. این فراکسیون‌ها با یک طبقه‌بندی هوا درمان شدند. یک طرح آزمایشی Box-Behnken برای بررسی اثرات اندازه ذرات، باز شدن شاتر و سرعت موتور بر بازده طبقه‌بندی استفاده شد. پارامترهای بهینه برای درجه، سرعت موتور ۴۰۰ دور در دقیقه، باز شدن شاتر ۱ میلی‌متر و اندازه تغذیه ۰.۰۶۶- میلی‌متر + ۰.۰۴۴ میلی‌متر بود. برای بازیابی، شرایط بهینه سرعت موتور ۱۲۰۰ دور در دقیقه، باز شدن شاتر ۲.۵ میلی‌متر و اندازه تغذیه ۰.۰۴۴- میلی‌متر بود. مطلوب‌ترین تعادل درجه (۶۷.۸٪) و بازیابی (۵۳.۲٪) با سرعت موتور ۱۲۰۰ دور در دقیقه، باز شدن شاتر ۴ میلی‌متر و اندازه تغذیه ۰.۰۶۶- میلی‌متر + ۰.۰۴۴ میلی‌متر به دست آمد. کار نتیجه می‌گیرد که طبقه‌بندی هوا به طور قابل توجهی فرآیند بهره‌برداری PAL را با افزایش ۵۰ درصدی در درجه افزایش می‌دهد و توصیه می‌کند تکنیک‌های آسیاب برشی کم را برای بهبود بیشتر بررسی کنید.

کلمات کلیدی: پالیگورسکیت، فرآوری مواد معدنی خاک رس، طبقه‌بندی هوا، طراحی جعبه-بنکن، تکنیک‌های جداسازی پایدار.