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Research on Anti-Caking Properties of Potassium Dihydrogen Phosphate Fertiliser

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Abstract

This study aims to solve the caking problem of Potassium Dihydrogen Phosphate (KH_2PO_4) powder in storage. Developed anti caking agent consisting of three optimized components (Surfactant, Emulsifier, and Polyol). The research results indicate that the application of this anti caking agent to the KH_2PO_4 powder has an optimal caking rate of 5.4%. By contrast, the clumping rate of the reference substance is as high as 54.1% at the same condition. Its quality and structure were characterized through contact angle analyzer, surface tension analyzer, and scanning electron microscopy (SEM). This research serves as a reference for the quality and improvement of compound fertilizer.

Keywords

Anti-caking; Potassium Dihydrogen Phosphate; Surface Tension.

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1. Introduction

With the rapid development of agricultural drone technology, KH_2PO_4, a traditional foliar fertilizer, has been widely applied in the cultivation of economic crops, grains, fruits, and vegetables. Its aqueous solution is weakly acidic and can be readily absorbed by plant roots and leaves. KH_2PO_4 offers multiple physiological benefits, including promoting photosynthesis, enhancing stress resistance, facilitating flower bud differentiation, and improving fruit set rate and fruit quality [1,2,3]. However, due to its fine particle size and formulation for agricultural drone applications, KH_2PO_4 is susceptible to aggregation and caking during prolonged storage or under conditions of high temperature and humidity. This adversely affects the fertilizer's flowability, dosing accuracy, and application uniformity, leading to uneven fertilization, equipment blockage, resource waste, and potential environmental pollution.

Fertilizer caking essentially results from the formation of stable aggregates between particles due to physical, chemical, or mechanical interactions. The primary mechanisms include: (1) Moisture absorption–recrystallization when the ambient relative humidity exceeds the fertilizer's critical relative humidity (CRH), particle surfaces absorb moisture and partially dissolve, forming liquid bridges that recrystallize into "crystal bridges" upon drying, causing particle adhesion [4,5,6]; (2) Compaction and mechanical interlocking under stacking pressure or vibration, increased contact area enhances van der Waals forces and capillary effects, while surface roughness facilitates mechanical interlocking [7,8,9]; and (3) Chemical reaction certain fertilizer components may undergo polymorphic transitions or metathesis reactions during storage, generating low-melting or viscous substances that promote caking [10,11,12,13].

To mitigate caking, industrial practices typically employ coating, drying, or the addition of anti-caking agents. Among these, the incorporation of anti-caking agents is most common due to its simplicity and cost-effectiveness. Conventional anti-caking agents mainly inert powders (e.g., diatomaceous earth, talc) or surfactants often exhibit limited efficacy, reduce solubility, or introduce impurities[14,15,16,17]. Furthermore, most existing agents focus on physical isolation without addressing the improvement of interfacial properties in aqueous solutions.

In this study, a novel composite anti-caking agent (S) was developed to mitigate the caking problems observed during the practical use of KH_2PO_4. Through systematic evaluation of its influence on the caking behavior, surface tension, contact angle, and microstructure of KH_2PO_4, the mechanism of action was clarified. The study aimed to develop modified KH_2PO_4 fertilizers with enhanced physical stability and application performance, offering theoretical insights and technical support for the production of high-quality water-soluble fertilizers.

2. Materials and Methods

2.1 Materials

Potassium dihydrogen phosphate Wengfu (Group) Co., Ltd ;magnesium sulfate anhydrous; ferric ammonium citrate; silicon dioxide; magnesium stearate; potassium ferricyanide; sodium aluminosilicate; glycerol; components Surfactant, Emulsifier, and Polyol were purchased from Titan Scientific Co., Ltd. (Shanghai, China).

Polyethylene (PE) pouches (5 cm × 6 cm) were used for packaging.

2.2 Preparation of Anti-caking Agent S

Surfactant and Polyol were mixed in a molar ratio of 5:6 in a reaction vessel and stirred at 40 °C and 1000 r/min until a homogeneous solution formed. Emulsifier was then added at a molar ratio of 7 relative to Emulsifier, and the mixture was stirred at room temperature for 30 min to ensure complete dispersion, yielding a paste-like composite anti-caking agent S.

2.3 Preparation of Modified KH_2PO_4

A total of 500 g of KH_2PO_4 was used as the base material. 0.5% (w/w) of anti-caking agent S was added and blended in a mixer for 10 min to ensure uniform dispersion. The mixture was then ground and sieved through an 80-mesh sieve to obtain S-type KH_2PO_4 with uniform particle size. A blank control group (CK) was prepared by subjecting unmodified KH_2PO_4 to the same sieving process.

2.4 Caking Rate Determination

Caking behavior was evaluated using a self-designed accelerated caking test apparatus. Briefly, 20 g of sample was sealed in a PE pouch and placed under a pressure plate applying 0.3 kg/cm² at room temperature (25 °C) for 24 h. After compression, the pouch was dropped freely once from a height of 1 m. The sample was then sieved through a standard sieve (aperture size determined according to product particle size), and the retained aggregates were collected. The caking rate was calculated as:

$$\text{Caking rate}=\frac{mass\ of\ large\ agglomerates\ (g)}{total\ sample\ mass\ (g)}$$

Each experiment was performed in quadruplicate, and the mean value was reported.

2.5 Contact Angle and Surface Tension Measurement

The blank and S-type KH_2PO_4 samples were dissolved in deionized water at a ratio of 1:50 (w/v). A 5 μL droplet was dispensed onto a clean polystyrene petri dish surface using a micropipette. The droplet profile was recorded over 0–9 s using an image analysis system to determine the initial contact angle. Measurements were conducted in five replicates, and the average value was calculated with an error margin of ±1°.

Surface tension of the fertilizer solutions (diluted 24-fold with deionized water) was measured using a tensiometer based on the pendant drop method. Each sample was measured in triplicate, and the mean value was reported.

2.6 Microstructural Morphology Observation

Scanning electron microscopy (SEM) was employed to examine the surface morphology of S-type KH_2PO_4 particles, with a focus on the distribution of the anti-caking agent on the crystal surface.

2.7 Simulated Extreme Environment Test

Samples were placed in a climate chamber and subjected to cyclic conditions: 50 °C and 70% relative humidity (RH) for 12 h (day), followed by 12 h at ambient temperature (approximately 25 °C, night), for a total duration of 25 days. Samples were taken at regular intervals to determine the caking rate, thereby evaluating stability under harsh storage conditions[18].

2.8 Data Processing

Experimental data were processed and statistically analyzed using Microsoft Excel 2007. Graphs and figures were generated with Origin 2021.

3. Results and Analysis

3.1 Preliminary Screening of Single Anti-caking Components

Several substances with potential anti-caking properties were selected for evaluation. For comparative purposes, two additional aliquots of KH_2PO_4 were prepared without any additive and subjected only to the same sieving process; these served as blank control groups.

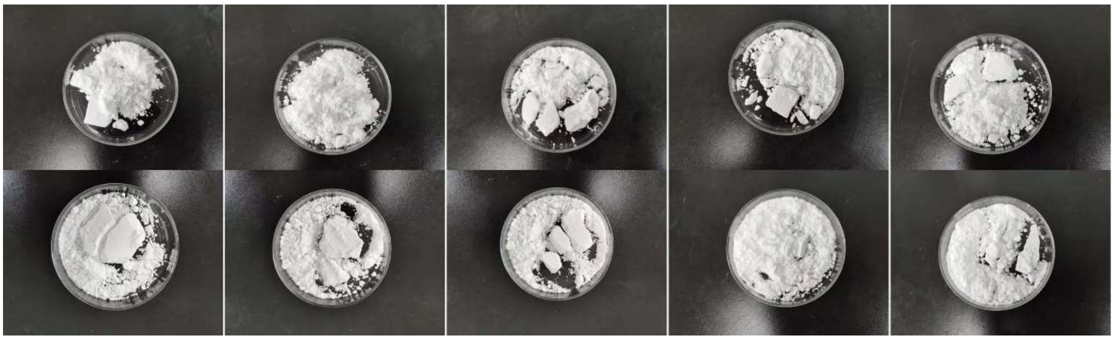

Figure 1. Caking Diagram

Table 1. Caking Rate of Different Additives

Type	Average caking rate(%)	Additive Solubility
Blank	54.1	Completely
Glycerol	44.0	Completely
Magnesium stearate	42.1	Insoluble
Ammonium iron citrate	44.4	Completely
Sodium aluminium silicate	43.8	Insoluble
Silicon dioxide	46.7	Insoluble
Potassium ferricyanide	43.6	Partially
Surfactant	23.4	Completely
Polyol	26.5	Completely
Emulsifier	18.7	Completely

A preliminary screening of various potential additives was conducted. The results showed that the caking rate of the blank group was 54.1%. The addition of glycerol, magnesium stearate, ferric ammonium citrate, or potassium ferricyanide slightly reduced the caking rate (42.1-46.7%), but the improvement was limited. Moreover, some of these substances exhibited poor solubility, which negatively affected the overall dissolution performance of the fertilizer. Among the tested materials, components A, C, and D showed relatively better performance, reducing the caking rate to approximately 23.4%, 26.5%, and 18.7%, respectively.

3.2 Evaluation of Composite Anti-caking Agent S

Based on the preliminary screening results, a composite anti-caking agent S was formulated using components A, C, and D. As shown in Table 2, the average caking rate of S-type KH_2PO_4 was 5.4%, significantly lower than that of the blank control group (54.1%), with a reduction exceeding 40%. The caking rates from four replicate experiments ranged from 9.3% to 12.9%, indicating good reproducibility and stability of the anti-caking agent. CK denotes the blank control group.

Table 2. S-type MKP versus CK control

Type	Caking rate(%)	Average caking rate(%)
S-1	6.1	5.4
S-2	5.3	
S-3	4.8	
S-4	12.9	
CK-1	57.2	54.1
CK-2	52.3	
CK-3	55.8	
CK-4	51.3	

To compare the performance of agent S with commercially available anti-caking agents, a comparative experiment was conducted under the same conditions using a commercial anti-caking agent following the above procedure. The results are presented below.

Table 3. Comparison of S with Commercial Anti-caking Agents

Type	Average caking rate(%)
CK	41.0
Comparison 1	31.7
Comparison 2	29.0
Comparison 3	20.0
S	5.4

The results indicate that the developed anti-caking agent exhibits superior performance compared to some commercial products, demonstrating significantly enhanced anti-caking efficacy.

3.3 Contact Angle Measurement

As shown in Figures 2 and 3, the contact angle of the blank KH_2PO_4 solution on a polystyrene surface was 104.67°, forming a distinctly spherical droplet with poor wettability. In contrast, the contact angle of the S-modified potassium dihydrogen phosphate solution decreased to 76.63°, with the droplet spreading more widely. This indicates that the S-modified solution has enhanced wetting and penetration capabilities, facilitating uniform adhesion to hydrophobic surfaces such as plant leaves.

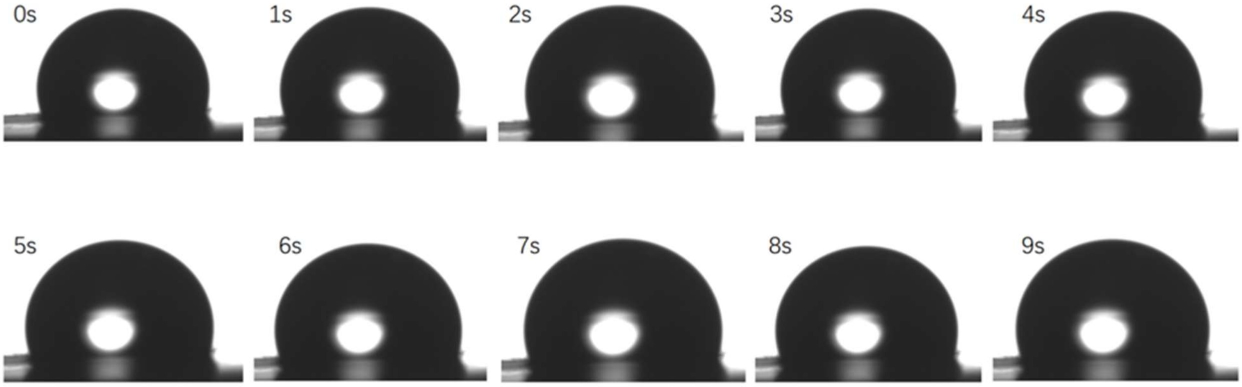

Figure 2. KH_2PO_4 contact angle image (104.674°)

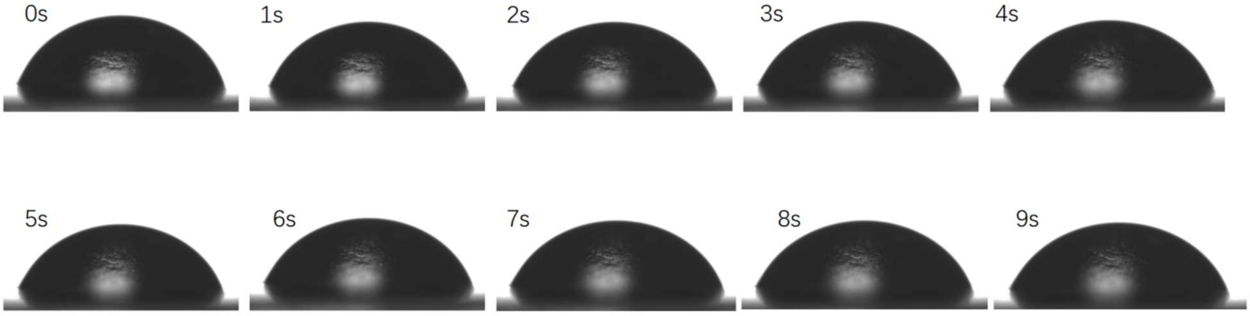

Figure 3. S- KH_2PO_4 contact angle image (76.625°)

3.4 Surface Tension Determination

Table 4. Surface Tension Measurement Results

	Type	Surface tension(mN/m)
		Dilute 24 times
1	Deionised water -1	71.12
2	Deionised water -2	71.911
3	Deionised water -3	72.315
4	CK-1	73.747
5	CK -2	75.263
6	CK-3	75.109
7	S-1	29.446
8	S-2	29.805
9	S-3	29.878

Table 4 indicates that the surface tension of the CK (74.72 mN/m), slightly higher than that of Deionised water (71.78 mN/m). In contrast, the surface tension of the S-type solution (29.34mN/m), representing a reduction exceeding 60%. The low surface tension aids in reducing droplet drift during spraying, enhances deposition efficiency, prolongs droplet retention time on leaf surfaces, and promotes nutrient uptake.

3.5 Microstructure and Long-term Stability

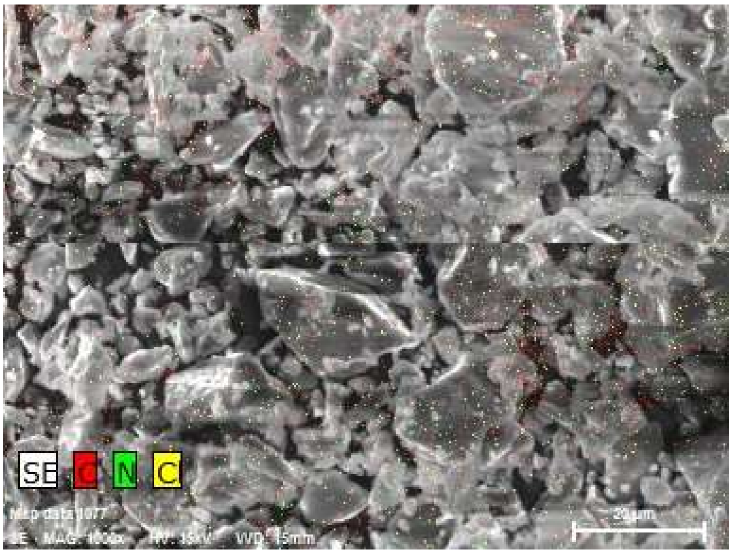

Figure 4. S-type MKP Electron Microscope Spectrum

SEM images reveal that anti-caking agent S forms a continuous and uniform coating layer on the surface of KH_2PO_4 crystals. This layer effectively prevents direct particle-particle contact, thereby inhibiting moisture migration and the formation of crystal bridges.

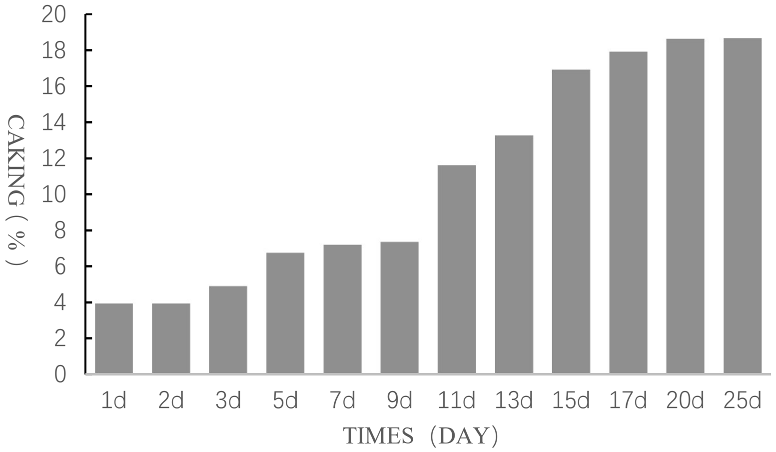

Figure 5. Clumping under extreme conditions after 25 days

Under simulated extreme conditions (alternating cycles of 50°C/70% RH), the S-type product exhibited a caking rate below 18.6% after 25 days, equivalent to the caking level observed after one month of natural stacking. Under standard ambient temperature and humidity conditions, the product remains stable for over 15 months of storage, meeting long-term warehousing requirements.

4. Discussion

The results of this study demonstrate that under simulated extreme environmental conditions (50 °C and 70% relative humidity for 25 days), S-type KH_2PO_4 maintains excellent physical stability. Its caking rate is significantly lower than that of conventional crystalline products, exhibiting outstanding long-term storage performance with an estimated shelf life of up to two years under standard ambient conditions (25 ± 3 °C, 60 ± 10% RH). The reduction in caking rate is closely associated with a lower degree of particle aggregation. The markedly fewer large aggregates observed further confirm the superior anti-caking performance of the S-type formulation.

Surface property analyses revealed that S-type KH_2PO_4 possesses a smaller contact angle and lower surface tension compared with the unmodified product. These characteristics indicate that its aqueous solution exhibits improved wetting and spreading behavior on hydrophobic surfaces such as plant leaves, thereby enhancing nutrient penetration and uptake efficiency. Scanning electron microscopy (SEM) images showed that functional component S forms a uniform coating over the KH_2PO_4 crystal surfaces, likely generating a continuous physical barrier that suppresses moisture migration and crystal bridge formation, effectively mitigating the caking process.

The superior anti-caking efficiency of the S-type product is primarily attributed to the synergistic interaction among additives A and D, which collectively improve particle flowability and reduce hygroscopicity by modulating crystallization dynamics and surface energy. Meanwhile, the inclusion of surfactant component C significantly decreases the contact angle and surface tension, thereby enhancing interfacial activity and dispersion properties. Together, these effects confer a balanced combination of structural stability and surface reactivity, ensuring both long-term storage durability and high functional performance during application.

Although this study successfully verified the stability of S-type KH_2PO_4 under accelerated aging conditions, several limitations remain. The present work did not systematically evaluate the influence of different packaging materials (e.g., moisture-resistant films or composite multilayer bags) on long-term storage stability. Future studies should incorporate extended field storage trials using actual packaging configurations to more comprehensively assess real-world warehousing performance. Additionally, further investigation into the aging mechanisms of S-type KH_2PO_4 under dynamic temperature–humidity cycling conditions is warranted. Coupling such experiments with molecular dynamics simulations could help elucidate the adsorption behavior of surface water molecules and interfacial interaction mechanisms on crystal surfaces, thereby providing theoretical insights for the rational design of next-generation stabilized phosphate fertilizers.

5. Conclusion

The results demonstrated that the caking rate of S-type KH_2PO_4 decreased by more than 40% relative to the blank control (CK), remaining below 20.0% even under simulated extreme conditions (50 °C, 70% RH, alternating cycles for 25 days). The contact angle of its aqueous solution was reduced from 104.67° to 76.63°, while the surface tension declined significantly from 74.72 mN/m to 29.34 mN/m, reflecting substantially improved wettability and adhesion. SEM analysis revealed that anti-caking agent S formed a continuous and uniform coating on the surfaces of KH_2PO_4 crystals, thereby suppressing crystal bridge formation and water migration. Overall, the findings demonstrate that the developed composite agent S effectively enhances the physical stability, storage durability, and surface activity of KH_2PO_4, offering strong potential for large-scale industrial application.

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