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Phosphorus Fractions in some Calcareous Soils of Egypt as Affected by Aging and their Properties

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IDENTIFYING the predominate individual forms of inorganic P in soils is helpful in determined potential availability of P in calcareous soils. Therefore, the current investigation was conducted to study the distribution of P amongst the different fractions of some calcareous soils as affected by period of soil cultivation and also the extent to which some soil properties affect such a distribution. Fulfilling such objectives was executed by carrying out a P fractionation experiment whose results showed that AB-DTPA extractable fraction (available) was highly and significantly affected by Fe,Al-P fraction. Ca-P fraction was very much greater than the other P-fractions in all the investigated soils. Within all the investigated soils, the Fe-Al-bound fraction contained P in concentrations far lower than the Ca-bound fraction. Occluded- P was found in the lowest concentrations. The residual P fraction was found in quantities almost equal to or even exceeding the other total extractable fractions. Fe, Al-P fraction correlated at a high level of significance with each of the occluded-P and residual-P fraction as well as the total-P content. This means that Fe, Al-P fraction can be under certain circumstances converted to either of both of occluded and residual fractions. Occluded-P fraction showed highly significant correlations with each of Fe, Al-P and residual-P fractions and at the same time, it correlated but only at a significance level of 5% with total-P. Non- of the investigated P-fractions was affected by either of the studied properties solely but more than of these properties contributed together to affect the different soil P fractions. For example, Fe, Al-P correlated significantly with sand, silt, $CaCO_3$ and EC while each of the occluded-P, residual-P and total-P content showed significant correlation with $CaCO_3$, EC and CEC together. Ca-P fraction was affected by sand, clay and EC. The relationships between AB-DTPA extractable fraction (the available P fraction) and the different soil properties indicate that both silt and $CaCO_3$ contents significantly and negatively affected the AB-DTPA extractable P. It can be deduced that the different soil properties, although varied in their contribution to determine the amount of AB-DTPA extractable P, yet the role of either of these properties cannot be neglected.

Keywords: P fractionation, Calcareous soils, Long-term fertilization, Soil properties.

Although the total P content in most soils can be large, only a small fraction is available or in an organic form for biological utilization because it is bound either in incompletely weathered mineral particles, adsorbed on mineral surfaces, or, over the time of soil formation, made unavailable by secondary mineral formation (occluded) (Yang *et al*., 2013). Also, phosphorus as an essential nutrient for plant growth and crop production when added to soil as phosphate fertilizers is very quickly and chemically fractionated among different soil fractions (Abbas, 1993, Mostashari *et al*., 2008, Sheng-Li *et al*., 2008, Wright, 2009 and Eger *et al*., 2013).

The inventory of soil phosphorus is subject to significant changes over time. Knowledge of transformation of phosphorus added to soil and the effect of soil properties on the transformation have become essential for long-term planning of fertilization strategies to sustain crop production (Sheng-Li *et al*., 2008 and Jalali and Matin, 2013).

Chemical fractionation of soil phosphorus provides a method for identifying the predominate individual forms of phosphorus in soils, most commonly soluble P, Al-P, Fe-P, occluded P and Ca-P (Olsen and Sommers, 1982, Abbas, 1993 and Mostashari *et al*., 2008).

Solis and Torrent (1989) found that occluded P (O-P) extracted with citrate dithionate bicarbonate (CDB) was associated with the crystal lattice of Fe-P minerals. Samadi and Gilkes (1999) reported that Al-P and Fe-P were amongst the most important forms of phosphorus in virgin and fertilized calcareous soils of Western Australia. Wright (2009) upon his study on effect of land use on phosphorus distribution among the soil chemical fractions, pointed out that increasing soil disturbance and long-term fertilization increased P allocation to inorganic fractions as Ca bound P contained 49% of total phosphorus for sugarcane but 28% for other land uses.

Phosphorus adsorbed or precipitated with Ca, Fe and Al is stable when soils are maintained under conditions leading to phosphorus fixation, but may be susceptible to dissolution and regeneration upon change in land use or environmental conditions. Of particular concern are land use changes that disturb soil and alter redox reactions which enhance phosphorus regeneration and potential for leaching and runoff (Moore and Reddy, 1994).

Samavati and Hossinpur (2006) showed that available P extracted by Olsen method was significantly correlated with (Ca-P) and (Al,Fe-P) which indicates that these fractions can be probably used by plant.

The objectives of this investigation were to study (i) the distribution of P amongst the different soil fractions as affected by period of soil cultivation (ii) the relationship between the determined soil fractions and some soil properties of the calcareous soils under study which vary considerably in their contents of $CaCO_3$.

Material and Methods

Material of study

Twelve surface soil samples (0-30 cm) varying in their cultivation period, chemical and physical properties were collected from different localities in Egypt to represent calcareous soils. These soils are classified as Typic Haplocalcid soil. The soil samples were air dried, crushed, sieved through a 2 mm sieve then stored in air-tight jars.

Methods of soil analyses

Particle size distribution was conducted by pipette method using Na-hexametaphosphate as a dispersing agent as described by Klute (1986). Organic matter content was estimated by Walkley and Black method, total carbonates was determined using Collin's calcimeter, pH was determined electrometrically in 1:2.5 soil – water suspension using a pH meter, electrical conductivity of the soil paste extract was determined using electrical conductivity bridge and cationic and anionic composition of the soil paste extract were determined using the standard methods outlined by Page *et al*. (1982). Cation exchange capacity (CEC) was determined by displacing the exchangeable cations by NaCl as described by Polemio and Rhoades (1977). Total P was determined according to Olsen and Sommers (1982), AB-DTPA extractable fraction (the available P fraction) was determined according to Soltanpour and Schwab (1977). Inorganic P fractions were determined using the fractionation procedure of Olsen and Sommers (1982), involving sequential extraction with 0.1 N NaOH to remove non-occluded Fe,Al-P, 1.0 M NaCl and citrate bicarbonate (CB) to remove P sorbed by carbonate during NaOH extraction, citrate dithionate-bicarbonate (CDB) to remove P occluded within Fe and Al oxides and hydrous oxides and finally 1.0 N HCl to remove Ca-P. Some physical and chemical properties of the studied soils are shown in Table 1.

Results and Discussion

Soil properties

Data presented in Table 1 reveal that particle size distribution of the studied soils was slightly affected with prolonging period of cultivation, however, it could be noticed that percentage of the fine fraction tended to increase with time. Likewise, organic matter content seemed more higher while $CaCO_3$ content slightly decreased with time. The usual agricultural practices account for such changes. Successive irrigations might led to dissolution of a portion of $CaCO_3$, fertilization and manuring beside of the plant residues give reason for increase of the organic matter and also the consequent increase in specific area of the investigated soils.

TABLE 1. Physical and chemical properties of the investigated soils.

Soil No.	Soil Location	Cultivation period (year)	Particle size distribution (%)			Textural class	Calcium carbonate (gkg^{-1})	Organic matter (gkg^{-1})	EC (dS m^{-1})*	pH (1:2.5) soil-water suspension	CEC cmol$_c$ kg^{-1}	Total P mg P kg^{-1} soil	AB-DTPA mg P kg^{-1} soil
			Sand	Silt	Clay								
1	El-Noubaria	1	84.82	3.68	11.50	SL	293.3	4.7	2.12	8.01	6.98	772.4	14.6
2		5	83.01	3.72	13.27	SL	237.8	4.9	2.26	8.11	7.08	1268.4	17.5
3		10	82.21	3.92	13.87	SL	201.2	5.45	2.69	8.09	7.80	1398.4	20.1
4	Sidi-Barani	1	72.28	13.29	14.43	SL	237.9	6.1	2.16	8.25	6.10	523.5	10.1
5		5	70.11	14.61	15.28	SCL	216.3	6.7	2.29	8.20	6.48	1096.2	15.0
6		10	70.00	13.02	16.98	SCL	201.4	9.3	2.58	8.32	6.72	1125.7	16.3
7	El-Amerya	1	56.26	19.11	24.63	SCL	293.2	4.9	3.54	7.98	10.12	604.4	14.0
8		5	53.15	20.00	26.85	LC	256.7	5.3	3.93	7.99	11.00	1183.4	12.2
9		10	51.28	18.33	30.39	LC	215.7	6.4	4.7	7.79	11.30	1254.6	16.1
10	El-Hamam	1	62.12	21.22	16.65	CL	456.3	2.9	1.09	8.22	8.20	527.0	8.8
11		5	60.18	20.98	18.84	CL	431.9	3.3	1.25	8.11	8.70	1334.8	9.3
12		10	58.92	20.18	20.90	CL	421.7	3.9	1.57	8.09	9.30	1367.7	10.1

* Soil paste extract, SL: Sandy Loam , SCL: Sandy Clay Loam, LC: Light Clay, CL: Clay Loam .

TABLE 1. Contd.

Soil No.	Soil Location	Cultivation period (year)	Soluble cations ($mmol_c L^{-1}$)*				Soluble anions ($mmol_c L^{-1}$)*			
			Ca^{2+}	Mg^{2+}	Na^+	K^+	CO_3^{2-}	HCO_3^-	Cl^-	SO_4^{2-}
1	El-Noubaria	1	10.80	2.62	7.60	0.15	0.00	4.21	14.00	2.96
2		5	10.88	1.22	10.22	0.32	0.00	3.98	17.08	1.58
3		10	13.22	1.03	12.31	0.29	0.00	4.00	22.11	0.74
4	Sidi-Barani	1	8.35	4.18	8.70	0.40	0.00	3.68	10.28	7.67
5		5	9.11	4.08	9.23	0.45	0.00	3.22	12.16	7.49
6		10	10.01	4.12	11.15	0.50	0.00	4.01	14.54	7.23
7	El-Amerya	1	8.98	9.22	16.00	1.22	0.00	2.98	17.20	15.24
8		5	10.19	9.45	18.65	1.01	0.00	3.68	18.62	17.00
9		10	13.48	9.30	23.13	1.13	0.00	4.30	20.70	22.67
10	El-Hamam	1	4.33	1.12	3.83	1.63	0.00	0.98	7.32	2.61
11		5	5.28	1.29	4.28	1.68	0.00	1.36	9.25	1.92
12		10	6.12	1.11	6.71	1.78	0.00	1.42	10.12	4.18

*Soil paste extract.

pH values were slightly changed owing to the buffer capacities of the investigated soils whereas EC and soluble ions concentrations changed in different ways due to the variation in the agricultural practices followed from a site to another and from a crop to another. Unefficient or restricted drainage on one hand and chemical composition of the water used for irrigation and fertilizer treatments on the other one, are additional causes of the changes occurred in chemical properties of the investigated soils.

Distribution of P amongst the soil fractions

Results in Table 2 illustrate the phosphorus distribution among the different soil fractions. Trends in P distribution among soil chemical fractions were similar among the different soil locations regardless of period of cultivation which only affected the magnitude of P found in each soil fraction. However, it can be deduced that distribution of P among the soil fractions is dependent on the indigenous contents of these fractions in soil. AB-DTPA extractable fraction (available) was highly and significantly affected by Fe,Al-P fraction. The other P- fractions although did not affect significantly the amount of AB-DTPA extractable fractions, yet these fractions contributed to this fraction to different extents, the highest was due to the residual P- fraction while the lowest was due to Ca-P fraction. Such a finding may lead us to conclude that the Ca-P fraction is hardly to be converted into AB-DTPA extractable fraction (i.e. the available P-fraction). However, Mostashari et al. (2008) showed that available P extracted by Olsen method was significantly correlated with Ca-P and concluded that this fraction i.e. Ca-P probably can be used by plant. Also, Samavati and Hossinpur (2006) reported that available P was significantly correlated with Ca-P. Therefore, Ca-P fraction was very much greater than the other P-fractions in all the investigated soils. This result is in line with those of Wright (2009) who indicated that Ca-bound P contained 49% of total P in some soils. This is expected in alkaline and calcareous soils where it seems that Ca^{2+} ions control the phosphate reaction through the solubility product (Abbas, 1993). Also, Zhang *et al*. (2012) found that Calcium bound phosphorus (Ca–P) was the main fraction of inorganic P for all the soils samples, the rank order P-fractions was Ca–P > organic phosphorus > Phosphorus bound to Al, Fe, and Mn oxides and oxyhydroxides (Fe/Al–P). Wright (2009) went almost to a similar finding and outlined that the calcareous nature of the investigated soils tended to promote the sequestion of P into the Ca-bound rather than the Fe-Al-bound fraction.

Within all the investigated soils, the Fe-Al-bound fraction contained P in concentrations far lower than the Ca-bound fraction. This low Fe,Al- P contents could be explained on a basis of pH effect where the low pH soils . the acidic soils contain P associated with amorphous hydroxide surface and crystalline Fe and Al oxides which are features not dominant in the calcareous soils. However, it is worthy to mention that P contained in this fraction may be unstable with fluctuating redox conditions (Moore and Reddy, 1994), thus, there may be considerable movement of P into and out of this fraction depending on environmental conditions for these soils (Wright, 2009).

TABLE 2. Distribution of phosphorus among different soil fractions as affected by aging (period of soil cultivation).

Soil No.	Soil Location	Cultivation period (year)	Inorganic P-fraction (mgPkg^{-1} soil)				Residual-P	Total P
			Fe, Al-P	Occluded-P	Ca-P	Total extractable fractions		
1	El-Noubaria	1	29.5	11.0	381.7	422.2	350.2	772.4
2		5	64.6	24.2	436.0	524.8	743.6	1268.4
3		10	69.3	29.6	509.1	608.0	790.4	1398.4
4	Sidi-Barani	1	18.4	16.5	218.1	253.0	270.5	523.5
5		5	53.5	23.3	410.7	487.5	608.7	1096.2
6		10	53.6	25.6	421.3	500.5	625.2	1125.7
7	El-Amerya	1	20.5	11.2	293.5	325.2	279.2	604.4
8		5	33.9	12.1	509.2	555.2	628.2	1183.4
9		10	37.6	15.6	581.7	634.9	619.7	1254.6
10	El-Hamam	1	11.1	15.7	253.1	279.9	247.1	527.0
11		5	38.4	21.8	631.8	692.0	642.8	1334.8
12		10	40.9	29.0	649.5	719.4	648.3	1367.7

However, Ryan *et al*. (1985) showed that P sorption in calcareous soils was related to oxalate- extractable Fe, *i.e*. amorphous forms of iron oxides.

Occluded- P was found in the lowest concentrations probably because it is associated the crystal lattice of Fe-P minerals such as strengite which is probably found in little amounts or completely absent in the investigated soils. Also, P can be occluded within coatings of Fe-oxides and hydrated oxides (Page *et al*., 1982).

The residual P fraction was found in quantities almost equal or even exceeding the summation of the other extractable fractions. Since the residual P fraction is generally considered to be a stable and recalcitrant chemical fraction (Turner *et al*., 2005), it is expected therefore, to find that most of P applied to soil in an available form will be converted into more stable chemical fractions. On the other hand, Bowman *et al*. (1978) reported that the actual availability of residual P fraction should be estimated by appropriate availability indices.

Data presented in Table 3 reveal that Fe, Al-P fraction correlated at a high level of significance with each of the occluded-P and residual-P fractions as well as the total-P content. This means that Fe, Al-P fraction can be under certain circumstances converted to either or both of occluded and residual fractions. This result stands in well agreement with those of Moore and Reedy (1994) and Wright (2009) who declared that there is a case of equilibrium between P bound Fe, Al fraction and P bound to the other fractions *i.e*. there is a reverse movement of P into and out of this fraction. The highly significant correlation between the Fe, Al-P fraction and the total-P fraction is expected since Fe, Al-P fraction represents considerable component of the total-P content and therefore, reversible relationship between these two variables is a logic one.

Occluded-P fraction showed highly significant correlations with each of Fe, Al-P and residual-P fractions and at the same time, it correlated but only at a significance level of 5% with total-P. Accordingly, it can be said that reversal significant relationships are present between occluded-P fraction and the aforementioned ones. Ca-P fraction seemed not to correlate with any of the other P-fraction except for the total-P content since this fraction is a component of the total-P content itself.

TABLE 3. Simple correlation coefficient ® between P-fractions of the investigated soils .

P- fraction	Fe, Al-P	Occluded-P	Ca-P	Residual-P	Total-P	AB-DTPA
Fe, Al-P	1.000**	0.758**	0.240	0.897**	0.796**	0.755**
Occluded-P	0.758**	1.000**	0.260	0.709**	0.667*	0.299
Ca-P	0.240	0.260	1.000**	0.538	0.672*	0.015
Residual-P	0.897**	0.709**	0.538	1.000**	0.970**	0.529
Total-P	0.796**	0.667*	0.672*	0.970**	1.000**	0.397
AB-DTPA	0.755**	0.299	0.015	0.529	0.397	1.000**

Relationships between the different P-fractions and different soil properties:

Data presented in Table 4 reveal that non- of the studied P fractions was affected significantly by any property of the studied soils. Such insignificant relationships do not mean a complete absence for the effect of the studied soil properties on distribution of P among the different soil fractions but may suggest that the distribution of P among the different soil fractions is a final product of the contribution of the soil properties together. Therefore, such a suggestion was put under examination through calculation of the stepwise regression equations.

TABLE 4. Simple correlation coefficient ® between P-fractions and soil properties of the investigated soils .

Soil property	Fe, Al-P	Occluded-P	Ca-P	Residual-P	Total- P	AB-DTPA
Sand	0.541	0.33	-0.174	0.208	0.083	0.556
Silt	-0.563	-0.233	0.153	-0.248	-0.114	-0.717**
Clay	-0.261	-0.34	0.37	0.061	0.17	-0.139
CaCO$_3$ content	-0.506	-0.054	0.133	-0.306	-0.149	-0.787**
Organic matter content	0.389	0.174	-0.097	0.227	0.115	0.537
EC	0.079	-0.366	0.121	0.16	0.141	0.477
CEC	-0.238	-0.343	0.457	0.103	0.232	-0.124
pH	-0.039	0.264	-0.408	-0.198	-0.283	-0.335

The results of stepwise regression shown in Table 5 assure the previous suggestion *i.e.* each of the investigated P-fractions was affected by some, and not all, soil properties. For example, Fe, Al-P correlated significantly (0.782^{**}) with sand, silt, $CaCO_3$ and EC while occluded-P seemed to be of significant relationships with $CaCO_3$, EC and CEC, respectively. Residual-P was affected by $CaCO_3$, EC and CEC. Total-P content showed significant correlation with $CaCO_3$, EC and CEC together. Ca-P was affected by sand, clay and EC. The simple correlation coefficients describing the relationships between AB-DTPA extractable fraction (the available P fraction) and the different soil properties indicate that both silt and $CaCO_3$ contents significantly and negatively affected the AB-DTPA extractable P. The negative significant relationship between available P content and $CaCO_3$ content was reported by (Farragallah, 2011).

TABLE 5. Stepwise regression equivalents between P-fractions and soil properties of the investigated soils .

P- fraction	Model	R^2
Fe, Al-P	162 - 4.82 X2 + 11.1 X3 - 0.369 X4 - 61.1 X6	0.782^{**}
Occluded-P	65.2 - 0.240 X4 - 26.8 X6 + 10.9 X7	0.753^{**}
Ca-P	- 2353 + 26.8 X1 + 86.0 X3 - 233 X6	0.704^{**}
Residual-P	1648 - 8.02 X4 - 815 X6 + 392 X7	0.839^{**}
Total-P	2588 - 12.9 X4 - 1358 X6 + 673 X7	0.758^{**}
AB-DTPA	16.49 – 0.017 X1 – 0.063 X2 + 0.39 X3	0.750^{**}

X1: sand, X2: silt, X3: clay, X4: $CaCO_3$, X6: EC, X7: CEC .

However, irrespective to the aforementioned significant relations, the other soil properties i.e. the sand and organic matter contents as well as the CEC affected but insignificantly the AB-DTPA extractable P fraction. On the other hand, insignificant positive correlations characterized the relationships between AB-DTPA extractable P and each of the soil cation exchange capacity and soil pH. The negative relationship between available P and soil pH was reported also by many investigators among them (Farragallah, 2011) who stated, highly significant negative correlations were obtained between soil pH and available P in the studied soils, thus it can be deduced that the different soil properties, although varied in their contribution to determine the amount of AB-DTPA extractable P, yet the role of either of these properties cannot be neglected. Moreover, the stepwise regression analysis resulted in a significant multiple correlations between AB-DTPA extractable P and sand, silt and clay together as shown in Table 5. Accordingly, it can be pointed out that these properties could contribute together to determine the AB-DTPA extractable P *i.e.* the available P content.

Conclusion

The total P content in most soils is distributed among the different soil fractions. Only a small fraction is available. However, a magnitude of the available P content is affected by aging beside of some physical and chemical properties. Moreover, the different soil properties, although varied in their contribution to determine the amount of AB-DTPA extractable P, yet the role of either of these properties cannot be neglected.

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الصور الكيميائية للفوسفور فى بعض الأراضى الجيرية بمصر والمتأثرة بالزمن وخواص التربة

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يعتبر التعرف على الصور السائدة من الفوسفور الغير عضوي في التربة عاملاً مهماً في تقدير مدى الفوسفور الميسر فى الأراضى الجيرية، وبالتالي أجرى هذا البحث لدراسة توزيع الفوسفور بين الصور المختلفة الموجودة في الأراضي الجيرية وإلى أى مدى تؤثر فترة إستزراع التربة وكذلك خواص التربة على مثل هذا التوزيع. ولتحقيق ذلك أجريت تجربة لدراسة توزيع الفوسفور بين مكونات التربة المختلفة أوضحت نتائجها أن الجزء الفوسفاتى المستخلص بواسطة AB-DTPA (الميسر) قد تأثر بدرجة معنوية بكل من الفوسفور المرتبط بالحديد والألومنيوم، وكان الفوسفور المرتبط بالكالسيوم أكثر صور الفوسفور تواجدا عن باقى الصور الآخرى فى الأراضي موضع الدراسة، واحتوت هذه الأراضى على الفوسفور المرتبط بالحديد والألومنيوم بتركيزات أقل كثيراً من الفوسفور المرتبط بالكالسيوم، أما الفوسفور المغطى بالأكاسيد occluded فقد تواجد بأقل التركيزات، وقد تواجد الفوسفور المتبقى بكميات تتساوى تقريباً وحتى أحياناً تفوق تلك التى تتواجد على الصور الآخرى مجتمعة والتى تم إستخلاصها.

وعلي الجانب الأخر أوضحت النتائج أن الفوسفور المرتبط بالحديد والألومنيوم إرتبط بمستوى عالى من المعنوية مع كل من الفوسفور المغطى بالأكاسيد والفوسفور المتبقى وكذلك الفوسفور الكلى مما يعنى أن الفوسفور المرتبط بالحديد والألومنيوم يمكن تحت ظروف خاصة أن يتحول لأى من صورتى الفوسفور المرتبط بالأكاسيد أو المتبقى. وبوجه عام فإن أى من صور الفوسفور المقدرة لم يتأثر بأى من خواص التربة كل على حدة ولكن أكثر من واحدة من تلك الخواص تشاركت معاً فى التأثير على صور الفوسفور المختلفة. فعلى سبيل المثال إرتبط الفوسفور المرتبط بالحديد والألومنيوم معنوياً مع محتوى التربة من كل من الرمل والسلت وكربونات الكالسيوم والتوصيل الكهربى مجتمعة، فى حين أن كلاً من الفوسفور المغطى بالأكاسيد والفوسفور المتبقى والفوسفور الكلى إرتبط بعلاقات معنوية مع كربونات الكالسيوم والتوصيل الكهربى والسعة التبادلية الكاتيونية مجتمعة، ولقد تأثر الفوسفور المرتبط بالكالسيوم بكل من محتوى التربة من الرمل والطين والتوصيل الكهربى مجتمعة. وقد أظهرت العلاقة بين الفوسفور الميسر والمستخلص بواسطة AB-DTPA من جهة وخواص التربة المختلفة من جهة أخرى أن كل من السلت وكربونات الكالسيوم قد أثرا معنوياً وسلبياً على الفوسفورالمستخلص بواسطة AB-DTPA ويمكن إستنباط أن خواص التربة المختلفة بالرغم من أنها قد إختلفت فى درجة مشاركتها فى تحديد كمية الفوسفور الميسر والمستخلص بواسطة AB-DTPA إلا أن دور أى من هذه الخواص لايمكن إهماله.