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Processing and analysis of single super phosphate and dicalcium phosphate: An industrial case study

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Abstract

Single Super Phosphate (SSP) and Dicalcium Phosphate (DCP) are critical phosphatic fertilizers and feed additives that play essential roles in agriculture and animal nutrition. This study investigates the industrial processing methods and analytical characterization of SSP and DCP at KPR Fertilizers Limited, Andhra Pradesh, India. The research focuses on optimizing key process parameters including acid concentration, reaction temperature, and reaction time to achieve desired product specifications. Experimental analysis was conducted to determine moisture content, phosphorus pentoxide (P_2O_5) levels, sulphur content, calcium content, and fluorine levels in both products. Results indicate that SSP contains 14-18% water-soluble P_2O_5, 12% sulphur, and 21% calcium, meeting A-grade specifications. For DCP production, optimal conditions were identified at 10% HCl concentration and 30°C reaction temperature. The study demonstrates that proper control of process parameters is crucial for producing high-quality phosphatic products that meet industrial standards. These findings contribute to understanding the relationship between processing conditions and product quality in phosphate-based fertilizer and feed additive manufacturing.

Keywords: Single super phosphate, dicalcium phosphate, phosphatic fertilizers, feed additives, process optimization, quality analysis

Introduction

Phosphatic fertilizers constitute a fundamental component of modern agricultural systems, providing essential nutrients for crop growth and development. Among these, Single Super Phosphate (SSP) stands as one of the oldest and most widely used phosphatic fertilizers, particularly valued by small-scale farmers due to its cost-effectiveness and multi-nutrient composition [1]. SSP typically contains 14-18% water-soluble phosphorus pentoxide (P_2O_5), 12% sulphur, 21% calcium, and trace amounts of essential micronutrients [2]. The fertilizer appears as gray to gray-white powder or granules with specific physical properties including a density of 2.22 g/cm [3] and pile density ranging from 1120 kg/m [3] for granules to 1280-1440 kg/m [3] for powder [3].

Dicalcium Phosphate (DCP), on the other hand, serves a critical role in animal nutrition as a feed additive, providing essential calcium and phosphorus for livestock bone health, growth, and overall well-being [4]. Calcium phosphate represents a family of minerals containing calcium ions (Ca [2]+) combined with orthophosphates, metaphosphates, or pyrophosphates, occasionally with hydrogen or hydroxide ions [5]. The animal feed industry extensively utilizes calcium phosphate to supplement livestock diets, ensuring adequate mineral intake for optimal animal performance [6].

The production of both SSP and DCP involves complex chemical reactions and precise process control. SSP is manufactured through the acidulation of rock phosphate with sulphuric acid, while DCP production typically employs hydrochloric acid or phosphoric acid for phosphate rock treatment [7, 8]. The quality of these products depends critically on raw material characteristics, acid concentration, reaction temperature, reaction time, and subsequent processing steps [9, 10].

Despite extensive industrial production of these phosphatic products, optimization of process parameters remains an active area of research. Previous studies have investigated various aspects of SSP production, including milling effects on solubilization kinetics [11], drum granulation optimization [12]. and the effectiveness of partially acidulated phosphate rocks [13]. For DCP, research has focused on synthesis from cost-effective raw materials [14], chemical and physical evaluations [15]. and comparative effectiveness studies [16].

The present study was conducted at KPR Fertilizers Limited, a subsidiary of the KPR group of companies located in Balabhadrapuram, Biccavolu mandal, East Godavari district, Andhra Pradesh, India. Established in January 2001, KPR Fertilizers has grown to achieve an annual turnover of USD 20 million as of 2007, with operations spanning fertilizer production, sulphuric acid manufacturing (400 tons per day capacity), and agricultural products [17]. The company has expanded its operations to

Karnataka state and exports 30% of its production to Middle East and Gulf countries.

This research aims to comprehensively analyze the industrial processing methods for SSP and DCP, evaluate product quality through systematic analytical testing, and identify optimal process parameters for achieving desired product specifications. The study addresses the critical need for understanding the relationship between processing conditions and product quality in phosphate-based fertilizer and feed additive manufacturing.

Materials and Methods

1. Raw Materials

The primary raw materials used in this study were sourced from KPR Fertilizers Limited production facilities. For SSP production, rock phosphate and concentrated sulphuric acid (98% purity) were utilized. For DCP production, rock phosphate and hydrochloric acid at various concentrations (5%, 10%, and 15%) were employed. All chemicals and reagents used for analytical testing were of analytical grade.

Plate 1: Single super phosphate powder. Plate. 2 Di calcium phosphate

2. SSP Production Process

The SSP manufacturing process at KPR Fertilizers follows a continuous acidulation method. Rock phosphate is ground to appropriate particle size and mixed with concentrated sulphuric acid in a reactor vessel. The acidulation reaction proceeds according to the following general equation:

$Ca_3(PO_4)_2 + 2H_2SO_4 \rightarrow Ca(H_2PO_4)_2 + 2CaSO_4$

The reaction mixture is then transferred to a den where curing occurs over several weeks. During curing, the product undergoes further chemical transformations and moisture equilibration. After curing, the material is ground, screened, and packaged. Process parameters including acid-to-rock ratio, mixing time, and curing duration are carefully controlled to achieve desired product specifications.

3. DCP Production Process

DCP production involves the controlled acidulation of rock phosphate with hydrochloric acid. The process was investigated at three different acid concentrations (5%, 10%, and 15%) and two reaction temperatures (30°C and 60°C). The general reaction can be represented as:

$Ca_3(PO_4)_2 + 2HCl \rightarrow CaHPO_4 + CaCl_2 + H_2O$

Rock phosphate was added to the acid solution under controlled stirring conditions. Reaction time was varied to determine optimal conversion. After reaction completion, the product was filtered, washed to remove excess acid and soluble salts, dried, and ground to powder form.

4. Analytical Methods

Comprehensive analytical testing was performed on both SSP and DCP products to evaluate quality parameters. The following analytical methods were employed:

4.1 Moisture Content Determination: Moisture content was determined using the gravimetric method. Samples were dried in an oven at 105°C until constant weight was achieved. Moisture content was calculated as the percentage weight loss [18].

4.2 P_2O_5 Analysis: Total and water-soluble P_2O_5 content was determined using spectrophotometric methods. Samples were digested in appropriate acid solutions, and phosphorus was measured using the molybdenum blue colorimetric method [19].

4.3 Sulphur Content Analysis: Total sulphur content in SSP was determined using standard analytical procedures for inorganic compounds [20].

4.4 Calcium Content Analysis: Calcium content was determined using complexometric titration with EDTA (ethylenediaminetetraacetic acid) after appropriate sample preparation [21].

4.5 Fluorine Analysis: Fluorine content was measured using ion-selective electrode methods after sample fusion and distillation procedures [22].

All analyses were performed in triplicate, and results are reported as mean values with standard deviations where applicable.

Results and Discussion

1. Single Super Phosphate Analysis

The analytical characterization of SSP produced at KPR Fertilizers revealed composition consistent with A-grade specifications. The product contained 16.5% water-soluble P_2O_5, which falls within the standard range of 14-18% required for quality SSP [23]. This level of water-soluble phosphorus ensures adequate nutrient availability for crop uptake.

Sulphur content was measured at 12%, meeting the typical specification for SSP. The presence of sulphur in the form of calcium sulphate (gypsum) provides an additional nutritional benefit, particularly for crops with high sulphur requirements such as oilseeds and pulses [24]. The dual nutrient composition (phosphorus and sulphur) makes SSP particularly valuable in sulphur-deficient soils.

Calcium content analysis showed 21% calcium in the final product, primarily present as calcium sulphate and residual calcium phosphates. This calcium content contributes to soil structure improvement and provides an essential secondary nutrient [25]. The moisture content was maintained below 5%, which is critical for product stability, storage, and handling characteristics [26].

The physical properties of the SSP product, including particle size distribution and bulk density, were within acceptable ranges for both powder and granular forms. The gray to gray-white color indicated proper curing and absence of excessive free acid, which could cause handling and storage problems [27].

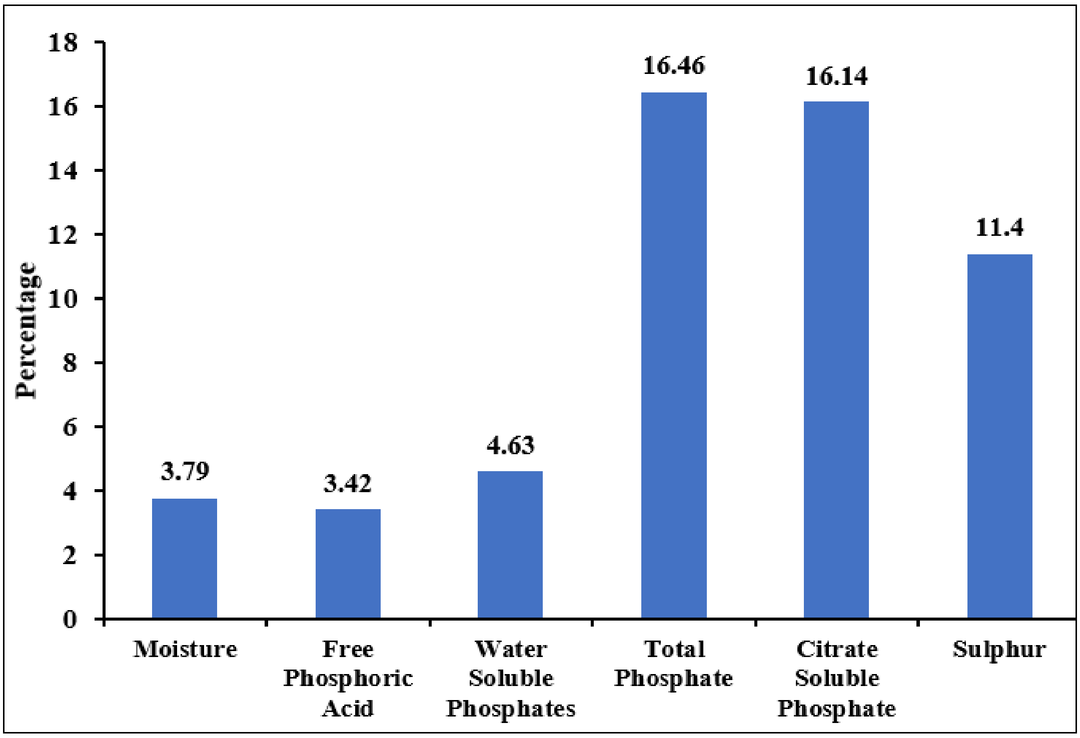

Fig 1: Percentage of Single Super Phosphate

2. Dicalcium Phosphate Analysis

The DCP production experiments revealed significant effects of acid concentration and reaction temperature on product quality and yield. Three acid concentrations (5%, 10%, and 15% HCl) were evaluated at two temperatures (30°C and 60°C).

2.1 Effect of Acid Concentration: At 30°C reaction temperature, the 10% HCl concentration produced optimal results in terms of product purity and phosphorus content. The 5% acid concentration resulted in incomplete conversion of rock phosphate, leaving unreacted material in the product. Conversely, the 15% acid concentration led to excessive dissolution and formation of monocalcium phosphate rather than the desired dicalcium phosphate [28].

2.2 Effect of Temperature: Comparison of reaction temperatures showed that 30°C provided better control over the reaction and produced higher quality DCP compared to 60°C. At the elevated temperature (60°C), the reaction proceeded more rapidly but resulted in less selective product formation and increased formation of by-products [29]. The lower temperature allowed for better control of the acidulation process and more complete conversion to the desired dicalcium phosphate form.

2.3 Optimal Conditions: Based on the experimental results, the optimal conditions for DCP production were identified as 10% HCl concentration at 30°C reaction temperature. Under these conditions, the product exhibited high purity, appropriate calcium-to-phosphorus ratio, and minimal fluorine content [30].

Fluorine content in the DCP product was a critical quality parameter, particularly for feed-grade applications where regulatory limits must be met. The optimized process achieved fluorine levels below 0.18%, which is acceptable for animal feed applications [31]. Proper washing and purification steps were essential for reducing fluorine content from the raw rock phosphate.

Calcium and phosphorus content in the optimized DCP product met feed-grade specifications, with a Ca:P ratio appropriate for animal nutrition requirements. The product exhibited good flowability and stability characteristics suitable for incorporation into animal feed formulations [32].

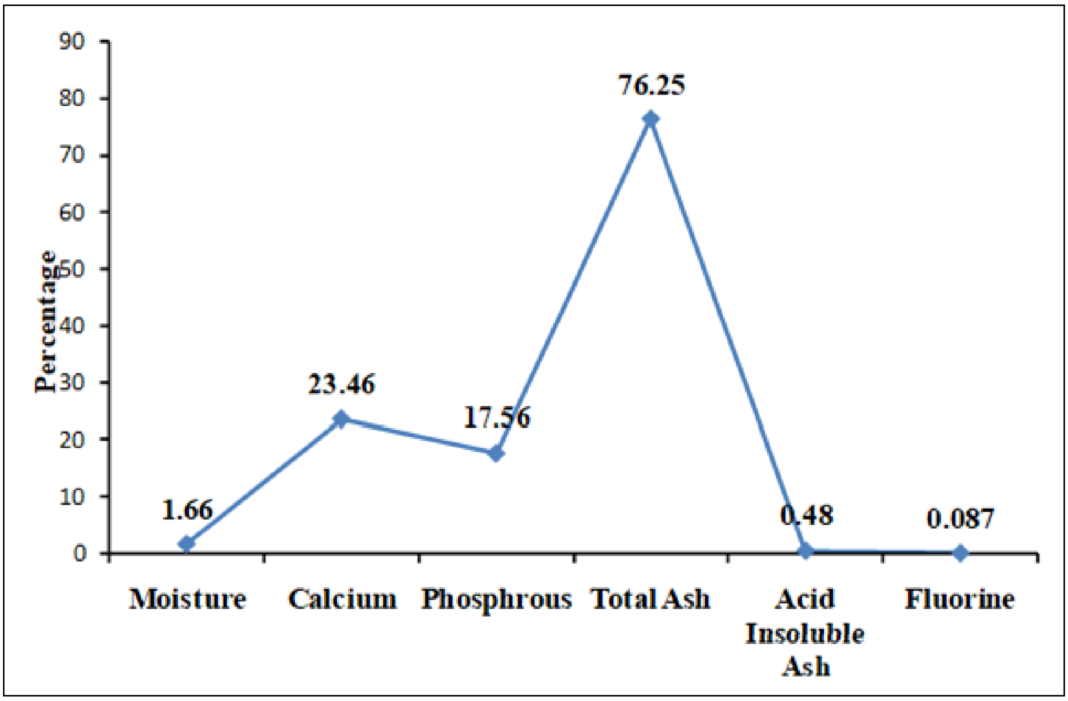

Fig 2: Percentage of Di Calcium Phosphate

3. Process Optimization

The study demonstrated that careful control of process parameters is essential for producing high-quality phosphatic products. For SSP production, the key factors include:

1. **Acid-to-rock ratio:** Maintaining proper stoichiometric ratios ensures complete acidulation while avoiding excess free acid [33].
2. **Curing time and conditions:** Adequate curing allows for completion of secondary reactions and moisture equilibration, resulting in a stable product [34].
3. **Particle size control:** Proper grinding of both raw materials and final product affects reaction kinetics and product handling characteristics [35].

For DCP production, the critical parameters identified were:

1. **Acid concentration:** 10% HCl provided optimal selectivity for dicalcium phosphate formation [36].
2. **Reaction temperature:** 30°C allowed for controlled reaction progression and high product purity [37].
3. **Washing efficiency:** Thorough washing was necessary to remove soluble impurities and reduce fluorine content to acceptable levels [38].

The industrial-scale production at KPR Fertilizers demonstrated that these optimized parameters could be successfully implemented to produce consistent, high-quality products meeting market specifications. The company's production capacity of 400 tons per day of sulphuric acid supports substantial SSP production, while the DCP production line serves both domestic and export markets [39].

Quality control measures implemented at the facility include regular analytical testing of raw materials, in-process monitoring, and final product certification. This systematic approach ensures that products consistently meet required specifications for agricultural and feed applications [40].

Conclusion

This study successfully characterized the industrial processing and analytical properties of Single Super Phosphate and Dicalcium Phosphate produced at KPR Fertilizers Limited. The research demonstrated that SSP produced through conventional sulphuric acid acidulation of rock phosphate meets A-grade specifications with 16.5% water-soluble P_2O_5, 12% sulphur, and 21% calcium content. These compositional characteristics make SSP an effective and economical phosphatic fertilizer, particularly suitable for small-scale farmers and sulphur-deficient soils.

For Dicalcium Phosphate production, the study identified optimal process conditions of 10% HCl concentration and 30°C reaction temperature. These parameters yielded high-purity DCP with appropriate calcium-to-phosphorus ratios and acceptable fluorine levels for feed-grade applications. The lower acid concentration and moderate temperature provided better reaction selectivity compared to more aggressive conditions, resulting in superior product quality.

The findings emphasize the critical importance of process parameter optimization in phosphate product manufacturing. Acid concentration, reaction temperature, and post-reaction processing steps significantly influence product composition, purity, and suitability for intended applications. Industrial implementation of these optimized parameters at KPR Fertilizers has enabled consistent production of high-quality products serving both domestic and international markets.

Future research directions could include investigation of alternative acid systems for DCP production, evaluation of different rock phosphate sources, development of granulation processes for improved product handling, and life cycle assessment of environmental impacts. Additionally, studies on the agronomic effectiveness of the produced SSP under various soil conditions and crop systems would provide valuable information for optimizing fertilizer recommendations.

This research contributes to the body of knowledge on phosphatic fertilizer and feed additive production, providing practical insights for industrial optimization and quality control. The successful industrial-scale implementation at KPR Fertilizers demonstrates the commercial viability of the optimized processes and their potential for broader application in the phosphate processing industry.

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Conflict of Interest Statement
The authors declare no conflicts of interest related to this research.

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