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REACTION OF ORTHOPHOSPHATE FERTILIZER IN CALCAREOUS SOIL AS AFFECTED BY PYROPHOSPHATE APPLICATION

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SUMMARY

The purpose of this study was to investigate the inhibitory effect of sodium pyrophosphate (NaPP) on P precipitation in two calcareous soils. Addition of NaPP to the studied soils tended to increase P concentration and it was more effective at the rate of 0.5% NaPP-P, especially when it was added either simultaneously at or 12 hours before orthophosphate application. The solubility data indicated that the major reaction products of OP in the calcareous soils with or without NaPP after one cycle was DCPD but its persistance was less in all treatments with no NaPP added during the further wetting and drying cycle. Generally, pyrophosphate acts as crystal growth inhibitor for DCPD, thereby maintaining higher soluble P concentration even after the inhibitory effect had disappeared.

INTRODUCTION

In calcareous soil, a considerable part of the applied orthophosphate (OP) precipitates as dicalcium phosphate dihydrate (DCPD). The later compound is also metastable with respect to more basic calcium phosphate (hydroxyapatite, HA) (Larsen, 1967). In general, in alkaline or calcareous soils calcium phosphate are the most stable compound. They decrease in solubility and availability to plant in the order: DCPD >OCP> HA. Hence, the transformation, the initially formed DCPD and OCP undertaken in soils are of practical significance.

One approach aimed for increasing fertilizer P efficiency is the use of a crystal growth inhibitor for retarding P immobilization (Amer, 1978 and Amer and Mostafa, 1981).Amer (1978),for instance ; first suggested that addition of a small amount of tetrasodium pyrophosphate (NaPP) to OP fertilizer may enhance the fertilizer P availability in calcareous soils due to the retarding effect of NaPP on the formation of DCPD in soils. Sodium pyrophosphate (NaPP) was shown to has a striking inhibitory influence upon the rate of crystal growth of DCPD (Marshall and Nancollas, 1969). The inhibitory effect was also analysed in terms of sorption of pyrophosphate ions at the vegetable crystal growth sites due to its long-chain P-O-P bonds which fit nicely to the calcite lattice (Larson, 1967). Work done by El-Zahaby *et al*., (1982) and Amer and Mostafa (1981) demonstrated that a small amount of NaPP can inhibit nucleation and / or crystal growth of DCPD in the system of their pure $CaCO_3$ or calcareous soil. Amer *et al.* (1982) found that NaPP tended to increase P recovery from MCP by the wheat and by the following corn on alluvial soil (4.1% $CaCO_3$), and only by the wheat on the desert soil (39% $CaCO_3$). One of the methods used for stabilizing the product in the manufacturing of DCPD involves the addition of a small amount of NaPP to the DCPD slurry (Toy, 1973). The purpose of this study is to investigate : (i) the reaction of orthophosphate fertilizer with or without NaPP in two calcareous soils and (ii) to test the effect of wetting and drying cycles on the inhibitory effect of NaPP.

MATERIALS AND METHODS

Samples of two calcareous surface soils (0 to 30 cm) different in origin were used. One was silty clay alluvial soil collected from the experimental station of Hartha and the other was loamy sand desert soil. The soils were air dried and sieved through a 1 mm screen. Their main characteristics are given in Table (1), using the standard procedures described by Page *et al.* (1982) for their determinations.

Table (1): Some characteristics of the soils.

Soil	pH (1:2.5)	$CaCO_3$ (mg/kg)	Organic matter (mg/kg)	CEC (Cmol/kg)	E.C. (dS/m)	P-$NaHCO_3$ (mg/kg)	Texture classes
Alluvial (Hartha)	8.03	250000	6500	18.42	5.84	11.78	silty clay
Desert (Zubair)	7.82	80000	1300	4.55	1.83	9.30	Loamy sand

Triplicates 200 gm samples of each soil were treated with one level of orthophosphate fertilizer to 2000 ppm P as KH_2PO_4 with powdered tetrasodium pyrophosphate ($Na_4P_2O_7.10H_2O$) at the equivalent rate of 0, 0.25 and 0.5 gm P per 100 gm soil in different combinations. They were thoroughly mixed and placed in round polyethylene containers. NaPP was added 12, 24 and 48 hours before or 24 and 48 hours after the addition of OP. Moisture content was raised to field capacity followed by incubation (25±1°C) for one and seven wetting and drying cycles. Each sample was irrigated in the ratio 1:1 (soil:water) and left for 24 hours for equilibrium, then the moistured soil was transferred to Buchner funnel fitted with filter paper and filtered under vacuum. The extract was collected in a 250 ml Erlenmeyer flask, and then used for pH, E.C., Mg and P determinations. Calcium and magnesium were determined by EDTA back titration as described by Graham et al. (1962) to eliminate P interference. Low concentration ions of P in the solution were measured by the method of Murphy and Riley (1962), whereas high P concentration were measured by the molybdovanadate method (Jackson, 1958).

The monocalcium phosphate potential (pH_2PO_4 + 1/2 pCa) and lime potential (pH-1/2pCa) were calculated using Debye-Huckel equation to obtain activity coefficients. Ion activities were calculated using an alternative programe employing the Davis equation (Davis, 1962), and E.C. was used as an estimate of ionic strength (Griffin and Jurinak, 1973). Correction for the following ions pairs were made; $CaSO_4$, $MgSO_4$, $MgHPO_4$, $CaHPO_4$, MgH_2PO_4 and CaH_2PO_4. All equilibrium constants used for ions pairs hydrolysis species, and mineral isotherms were selected from Lindsay (1979).

RESULTS AND DISCUSSION

Results in Table 2 and 3 indicated that the recovery percentage of the added OP in a water soluble form after the first wetting and drying cycle was 13% and 19%, respectively. The initial rapid drop of P concentration in the both studied soils can either be related to P adsorption which preceeds cluster formation and heterogenous nucleation of calcium phosphate on the carbonate surface (Griffin and Jurinak, 1973) or to P precipitation which is the major reaction mechanism as observed in these soils (Awad, 1985 and Awad and Al-Obaidy, 1989). The reduction in soluble P was lowered through seven cycles (Tables 2 and 3). Such changes reflect the hydrolysis of the initial reaction products to more basic calcium phosphate. Solubility criteria were employed to monitor the supersaturation of the P solutions relative to the known calcium phosphate at the early stage of the reaction and to determine which calcium phosphate may have existed after one and seven wetting and drying cycles. Figure (1) indicated that phosphate present in unfertilized soils (check treatments) was more soluble than hydroxyapatite (HA) but less soluble than OCP. Many workers such as Awad And Al-Obaidy (1989), Fixen et al. (1985) and Olsen et al. (1983) obtained similar results on unfertilized calcareous soils. The solubility data, further, showed that after the first wetting and drying cycle, the 2000 ppm P rate saturated the desert sandy loam soil with respect to DCPD. Upon further wetting and drying cycles, the P rate suffered a small increase in the monocalcium phosphate potentials and became supersaturated with respect to OCP during the 7th cycle. The alluvial silt clay soil, on the other hand, (Fig.1) was failed to saturate with respect to DCPD; the corresponding point after the first cycle was slightly below the DCPD line then droped above the HA line within the 7th cycle. DCPD could be produced initially in

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Table (2): Phosphorus solubility in alluvial silty clay soil after one and seven wetting cycles.

Treatments	One cycle pH	 soluble-P X10-4 M/L	 Soluble Ca X10-3 M/L	Seven cycles pH	 soluble-P X10-4 M/L	 soluble Ca X10-3 M/L
Soil	8.22	19.42	21.25	7.54	9.61	9.85
Soil+OP	8.16	282.90	18.20	7.40	85.10	7.90
Soil+NaPP1*	8.49	57.13	19.31	8.08	30.00	6.96
Soil+NaPP2**	8.54	69.50	17.41	8.47	46.11	1.36
Soil+(OP+NaPP1)	8.24	492.58	20.00	8.15	211.55	1.71
Soil+(OP+NaPP2)	8.46	577.74	16.75	8.58	410.00	1.74
Soil+NaPP1 <u>12 hrs</u> OP	8.23	498.90	18.20	7.87	385.10	1.87
Soil+NaPP1 <u>24hrs</u> OP	8.11	380.50	16.20	8.13	272.50	1.45
Soil+NaPP1 <u>48 hrs</u> OP	8.16	385.10	15.80	8.29	265.00	1.33
Soil+NaPP2 <u>12 hrs</u> Op	8.53	580.10	15.70	7.94	435.50	2.44
Soil+NaPP2 <u>24 hrs</u> OP	8.63	430.00	14.80	8.08	370.15	1.75
Soil+NaPP2 <u>48 hrs</u> OP	8.60	380.00	15.60	8.62	305.00	2.44
Soil+OP <u>24 hrs</u> NaPP1	8.13	315.00	16.40	8.57	139.65	1.70
Soil+OP <u>48 hrs</u> NaPP1	8.08	325.10	17.50	8.57	151.10	2.41
Soil+OP <u>24 hrs</u> NaPP2	8.61	400.00	15.10	8.54	165.50	2.57
Soil+OP <u>48 hrs</u> NaPP2	8.65	410.00	15.10	8.55	171.13	2.56

*NaPP1 at the equivalent rate of 0.25%
**NaPP2 at the equivalent rate of 0.5%.

all fertilized soils, but its persistance was less in alluvial soil than that in the desert soil. Many workers found similar effects due to wetting and drying cycles on the solubility of P applied to calcareous soil (El-Damaty et al., 1971). This could either be due to the phosphate compounds losing water on drying and becoming more insoluble (Amer et al., 1980) or that rewetting processes increased $CaCO_3$ solubility and produced a higher Ca ion concentration (Awad, 1985). Addition of NaPP to the studied soils tended to increase P concentration and it was more effective at the rate of 0.5% NaPP-P than at the rate 0.25% NaPP-P, especially when it added either 12 hours before OP rate or

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Table (13): Phosphorus solubility in sandy loam soil after one and seven wetting and drying cycles.

Treatments	One cycle			Seven cycles		
	pH	soluble-P X10-4 M/L	Soluble Ca X10-3 M/L	pH	soluble-P X10-4 M/L	Soluble Ca X10-3 M/L
Soil	8.23	0.95	3.20	7.77	0.55	2.20
Soil+OP	7.99	374.80	2.40	7.95	146.00	0.24
Soil+NaPP1*	8.08	38.98	1.25	8.12	41.22	0.90
Soil+NaPP2**	8.27	51.60	1.10	8.57	39.50	0.72
Soil+(OP+NaPP1)	7.99	445.10	1.15	8.49	194.50	0.60
Soil+(OP+NaPP2)	8.17	610.00	0.91	8.44	290.10	0.50
Soil+NaPP1 12 hrs OP	8.17	514.20	0.85	8.25	247.05	0.73
Soil+NaPP1 24hrs OP	8.69	412.25	0.80	8.16	211.02	0.73
Soil+NaPP1 48 hrs OP	8.68	433.52	0.70	8.53	229.50	0.60
Soil+NaPP2 12 hrs Op	8.47	641.12	0.75	8.33	450.17	0.70
Soil+NaPP2 24 hrs OP	8.67	450.04	0.60	8.23	315.00	0.50
Soil+NaPP2 48 hrs OP	8.52	420.04	0.80	8.25	286.19	0.40
Soil+OP 24 hrs NaPP1	8.86	337.82	0.75	8.37	186.95	0.66
Soil+OP 48 hrs NaPP1	8.47	382.07	0.85	8.35	170.80	0.68
Soil+OP 24 hrs NaPP2	9.19	430.74	0.80	8.42	196.50	0.43
Soil+OP 48 hrs NaPP2	9.18	465.98	0.85	8.37	223.10	0.46

*NaPP1 at the equivalent rate of 0.25%
**NaPP2 at the equivalent rate of 0.5%.

simultaneously with OP rate. Whereas a little effect was observed when the reverse order was followed, furthermore, little inhibition was also observed when OP level was added 24 or 48 hours prior to NaPP addition, indicating that NaPP has a little inhibitory effect if added after OP. However, less then 20% of added OP was sorbed by the both studied soils even in the presence of NaPP at or before 12 hours of OP-reaction time. The increase in water-soluble OP-P due to the inhibitory effect of NaPP was calculated by subtracting the amount of water-soluble OP-P in the soils

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Table (2): Phosphorus solubility in alluvial silty clay soil after one and seven wetting cycles.

Treatments	One cycle pH	One cycle soluble-P X10-4 M/L	One cycle Soluble Ca X10-3 M/L	Seven cycles pH	Seven cycles soluble-P X10-4 M/L	Seven cycles soluble Ca X10-3 M/L
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Table (3): Phosphorus solubility in sandy loam soil after one and seven wetting and drying cycles.

Treatments	One cycle			Seven cycles		
	pH	soluble-P X10-4 M/L	Soluble Ca X10-3 M/L	pH	soluble-P X10-4 M/L	soluble Ca X10-3 M/L
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simultaneously with OP rate. Whereas a little effect was observed when the reverse order was followed, furthermore, little inhibition was also observed when OP level was added 24 or 48 hours prior to NaPP addition, indicating that NaPP has a little inhibitory effect if added after OP. However, less than 20% of added OP was sorbed by the both studied soilseven in the presence of NaPP at or before 12 hours of OP-reaction time. The increase in water-soluble OP-P due to the inhibitory effect of NaPP was calculated by subtracting the amount of water-soluble OP-P in the soils

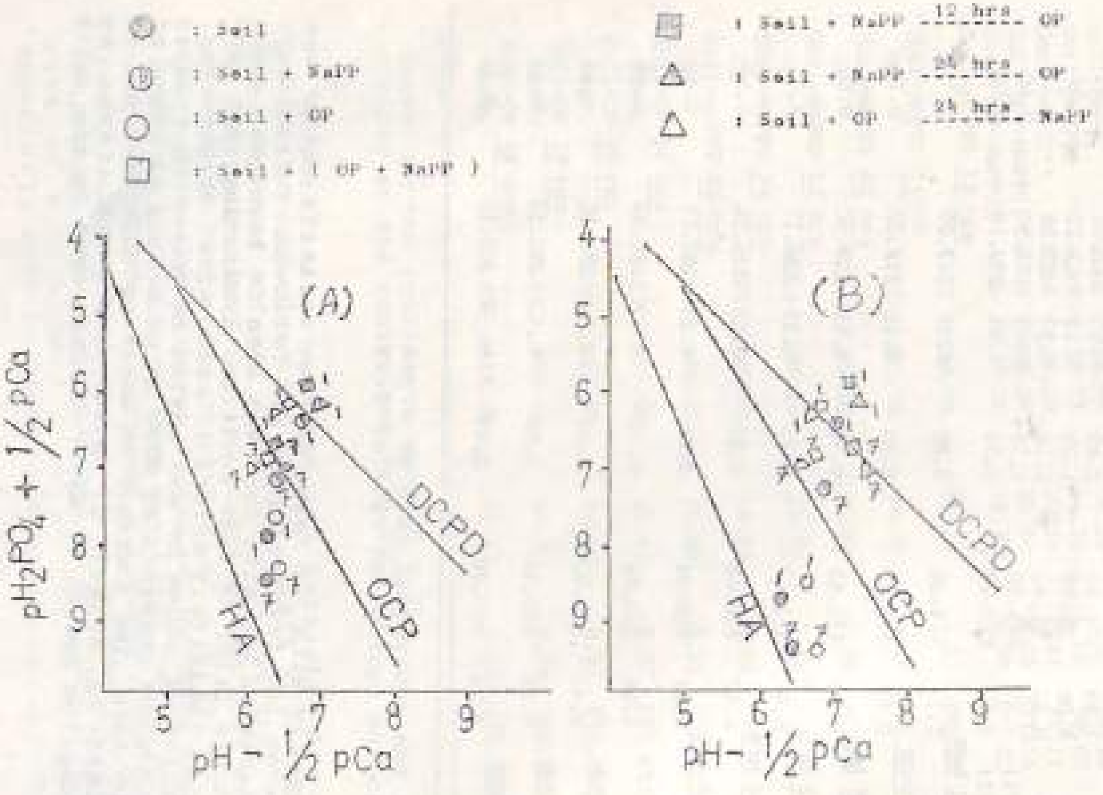

Fig.(1) : Solubility diagrams showing the effect of addition of NaPP (0.25%P) on orthophosphate reaction in alluvial silt clay soil (A) and in desert loamy sand soil (B) applied at rate of 0(●)and 2000 ppm P. Numbers 1 & 7 indicate wetting and drying cycles .

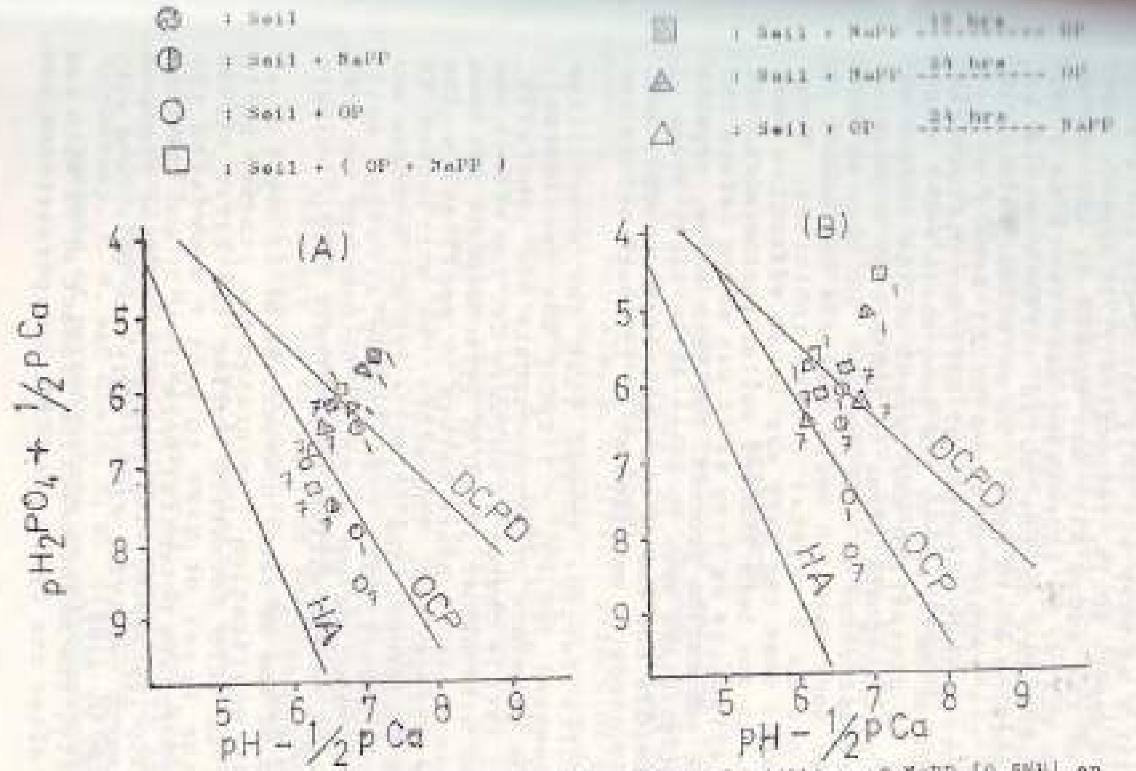

Fig.(2) : Solubility diagrams showing the effect of addition of NaPP (0.5%P) on orthophosphate reaction in alluvial silt clay soil (A) and in desert loamy sand soil (B) applied at rate of 2000 PPm P . Numbers 1 & 7 indicate wetting and drying cycles .

treated with NaPP and OP separately from the amount of water-soluble P in the soils treated with mixture of NaPP and OP. Hence the amount of increase in water-soluble OP-P in the soils treated with NaPP was assumed to by the same with or without OP. It is known that the pyrophosphate hydrolysis in soil is reduced in the presence of OP (Savant and Racz, 1973). Therefore, calculated amount of increase in water-soluble OP-P due to the inhibitory effect of NaPP in the soils cannot be attributed to the hydrolysis of NaPP in the soils.

The data in Table 2 and 3 showed that the recovery of the added (0.25%) P as NaPP was only about 45% and 47% in the alluvial and desert soils, respectively. It appears that more than 50% of the added NaPP was sorbed by the solid surface in both soils. On increasing NaPP level to 0.5%, more adsorption sites on the solid surface would be occupied, and more OP would remain in solution. This is in agreement with the result obtained by El-Zahaby et al. (1982) who reported that sorption of NaPP may distort the surface structure of $CaCO_3$ due to its long-chain P-O-P bonds and the degree of distortion may depend on the amount of NaPP used. The pyrophosphate seems to subject to adsorption and possibly precipitation on soil surface, and more so when added before OP. Such reaction would decrease the amount of NaPP left to act as a crystal growth inhibitor. These results indicated that the NaPP was least effective when added after OP, in the both studied soils. Furthermore, the NaPP remained effective as long as it was added before the starting of the nucleation process. For both soils, crystal growth and precipitation seem to start at an early stage of the reaction before the end of the nucleation process and, therefore, NaPP needs to be added at or 12 hours before the addition of OP.

The results further showed that the monocalcium phosphate potential ($pH_2PO_4 + 1/2pCa$) was considerably lowered by the presence of pyrophosphate and generally, there was very little change in soluble Ca. The effect was more pronounced with higher level of pyrophosphate and when it added within or at 12 hours before OP application. The pyrophosphate effect can, therefore, be attributed to crystal growth inhibition. The $pH_2PO_4 + 1/2\ pCa$ decrease was greater in the desert soil than in the alluvial soil. Difference in P retention and potential between the two soil may be attributed to the difference in their $CaCO_3$, soluble salts and clay contents.

Addition of the OP rate, however, at or 12 hours after NaPP (0.25% P) application saturated the studied soils with respect to DCPD formation during the first cycle. Increasing the level of NaPP to 0.5% P decreased the monocalcium phosphate potential more (Fig. 2) and the soil solution was supersaturated with respect to DCPD as compared that to treatments receiving either only the OP or/and with the first level of NaPP.

Generally, the major reaction product of OP with and without NaPP after one cycle was DCPD, but persistance was less in all treatments that not having NaPP. This observation again suggests the inhibitory of DCPD. The effect of reaction time on the inhibition of NaPP (Figs. 1 and 2) showed that the inhibition was only temporary. The P-solubility in Fig. (1) shows that NaPP failed to act as a crystal growth inhibitor for DCPD after subjecting the soils to wetting and drying cycle and all corresponding points moved slightly below. Pyrophosphate hydrolysis is considered to be relatively fast process (Khasawneh *et al*., 1979). Before its hydrolysis, however, NaPP may act as a crystal growth inhibitor for DCPD compound in calcareous soils and hence enhance the fertilizer P efficiency. Furthermore, the formation of DCPD from water-soluble fertilizers of the higher soluble P contents even after the inhibitory effect disappeared as compared that with OP-treated soils without NaPP during seven wetting and drying cycles.

In agreement with the above findings El-Zahaby and Chien (1982) reported that the smaller crystal size of DCPD formed in the presence of NaPP may explain why more water-soluble P was extracted from the DCPD in the calcareous soils treated with NaPP than without even after the inhibitory effect had disappeared.

Agronomic work is needed to see whether this inhibitory effect would enhance the fertilizer P efficiency for crop response.

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تفاعل سماد الاورثوفوسفيت في الترب الكلسية بتاثير اضافة البايروفوسفات

كاظم مشحوت عواد ** علي حمضي ذياب احلام منصور شعيا

قسم التربة واستصلاح الاراضي - كلية الزراعة - جامعة البصرة

الخلاصة

ان الغرض من الدراسة الحالية هو اختبار التاثير المانع لمركب بايروفوسفات الصوديوم على ترسيب الفسفور في تربتين كلسيتين. ان اضافة مركب البايروفوسفات للترب قيد الدراسة عمل على زيادة تركيز الفسفور حيث كان أكثر فعالية عند اضافته بمستوى (٥ر٠٪) مقارنة بمستوى (٢٥ر٠٪) وبالتحديد عند اضافته مع او قبل اضافة الاورثوفوسفات بـ (١٢) ساعة . ان نتائج الاذابة تؤكد ان الناتج الرئيسي لتفاعل الاورثوفوسفات في الترب الكلسية بوجود أو عدم وجود مركب الاورثوفوسفات وبعد انتهاء دورة واحدة من الترطيب والتجفيف هو مركب فوسفات ثنائي الكالسيوم الذي يكون اقل ثباتية في جميع المعاملات الخالية من مركب البايروفوسفات خلال دورات الترطيب والتجفيف اللاحقة .

وبشكل عام فان مركب البايروفوسفات يعمل كمانع لنمو بلورة مركب فوسفات ثنائي الكالسيوم ويسهم في المحافظة على تركيز عالي للفسفور الذائب حتى بعد انتهاء التاثير المانع لمركب البايروفوسفات.

المجلد (٥) العدد (٢) ١٩٩٢

مجلة البصرة للعلوم الزراعية