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# The influence of different operational variables on the production of crystalline monopotassium phosphate

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**Abstract:** Monopotassium phosphate stands out as a valuable fertilizer due to its abundance of potassium and phosphate in a form that plants can easily utilize. Boasting an impressive total plant food content of almost 87%, it outshines numerous other fertilizers in terms of nutrient concentration. Moreover, its low salt index and high water solubility make it an indispensable resource for promoting robust root development in plants. The production of monopotassium phosphate (MKP) is highly important to various industries due to its wide range of applications, particularly in agriculture, fertilizer manufacturing, and other sectors. Experimental investigations were carried out in this research to identify the most feasible operating conditions for synthesizing pure and clear crystalline monopotassium phosphate by reacting commercial potassium hydroxide with commercial furnace-grade phosphoric acid. The study focused on analyzing five operating variables, which comprised reaction temperature and time, crystallization temperature and time, and the molar ratio of $H_3PO_4$ to KOH. The primary factors considered in determining the best operating conditions were the size of the crystals obtained and the yield. The most feasible operating conditions were found to be a reaction temperature of 50°C, a reaction time of 60 minutes, a crystallization temperature of 10°C, a crystallization time of 60 minutes, and a molar ratio of 1 $H_3PO_4$ to 1 KOH, which resulted in a reasonable yield (73 wt%) and a satisfactory monopotassium phosphate crystal size.

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***Keywords:*** *Crystallization, Fertilizer, Furnace grade acid, Monopotassium phosphate, Potassium hydroxide.*

## 1. Introduction

Fertilizer serves as a vital component in soil enrichment to promote plant development and increase crop yield. Originating from ancient farming practices, the technology behind fertilizers has advanced significantly with the understanding of plants' chemical requirements. Modern synthetic fertilizers are predominantly formulated with nitrogen, phosphorus, and potassium compounds, along with additional secondary nutrients. The utilization of synthetic fertilizers has notably enhanced the quality and quantity of food production today, despite ongoing debates regarding their environmental impact [1].

Plants suffering from nitrogen deficiency exhibit stunted growth and yellowing leaves. Phosphorus, a crucial element in nucleic acids, phospholipids, and various proteins, is necessary for powering metabolic reactions. Inadequate phosphorus results in decreased plant growth. Potassium, another important substance for protein synthesis and other essential plant processes, is required to prevent yellowing, dead tissue spots, weak stems, and roots [2].

Mono-potassium phosphate (MKP) fertilizer, with a chemical formula of ($KH_2PO_4$), is classified as a straight phosphorous fertilizer. It contains 52% $P_2O_5$ and 34% $K_2O$, and is labeled as 0-52-34. MKP is a soluble salt that serves as a fertilizer, food additive, and fungicide. It is the preferred source of phosphorus and potassium in situations where nitrogen fertilization needs to be limited, such as during the early

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growing season when high rates of phosphorus and potassium are required for root system establishment [3].

The utilization of MKP during the growth stages of sugar-rich fruit crops is beneficial in enhancing sugar levels and enhancing the overall quality of the crops. MKP can be combined with other fertilizers to fulfill the nutritional requirements of the crops throughout their growth cycle. Its high purity and water-solubility make MKP an excellent choice for fertigation and foliar application. Moreover, MKP is well-suited for creating fertilizer blends and liquid fertilizers. When used as a foliar spray, MKP helps in controlling powdery mildew. When incorporated into fertilizer mixtures with urea and ammonium phosphates, MKP helps in reducing ammonia escape by maintaining a lower pH level [4]. With its composition of 52% $P_2O_5$ and 34% $K_2O$, MKP is commonly utilized as a nutrient source in the greenhouse industry and hydroponics [5, 6].

With the increasing demand for high-efficiency fertilizers and environmentally friendly agricultural practices, the market for MKP is expected to grow steadily. Innovations in production methods, especially those focused on reducing environmental impact, are anticipated to drive further market growth.

## 2. MKP Production Processes

The production of Mono Potassium Phosphate (MKP) is a well-established process, with most methods relying on an acid-base reaction between phosphoric acid and potassium salts. Recent advancements focus on improving the efficiency, sustainability, and environmental impact of production. As the global demand for high-quality fertilizers increases, ongoing research into alternative raw materials, process optimization, and waste reduction will be key drivers in shaping the future of MKP production. In the following section, the most available methods of MKP production processes are presented.

Numerous methods have been suggested in academic literature for the synthesis of monopotassium phosphate through a direct reaction between commercial wet process phosphoric acid and potassium chloride. The wet process phosphoric acid is known to contain a variety of impurities, such as organic materials, metals like iron, aluminum, magnesium, potassium, and cadmium, as well as elements like fluorine, calcium, and sodium [7, 8]. These impurities found in the wet process phosphoric acid pose significant challenges when utilizing it as the primary acid source due to the presence of chloride content in the final monopotassium phosphate product, resulting in the inability to achieve relatively pure monopotassium phosphate products [9].

The first preparation of monopotassium phosphate involved the neutralization of phosphoric acid with a potassium carbonate solution, followed by the crystallization of the resulting product. Despite its initial use, this method is not practical for large-scale fertilizer production and is only of theoretical interest. Manor, et al. [10] conducted a reaction between phosphoric acid and potassium chloride in the presence of an organic solvent containing a long-chain primary amine with 6 to 28 carbon atoms to synthesize monopotassium phosphate. However, the high production costs associated with this process, along with the expensive organic solvent, prevent its commercial application.

Holcomb, et al. [11] introduced a method to create monocalcium phosphate, phosphoric acid, and potassium phosphate fertilizers through the acidulation of phosphate rock with phosphoric acid, silicon dioxide, and potassium ions. The process involves the reaction of monopotassium sulfate with phosphate rock or dicalcium phosphate in the presence of phosphoric acid at a temperature ranging from 40 to 100 °C. The impurities found in the initial phosphate rock are effectively removed, resulting in the formation of calcium sulfate. After the separation of calcium sulfate, the filtrate is neutralized to a pH between 3 to 7 using calcium-containing compounds like $Ca(OH)_2$, CaO, or $CaCO_3$, and pure MKP devoid of chloride ions is crystallized from the filtrate.

Through a process developed by Erickson and his team, a relatively pure monopotassium phosphate was obtained by mixing phosphoric acid with potassium chloride at a temperature of 235°C. This method enables the release of hydrogen and chloride gases, leading to the recovery of MKP as a pure crystalline product with minimal chloride content. However, the high reaction temperature and the evolution of $Cl^-$ gas are identified as the primary disadvantages of this approach [9].

By combining wet-process phosphoric acids with potassium chloride and small quantities of sulfuric acid [12] low-chloride potassium phosphate fertilizers were produced. The mixture is then heated to a temperature ranging from 70°C to 200°C, and the chlorides are extracted as hydrochloric acid from the melt by passing a non-reactive gas or steam through it while maintaining the melt in a reduced-pressure atmosphere. This process yields potassium acid phosphate, which can be used to produce concentrated fertilizers containing no more than 2% chloride. Using N-methyl diethanolamine as an extractant of HCl, Zhang, et al. [13] used KCl and $H_3PO_4$ to precipitate $KH_2PO_4$. This process produces a high-quality product of $KH_2PO_4$ with high conversion and low energy consumption as claimed by the authors.

The choice of manufacturing process and raw materials is heavily influenced by both economic and environmental factors, making sustainability an important consideration in future developments.

### *2.1. Current Process Description*

In the ongoing research, the process of producing monopotassium phosphate commences with the reaction of a commercial potassium hydroxide (approximately 34 wt% KOH solution) with a commercial furnace-grade phosphoric acid (68 wt% $P_2O_5$) at a moderately low reaction temperature ranging from 35 to 60°C as shown in Figure 1. The reaction between potassium hydroxide and phosphoric acid can be expressed by the following equation:

$$KOH + H_3PO_4 \rightarrow KH_2PO_4 + H_2O$$

It has been determined that the gradual addition of phosphoric acid is crucial for obtaining a high conversion rate and controlling the reaction temperature effectively [14]. Once the reaction is finalized, the mixture should be cooled down with continuous stirring to promote the crystallization of MKP at a temperature within the range of 0 to 25°C, allowing for additional MKP nucleation and crystal growth. The resulting slurry containing MKP crystals and mother liquor is then processed in a centrifuge to separate the solid MKP crystals from the mother liquor.

The centrifuge's outputs consist of wet MKP crystals that require drying and mother liquor that can be recycled or directly used. To achieve the desired moisture content in the MKP crystals, a counter-current rotary dryer is employed to reduce the moisture content from about 2.5 wt% to 0.3 wt%. The rotary dryer utilizes air at a temperature of 120°C and a relative humidity ranging from 15 to 30% to achieve this objective. The solid MKP crystals leave the dryer at a temperature between 40 to 50°C. Part of the mother liquor produced can be recycled and combined with the reactants to improve the yield, while the other part can be used as a liquid fertilizer [15].

## 3. Experimental Part

The primary purpose of this experimental work is to determine the optimal operating conditions for the production of monopotassium phosphate using commercial potassium hydroxide and white phosphoric acid. These operating conditions encompass the reaction temperature, reaction time, crystallization temperature, crystallization time, and $H_3PO_4$ to KOH mole ratio.

The experiments conducted to attain a high MKP yield and a good crystal size are displayed in Table 4.1. The table clearly illustrates that the experimental work was segmented into five parts, with each part involving a change in one variable while keeping the other variables constant.

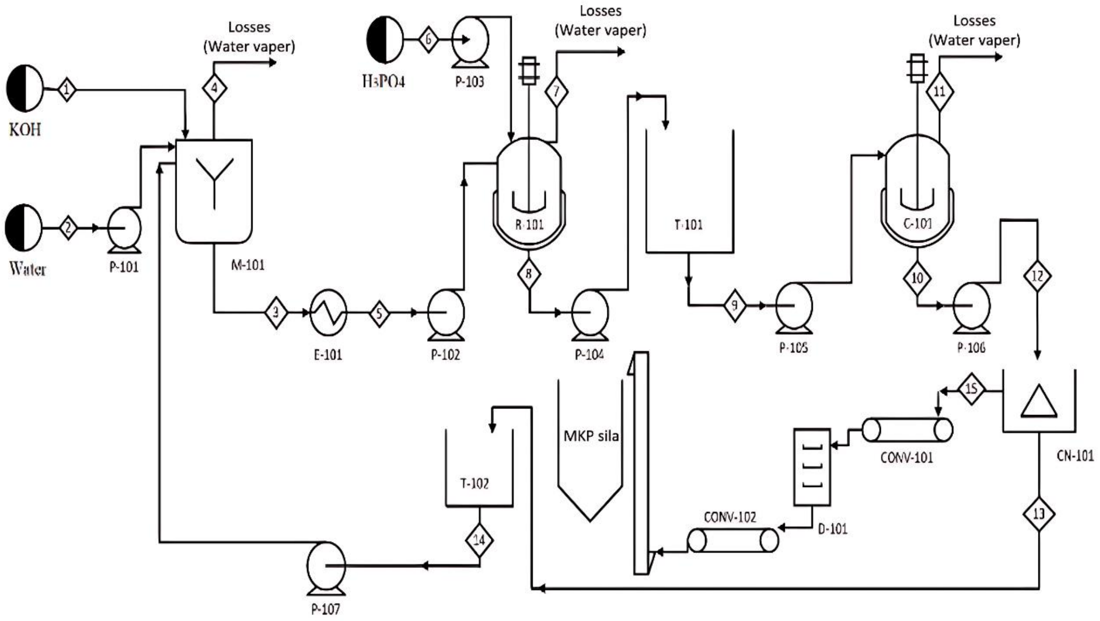

**Figure 1.**
Process flow sheet for the production of MKP.

**Table 1.**
Set of experiments and the parameters used for each MKP production run.

| Run | $H_3PO_4$ mole | KOH mole | Reaction temperature (°C) | Reaction time (Min.) | Crystallization temperature (°C) | Crystallization time (hr) |
|---|---|---|---|---|---|---|
| 1 | 1 | 1 | 50 | 60 | 10 | 60 |
| 2 | 1.1 | 1 | 50 | 60 | 10 | 60 |
| 3 | 1.2 | 1 | 50 | 60 | 10 | 60 |
| 4 | 1 | 1.1 | 50 | 60 | 10 | 60 |
| 5 | 1 | 1.2 | 50 | 60 | 10 | 60 |
| 6 | 1 | 1 | 35 | 60 | 10 | 60 |
| 7 | 1 | 1 | 40 | 60 | 10 | 60 |
| 8 | 1 | 1 | 45 | 60 | 10 | 60 |
| 9 | 1 | 1 | 50 | 60 | 10 | 60 |
| 10 | 1 | 1 | 55 | 60 | 10 | 60 |
| 11 | 1 | 1 | 60 | 30 | 10 | 60 |
| 12 | 1 | 1 | 50 | 90 | 10 | 60 |
| 13 | 1 | 1 | 50 | 120 | 10 | 60 |
| 14 | 1 | 1 | 50 | 60 | 25 | 60 |
| 15 | 1 | 1 | 50 | 60 | 20 | 60 |
| 16 | 1 | 1 | 50 | 60 | 5 | 60 |
| 17 | 1 | 1 | 50 | 60 | 0 | 60 |
| 18 | 1 | 1 | 50 | 60 | 10 | 30 |
| 19 | 1 | 1 | 50 | 60 | 10 | 90 |
| 20 | 1 | 1 | 50 | 60 | 10 | 120 |

### *3.1. Materials*

Potassium hydroxide flakes, with an 85% purity, can be acquired from the local market. The specifications for KOH flakes are outlined in Table 2. Commercial furnace-grade phosphoric acid, also with an 85% purity, serves as the primary source of $P_2O_5$, and the remaining $H_3PO_4$ specifications can be found in Table 3.

**Table 2.**
Potassium hydroxide flakes specifications.

| **Description** | **White deliquescent flakes** |
|---|---|
| Essay | Min. 85.0% |
| Carbonate (as $K_2CO_3$) | Max. 2.0% |
| Chloride (Cl) | Max. 0.03% |
| Melting Point/ Freezing point [°C] | 361 |
| Density [g/cm$^3$] | 2.04 |
| Solubility in water [% weight] | Soluble in water |

**Table 3.**
Specifications of the furnace-grade phosphoric acid used.

| **Appearance** | **Water-white liquid, colorless, odorless.** |
|---|---|
| Specific gravity (25°C/15.5°C) | 1.686 |
| Phosphoric Acid (%$H_3PO_4$) | 85.0 – 85.5 |
| $P_2O_5$ (%) | 61.7 |
| Viscosity (25°C) cp | 43.5 |
| Specific heat (cal/gm.°C) | 0.493 |
| Melting point | 70°F (21°C) |

### *3.2. Procedure*

The schematic diagram in Figure 2 illustrates the process carried out in the current work to generate the monopotassium phosphate crystals.

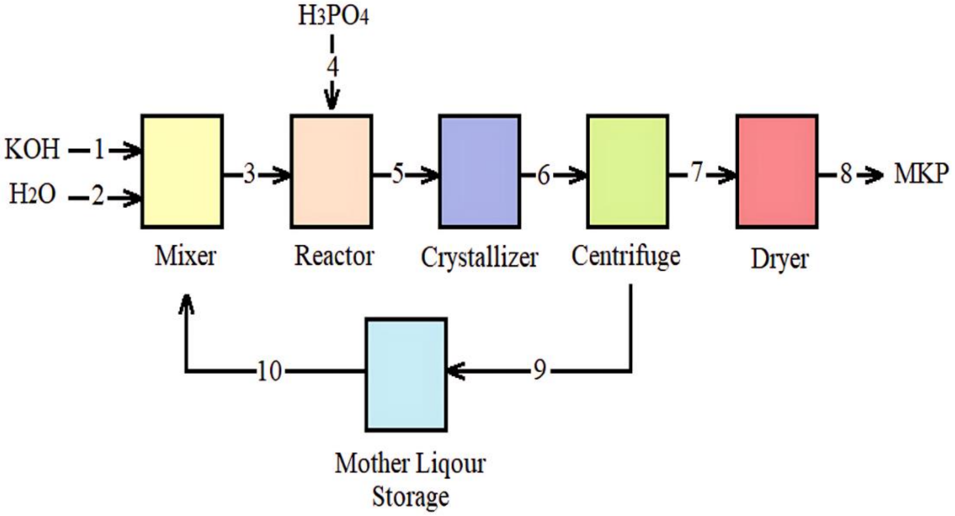

**Figure 2.**
Block flow diagram for MKP production.

A solution of potassium hydroxide with a concentration of 34% KOH is prepared by combining the necessary quantity of potassium hydroxide flakes with the appropriate amount of water. The specific volume of the recycled mother liquor is introduced into the container holding the potassium hydroxide solution at ambient temperature. As the mixture in the container is stirred at 150 rpm, the necessary quantity of $H_3PO_4$ is slowly added to avoid a sudden rise in the mixture's temperature and minimize losses and foaming.

Following the addition of $H_3PO_4$, the solution in the reaction vessel is moved to a water bath at a set temperature for a specific duration while being mixed continuously to finalize the reaction and allow MKP nucleation. Subsequently, MKP crystallization occurred in a separate water bath at a lower temperature with constant mixing at an appropriate speed to prevent crystal settling, and this process continued until crystallization was finished.

The MKP crystals are separated from the mother liquor using a basket centrifuge spinning at 1200 rpm. Subsequently, the final crystals are dried at 60°C to reach the desired moisture content before undergoing analytical testing to ascertain the levels of $K_2O$ and $P_2O_5$.

## 4. The Studied Parameters

### *4.1. Reaction Temperature Effect*

The reaction temperature in this section was adjusted to 20, 25, 30, 35, 40, 45, 50, 55, and 60°C, with all other variables held constant at a 60-minute reaction time, a crystallization temperature of 10°C, a 60-minute crystallization time, and a 1 to 1 molar ratio of $H_3PO_4$ to KOH.

The impact of the reaction temperature on MKP recovery is illustrated in Figure 3. In general, it is observed that the yield decreases with an increase in reaction temperature beyond 50°C, with a peak yield of 73% attained at a reaction temperature ranging from 45 to 50°C. Beyond 50°C reaction temperature, there is a noticeable delay in the commencement of the nucleation step, resulting in the production of larger crystals.

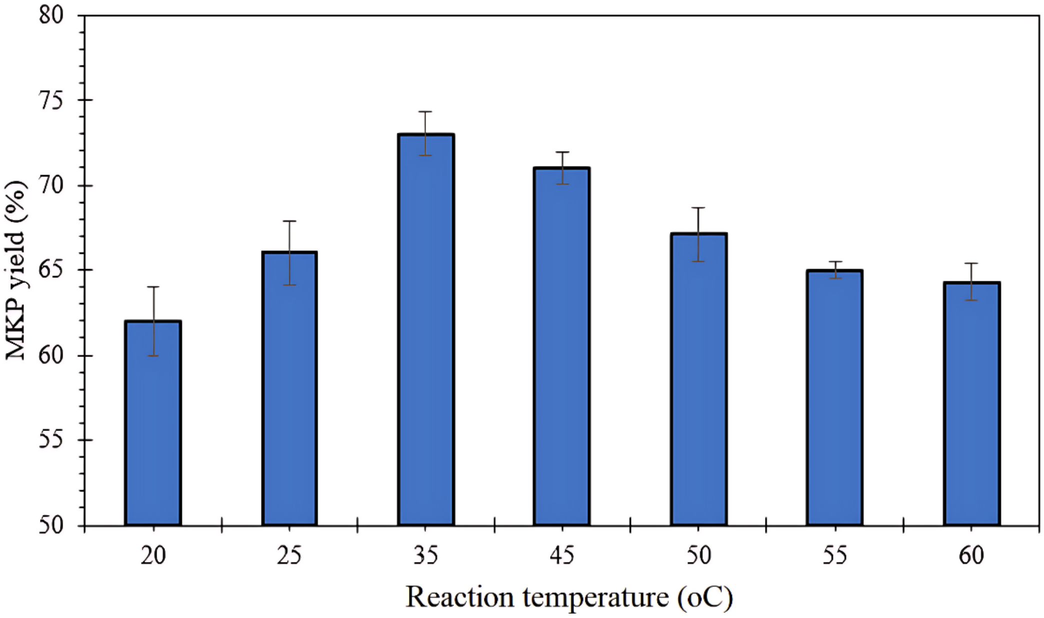

**Figure 3.**
MKP yield as a function of reaction temperature.

### *4.2. Reaction Time Effect*

Four experimental runs were performed at reaction times of 30, 60, 90, and 120 minutes, with the other factors maintained at a constant reaction temperature of 50°C, a crystallization temperature of 10°C, a crystallization time of 60 minutes, and a molar ratio of $H_3PO_4$ to KOH of 1 to 1.

The data presented in Figure 4 highlights the influence of reaction time on the recovery of the monopotassium phosphate product. It is evident that the yield experiences a substantial growth as the reaction time escalates from 30 to 60 minutes, showing an increment of about 18%. However, extending the reaction time from 60 to 120 minutes results in a minor increase in yield of approximately 1.4%. It can be observed from the figure that prolonging the reaction time beyond 1 hour has little effect on the final product yield. Adequate nucleation time is necessary in order to obtain large monopotassium phosphate crystals when the cooling process begins [15, 16].

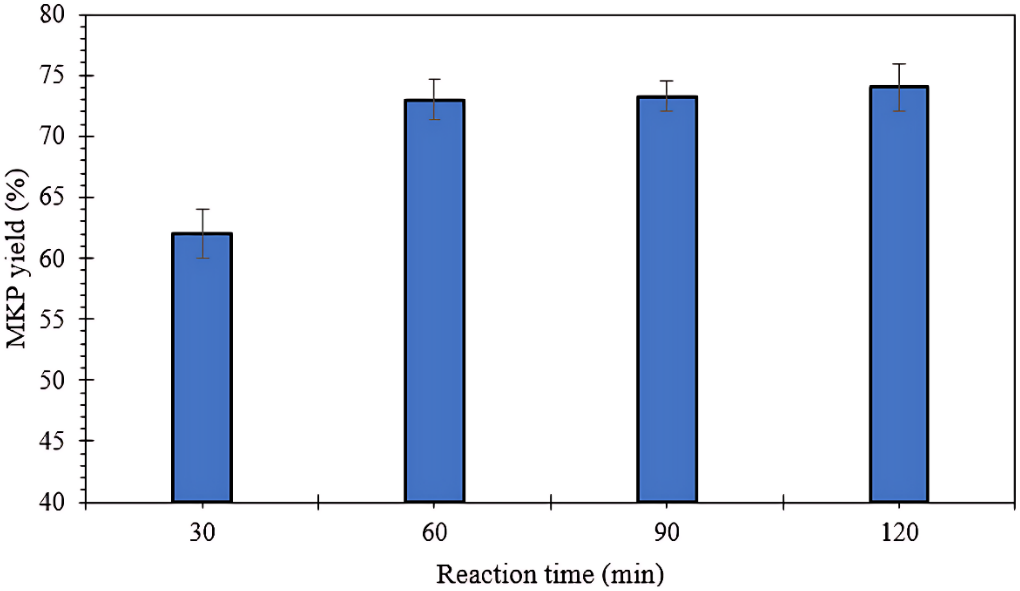

**Figure 4.**
The yield of MKP in connection with the duration of the reaction.

### *4.3. Crystallization Temperature Effect*

Within this section, the crystallization temperature was established at 5, 10, 20, and 25°C, with all other parameters held constant at a reaction temperature of 50°C, reaction time of 60 minutes, crystallization time of 60 minutes, and a molar ratio of $H_3PO_4$ to KOH set at 1 to 1.

Raising the temperature of the crystallizer leads to a decrease in the MKP yield while increasing the size of the resulting crystals. The impact of the crystallizer temperature on the yield of MKP produced is presented in Figure 5. It is apparent that reducing the crystallizer temperature will boost the yield of MKP crystals, with approximately 76% achievable at a crystallizer temperature of 5°C, although the crystals are smaller and more delicate. MKP crystals of increased size were acquired at a temperature of 25°C, albeit with a reduced yield of approximately 65%. Conversely, a satisfactory yield of 73% is achieved when the crystallization temperature is lowered to 10°C, resulting in MKP crystals of acceptable size. A smaller size of the MKP crystal results in an increased difficulty and duration of the drying process in the rotary dryer, leading to higher energy consumption.

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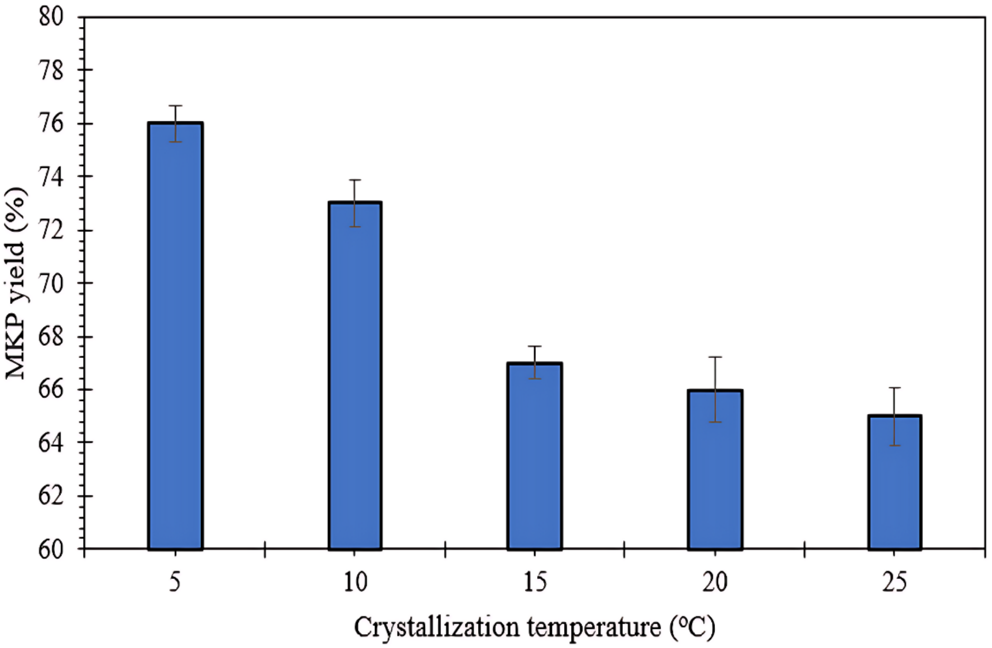

**Figure 5.**
The impact of crystallization temperature on MKP yield.

### *4.4. Crystallization Time Effect*

By manipulating the duration of crystallization, the study investigated the influence on both the recovery rate and the dimensions of MKP crystals. The crystallization time was varied at 30, 60, 90, and 120 minutes, with the reaction temperature set at 50°C, the crystallization temperature at 10°C, the reaction time at 60 minutes, and the molar ratio of $H_3PO_4$ to KOH maintained at 1 to 1.

The relationship between MKP yield and crystallization time is depicted in Figure 6. As the crystallization time increases, both the yield and crystal size increase until a crystallization time of 1 hour. Beyond this point, there is a slight increase in MKP yield with an increase in crystallization time from 60 to 90 minutes. Further prolonging the crystallization time does not enhance the yield of MKP; instead, it is accompanied by a decrease in yield attributed to the damage and dissolution of crystals resulting from continuous agitation during crystallization. A crystallization period ranging from 60 to 90 minutes is deemed adequate for achieving crystallization.

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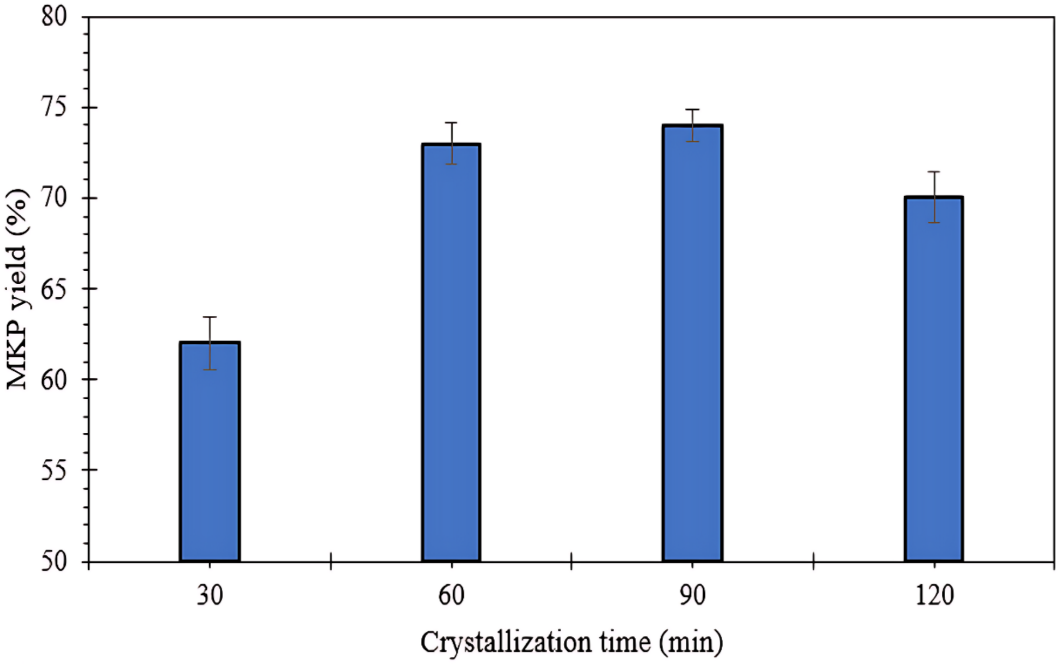

**Figure 6.**
The yield of MKP in relation to the time taken for crystallization.

### *4.5. Molar Ratio Effect*

The determination of the limiting reactant and the optimal molar ratio between $H_3PO_4$ and KOH was carried out through two separate sets of trials. In the first set, the moles of $H_3PO_4$ were set at 1.0, 1.1, and 1.2, while the moles of KOH were maintained at 1.0. In the second set, the moles of KOH were set at 1.0, 1.1, and 1.2, while the moles of $H_3PO_4$ were kept at 1.0. The remaining parameters were held constant, with the reaction temperature set at 50°C, the crystallization temperature at 10°C, the reaction duration at 60 minutes, and the crystallization duration at 60 minutes.

Figure 4.7 displays the relationship between MKP yield and the molar ratio of $H_3PO_4$ to KOH. The results indicate that an increase in the number of $H_3PO_4$ moles leads to a slight increase in yield, whereas an increase in the number of KOH moles does not have a notable impact on MKP yield. Therefore, it is preferable to maintain a molar ratio of 1:1, as a 20% increase in $H_3PO_4$ moles results in only about a 1% increase in yield, which is not a substantial improvement.

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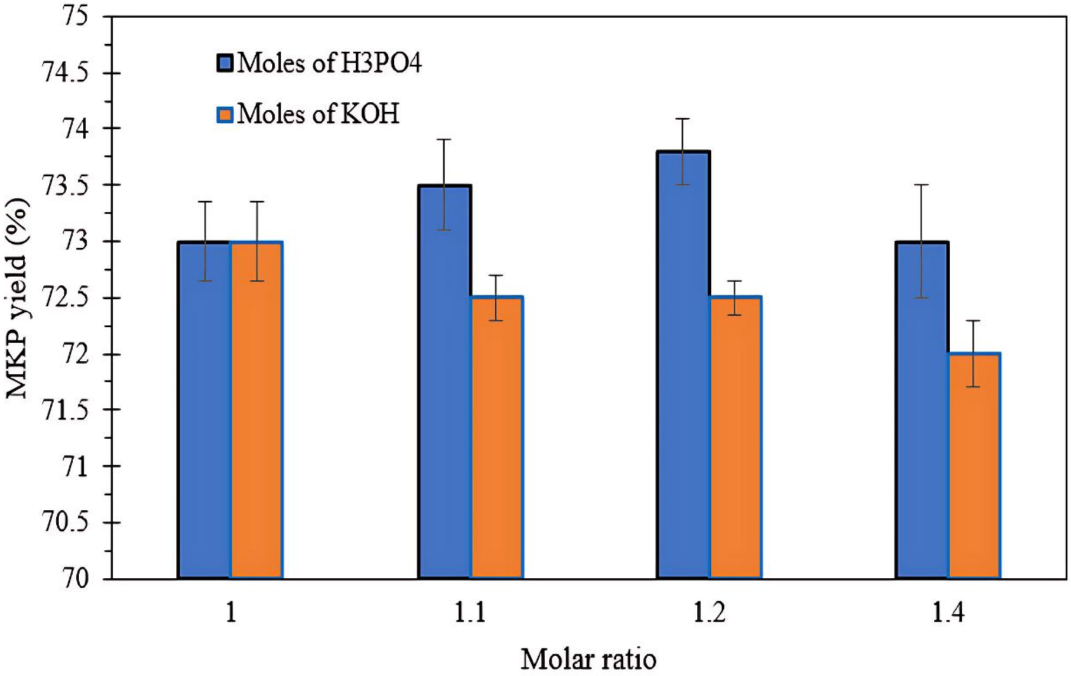


**Figure 7.**
The relationship between MKP yield and $H_3PO_4$/KOH molar ratio.

### *4.6. Best Operating Conditions*

According to the findings presented in this research, the most effective parameters for generating crystalline monopotassium phosphate include a reaction temperature of 50°C, a crystallization temperature of 10°C, a reaction duration of 60 minutes, a crystallization duration of 60 minutes, a molar ratio of 1.0 to 1.0, agitation speed at 150 rpm, and a centrifuging speed of 1200 rpm.

When the operating conditions are carefully controlled, MKP products can be obtained with large crystal sizes and an excellent yield. Figure 8 provides visual representations of MKP crystals grown under various operational parameters.

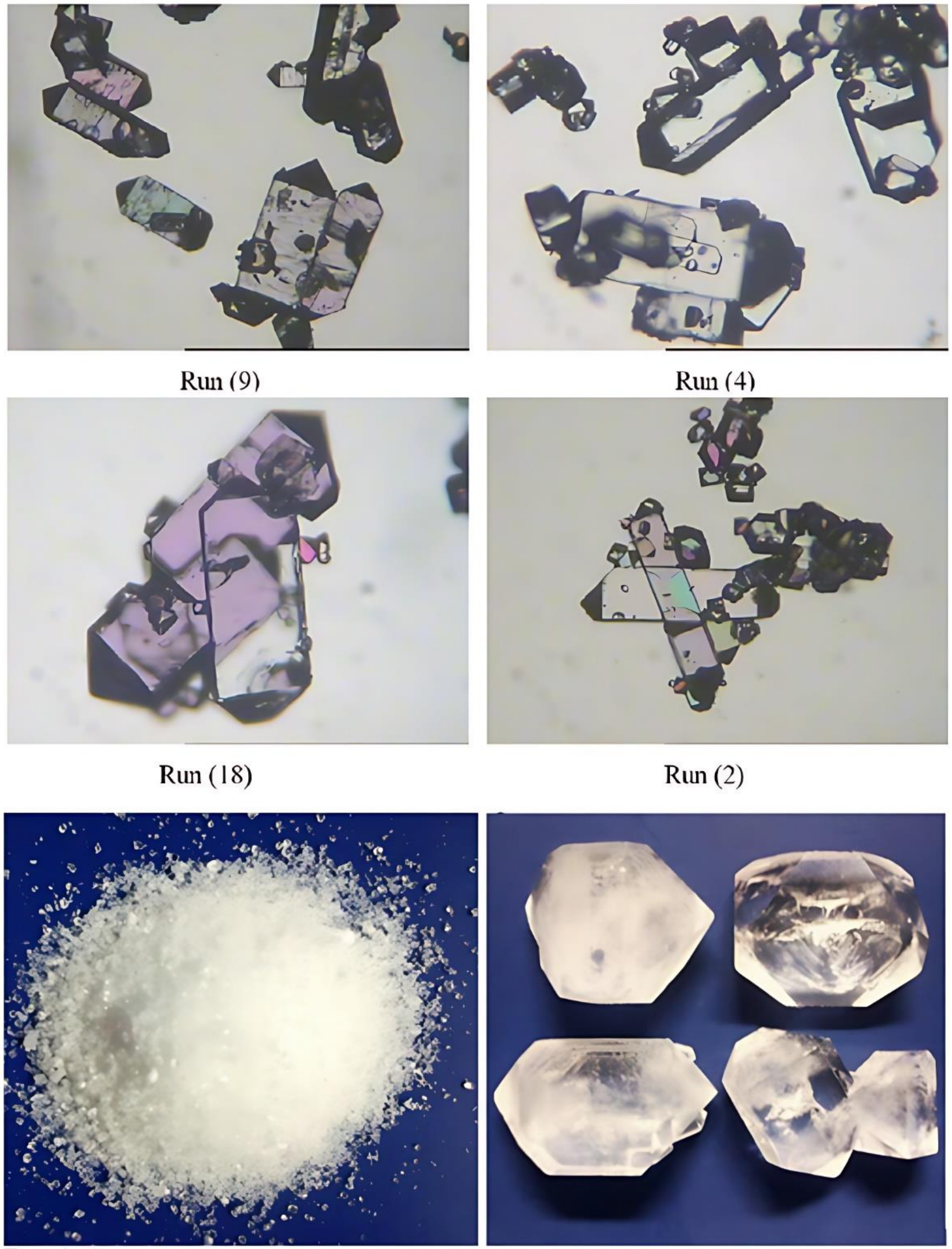
In light of the current work, it is crucial to separate the nucleation step from the growth step to achieve large crystals of crystalline monopotassium phosphate. Two separate pieces of equipment - a reactor and a crystallizer - should be used, rather than a combined reactor-crystallizer unit.

In accordance with the prescribed procedure and operational settings, a high drying temperature is not required. The monopotassium phosphate that is generated will possess large crystals with a non-sticky surface, thus demanding lower drying temperatures and decreased residence time in the dryer. This adjustment will effectively minimize the fuel consumption during the drying process.

## 5. Conclusions

High purity and clarity of monopotassium phosphate is achieved through the reaction of commercial potassium hydroxide with commercial furnace-grade phosphoric acid at a moderately low reaction temperature. The experimental study has determined that the best-operating conditions include a reaction temperature of 50°C, a reaction time of 60 minutes, a crystallization temperature of 10°C, a crystallization time of 60 minutes, and a molar ratio of 1.0 to 1.0, resulting in a yield of about 73 wt% and a reasonable crystal size.

Achieving the necessary size of monopotassium phosphate crystals requires the separation of the nucleation step from the growth rate step. Therefore, it is recommended to utilize a separate reactor and a separate crystallizer instead of a reactor-crystallizer equipment. The mother liquor acquired as a filtrate during this procedure holds significant value and can be marketed as a liquid fertilizer without the need for additional processing.



Run (9)

Run (4)

Run (18)

Run (2)

Run (20)

**Figure 8.**
Photographs of MKP crystals obtained from different runs.

### *5.1. Institutional Review Board Statement*

Approval for this research project has been obtained from the Institutional Review Board (IRB) at the University of Jordan and the American University of Beirut. The IRB assesses the ethical, scientific, and regulatory aspects of research involving human participants, ensuring that all activities are conducted in line with the highest standards of integrity and in compliance with relevant laws and regulations. The IRBs of both universities are devoted to protecting the rights, safety, and welfare of research participants, while also ensuring that the research is carried out ethically and responsibly.

## Transparency:

The author confirms that the manuscript is an honest, accurate, and transparent account of the study; that no vital features of the study have been omitted; and that any discrepancies from the study as planned have been explained. This study followed all ethical practices during writing.

## Copyright:

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