Environment, Biodiversity & Soil Security

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Impacts of P inputs on availability of Fe, Mn, Zn and Se in soils

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PHOSPHATE IONS are hard Lewis-bases, which might not preferably bind with Fe, Mn, and Zn (borderline Lewis-acids); though P-applications diminish considerably the availability of micronutrients and some other elements such as selenium. Interactions between P and these elements are still in need for more detailed investigations. Therefore, the current study was executed as a trial to through more light on such mutual-relations. A pot experiment was, therefore, conducted on a clayey non-calcareous soil and a sandy calcareous one, following a complete-randomized design to attain this aim. These soils were enriched with 5 mg Fe, 1 mg Mn, 1.5 mg Zn, and 10 mg Se kg^{-1}; thereafter, they received elevated P-doses (15, 30, 60, and 120 mg P kg^{-1}) and incubated for 72 h while keeping soil moisture gravimetrically at field capacity throughout this study. Key results revealed that AB-DTPA-extractable-P increased significantly with increasing the rate of applied-P and such increases were noticeable with aging. In contrast, AB-DTPA-Fe content was not affected significantly by P-applications. Regarding AB-DTPA-extractable-Mn and Zn, their contents increased progressively in the non-calcareous soil upon application of 60 mg P kg^{-1} soil (P_{60}) or higher for an incubation period extended up to 48h while remained statistically unchangeable in the non-calcareous soil. In both soils, AB-DTPA-Se was not significantly affected by the dose of applied-P, yet this available-fraction was affected by soil ageing showing fluctuations in form of cycles of increases and decreases. More experiments are needed using additional time sequences within the first 48h of P-application

Keywords: phosphate fertilizers; Available-Fe; Available-Mn; Available-Zn; Available-Se.

1. Introduction

Phosphorus is a crucial component of plant growth and productivity (Bindraban *et al.* 2020; Abdelhafez *et al.* 2021; Owodunni *et al*. 2023; Sharma *et al*. 2023). It is applied continuously and in excess as chemical fertilizers to attain optimum yield (Abdalla *et al*. 2022; El-Ramady *et al*. 2022; Wendimu *et al*. 2023); nevertheless, these additives diminish considerably availability of other nutrients (Wahba *et al*. 2019; Abd El-Aziz *et al*. 2020), such as Fe, Mn (Rutkowska *et al.* 2014) and Zn (Ahn *et al*. 2015; Zhang *et al*. 2017). Despite that high P-inputs enhance considerably plant growth and productivity, especially in soils of low P content (Chien *et al*. 2011). These results could be confusing as the law of minimum states that all essential nutrients should be provided in adequate and balance amounts to attain optimum growth (Shahid *et al*. 2016; Koch *et al*. 2020).

Retention of inorganic P takes place rapidly after soil application (Harvey and Rhue 2008; Farid *et al.* 2023) and reaches an almost stable state after 24 h of application (McGechan and Lewis 2002). Mostly, adsorption/desorption reactions and precipitation/dissolution processes are responsible for P precipitation within this short time period (Lair *et al*., 2009). Phosphate ions act as hard Lewis bases (Wang *et al*. 2021), while many micronutrients such as Fe, Mn and Zn, are considered as borderline Lewis acids (Appenroth 2010). Based on the HSAB principle, extra stabilization takes place among ions of

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Received: 11/07/2023; Accepted: 10/08/2023
DOI: 10.21608/JENVBS.2023.222204.1224

a hard–hard combination, or a soft–soft one (Pearson 2005). For this reason, we anticipate that phosphate ions may not preferably bind with micronutrients and therefore formation of these complexes may take longer time periods than expected. In this context, substantial hysteresis was noticed between Zn and P (Zhao and Selim 2010). Also, P availability was noticed to follow consecutive cycles of increasing/decreasing in soils within short time periods after application (Ahmed *et al*. 2013).

Concerning Se, it is a beneficial element for plants (El-Ramady *et al*. 2021 & 2023; Elshinawy et al 2023; Sári *et al*. 2023) and therefore all fertilizers in Finland have been enriched with this element since 1984 (Keskinen *et al.* 2009). Although, Se reacts fast with oxygen forming two major oxoanions ($SeO^{2-}{}_4$ and $SeO^{2-}{}_3$) (Nakamaru and Sekine 2008); yet, Se-oxides can be easily reduced because of the lack of π-bond (Reich and Hondal, 2016). A point to note is that Se oxoanions can be substituted easily on soil particles by phosphate ions to be set free in soil solution (Nakamaru and Sekine 2008; Keskinen *et al*. 2009).

The current study represents a trail towards evaluating the impacts of amending soils differing in their properties (a clayey non-calcareous soil and a sandy calcareous one) with chemical P fertilizer at elevated doses on AB-DTPA extractable Fe, Mn, Zn and Se contents within the first 72 h after application. Specifically, we anticipate that adding P fertilizers increases the available indices of micronutrients in soil within a short time period after P- application (<24 h), especially with increasing the dose of applied P because major soil cations such as Ca compete with micronutrients on sorption sites; yet in presence of high P-inputs, these cations precipitate in in the form of insoluble phosphate salts. Hence sorption of micronutrients on soil particles substantially intensifies and as a result their availability increases (hypothesis 1). Later (>24h), these micronutrients precipitate in the form of insoluble phosphate soils; so their availability decreases (hypothesis 2). Likewise, Se bioavailability increases in soil owing to application of P fertilizers because of the competitive sorption behavior between phosphate ions and Se oxoanions (hypothesis 3). We believe that the results of this study might improve our knowledge about the impact of phosphate additives on micronutrient availability in soil within the first 72 h after application.

2. Materials and Methods

2.1. Materials of study

Two surface soil samples (0-30 cm) were collected to attain the aim of the study i.e. a clay non-calcareous soil from the experimental farm of the Faculty of Agriculture, Benha University, Qalubia Governrate, and a sandy calcareous one from El-Noubaria, El-Behira governorate, Egypt. These samples were air-dried, crushed and sieved via a 2-mm sieve, then analyzed for their physical and chemical properties as outlined by Sparks *et al*. (1996) and Klute (1986), respectively and the results are presented in Table 1.

Table 1. Particle size distribution and chemical properties of the investigated soils.

Property	Clayey non calcareous soil	Sandy Calcareous soil
Particle size distribution		
Sand%	35.9	93.2
Silt%	17.3	3.6
Clay%	46.8	3.2
Textural class	clay	Sand
Organic matter ($g\ kg^{-1}$)	10.07	3.35
Calcium carbonate ($g\ kg^{-1}$)	38.5	229
pH*	7.28	8.14
EC** ($dS\ m^{-1}$)	1.33	8.96
Field capacity (%)	61.45	32.89
AB-DTPA extractable elements ($mg\ kg^{-1}$)		
P	12.03	7.87
Fe	25.74	20.12
Mn	20.36	18.28
Zn	1.12	1.26
Se	0.31	0.26

* Soil pH was determined in soil: water suspension (1:2.5), ** while soil EC was measured in soil paste extract

Soil samples were then mixed with 5 mg Fe kg^{-1} ($FeSO_4.7H_2O$, Sigma-Aldrich), 1 mg Mn kg^{-1} ($MgSO_4$. H_2O, Reidel-de Haën), 1.5 mg Zn kg^{-1} ($ZnSO_4$. $2H_2O$, Sigma-Aldrich) and 10 mg Se kg^{-1} (Selenium (IV) oxide, ACROS chemicals).

2.3. Experimental procedure

Hundred-gram portions of artificially contaminated soils were placed uniformly in plastic pots (6 cm diameter × 12 cm height) and then received P inputs in the form of KH_2PO_4 (purity >99%) at either of the following rates: 15, 30, 60 or120 mg P kg^{-1}. The experimental design was a complete randomized one of 12 replicates per treatment. Soil moisture was kept gravimetrically at the field capacity throughout this investigation which lasted for 72 h.

Three pots were collected from the abovementioned treatments at each of the following periods: 0, 24, 48, and 72 h after P application to determine their AB-DTPA extractable contents of the considered micro nutritive elements and Se, where 10-gram portion of soil were collected from each pot and placed in a plastic bottle together with 20 mL of AB-DTPA (0.005 *M*). This mixture was shaken for 30 minutes. then centrifuged according to Soltanpour (1991) and their contents of available Zn, Mn, Fe, and Se were determined by atomic absorption spectrophotometer (AAS) (UNICAM 929 AA spectrometer). Likewise, available phosphorus (P) was extracted by AB-DTPA and then determined following the ascorbic acid method via spectrophotometer (spectronic20D)

2.4. Data analyses

Chemicals of study were of analytical grade. All treatments were conducted in triplicates and the data were analyzed via SPSS statistical software (ver 18) using two-way ANOVA and Dunken's post-Hoc test to compare among means. Thereafter, data were presented graphically by Sigma Plot 10 software.

3. Results and Discussion

3.1. Effect of dose of the applied P and aging of its application on AB-DTPA extractable Fe

AB- DTPA extractable-Fe was not affected significantly by either the dose of applied P or even soil aging in both the clayey non-calcareous soil and the sandy calcareous one, yet the interaction between these two factors was of significant effect only in the non-calcareous soil (Fig 1A). In this concern, the highest increases in AB-DTPA extractable Fe were achieved after 24 h of applying the lowest doses of P (P_{15} and P_{30}); subsequently, AB-DTPA-Fe decreased considerably (Fig 1). In case of the higher applied doses of P (P_{60} and P_{120}), the highest increases in AB-DTPA extractable Fe were obtained after 48 h of P-application.

It is well known that Fe tends to combine with inorganic P (P_i) (Shaheen *et al*. 2022; Wang *et al*. 2022a) forming insoluble salts such as vivianite, strengite, and amorphous Fe–P precipitates (Yang *et al*., 2023). These reactions speed up with increasing the solubility of both P and Fe under acidic soil conditions (Ding *et al.* 2023); but not on the insoluble ferric minerals (Lemos *et al.* 2022

The slightly alkaline conditions of the investigated non-calcareous arid soil probably increased P fixation (Ahmed *et al*. 2013; Wang *et al*. 2022b) and, at the same time, lessened considerably Fe mobility (Vélez-Bermúdez and Schmidt, 2023). Thus, reactions between P and Fe might take longer time periods than expected. For this reason, no significant variations were noticed in AB-DTPA-extractable Fe owing to increasing the dose of P-applications within this short period of study. On the other hand, flocculation in P available content throughout the experimental period might be the main reason beyond variations in AB-DTPA-Fe with soil aging

In the calcareous soil, its alkaline conditions promoted Fe oxidation (Zhang *et al*. 2022; Molnár *et al*. 2023). Also, calcite surfaces therein exhibit high affinity to sorb Fe (Abbas and Salem 2013; Rasheed 2023) and this might account for extensive limitations for the available indices of both P and Fe in soil as well as their interactions (Luo *et al*. 2022). Accordingly no significant variations were detectable for the applied P-dose, aging of its application or even the interaction between these two factors on AB-DTPA-Fe in the calcareous soil.

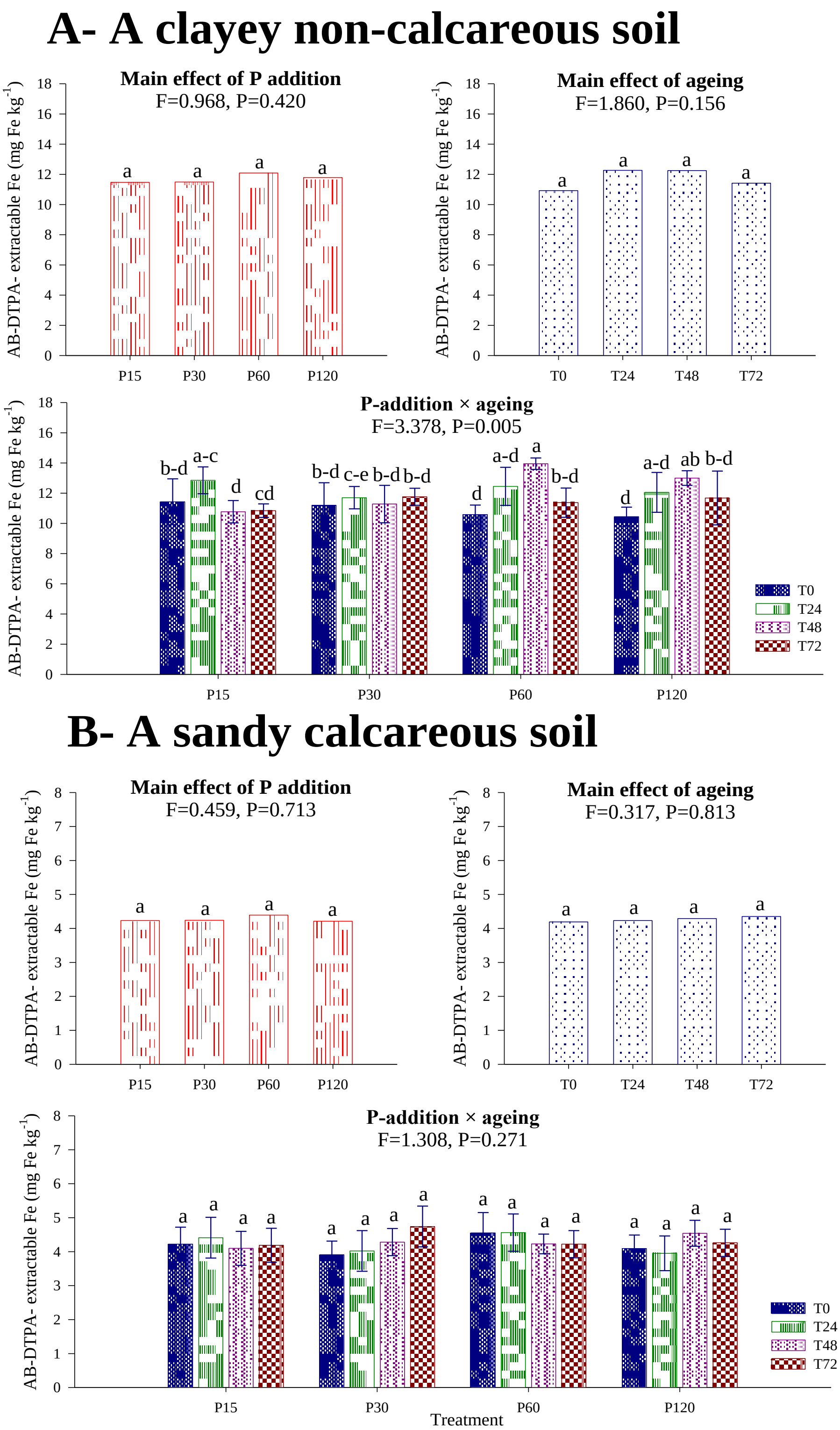

Fig. 1. AB-DTPA extractable Fe from the investigated soils as affected by increasing dose of the applied P and aging of its application. Note: P_{15}: 15 mg P kg^{-1}, P_{30}: 30 mg P kg^{-1}, P_{60}: 60 mg P kg^{-1}, P_{120}: 120 mg P kg^{-1}. Similar letters indicate no significant variations among treatments.

3.2. Effect of dose of the applied P and ageing of its application on AB- DTPA extractable Mn

Application of P significantly raised AB-DTPA extractable-Mn in the non-calcareous soil upon its application at a dose of 60 mg kg^{-1} or higher (Fig 2A). Mostly, Mn was found as impurities in P-fertilizers

(*Cheraghi et al.* 2012; Chen and Graedel 2015); yet, in our case, the used P additive (KH_2PO_4) was of high purity. Thus, the reasonable explanation for such increases is that soil cations (e.g. Ca) that compete with Mn on sorption sites became precipitated in soil in the form of insoluble phosphate salts (McGowen *et al*. 2001; Arai and Sparks 2007). Thus, Mn became more easily sorbed on soil particles and this in turn raised the available fraction of Mn in soil.

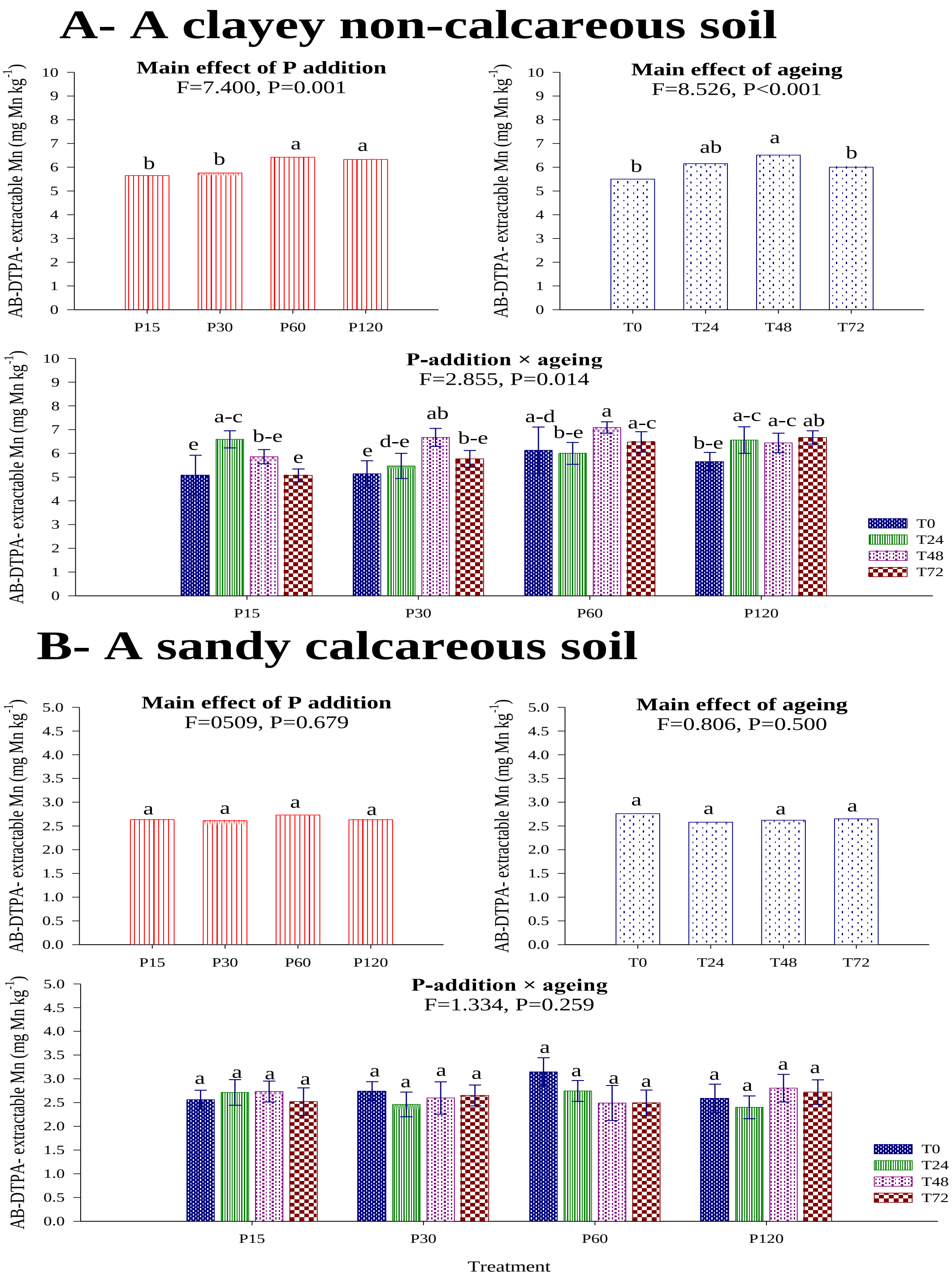

Fig. 2. AB-DTPA extractable Mn in the investigated soils as affected by increasing the rate of applied P and its aging of application. See footnote Fig 1. Similar letters indicate no significant variations among treatments.

Concerning the effect of aging of the applied P on AB-DTPA extractable-Mn in such a soil, the highest increases were detected after 24-48 h of P application, with no significant variations between these two periods (24 and 48h). The combination of the two factors of study was also of significant effect on AB-DTPA extractable-Mn. In this concern, the highest value of AB-DTPA extractable-Mn achieved due to application of P_{15} was at T_{24}. For both P_{30} and P_{60}, the highest increases in AB-DTPA extractable-Mn were recorded at T_{48}, while the highest increases attained due to P_{120} were recorded at T_{72}.

In the calcareous soil, neither of the elevated doses of applied P nor aging of its application could significantly affect the extractable AB- DTPA-Mn (Fig 2B). Likewise, the interactions between these two factors recorded no significant impacts on AB-DTPA-Mn. This might indicate the high capability of the calcareous soil to immobilize Mn (Moharami and Jalali 2013), regardless of the applied P dose i.e. recalcitrant in an oxidation form (MnO_2) (Gao *et al.* 2020).

3.3. Effect of dose of the applied P and its aging of application on AB-DTPA extractable Zn content

Fig 3A reveals that the application dose of P to the non-calcareous clayey soil did not significantly affect the extractable amounts of AB-DTPA-Zn in soil when P was applied at either P_{15} or P_{30}; yet significant increases were detectable when P was applied at the higher doses i.e. P_{60} then P_{120} (Fig 3A). Mostly, the exchange sites of soil exhibit relatively low affinity for Zn sorption (Usman *et al*. 2008; Lu *et al*. 2009; Vidal *et al*. 2009), especially in the presence of Ca (Acosta *et al*. 2011).

At high P-doses, soluble cations that compete with Zn on sorption sites might undergo precipitation (McGowen *et al*. 2001; Arai and Sparks 2007); thus, exchangeable Zn- ions increased considerably (Rupa *et al.* 2003). Also, K ions in KH_2PO_4 may substitute sorbed Zn and set it free in soil solution.

On the other hand, this cation (Zn) might undergo precipitation in soil due to the application of phosphate fertilizers at high doses (Lambert *et al*. 2007) and this explains the slight reductions that occurred in AB-DTPA-Zn when P was applied at the dose P_{120} versus its application at the dose P_{60}. The effects of the aging of P as well as the interaction between P-inputs and the aging of their application on AB-DTPA-Zn were not significant.

In the calcareous soil, neither P-inputs nor their aging of application affected significantly concentrations of AB-DTPA-extractable Zn (Fig 3B). This is because of the rapid fixation of Zn in calcareous soil (Abbas 2013; Duffner *et al*. 2012; Hui *et al*. 2019) which occurred within the first few hours after application (Abbas 2013). On the other hand, interactions between the two factors (P-doses×aging of their applications) were of significant effects on the extractability of Zn by the AB-DTPA. In this concern, the highest concentrations of AB DTPA Zn at low P application doses (P_{15} and P_{30}) were found at T_{24}. At higher P doses (P_{60} and P_{120}), AB DTPA- Zn contents were initially high then decreased followed by significant increases as if Zn availability took the form of consecutive peaks of increases and reductions till equilibration.

3.4. Effect of dose of the applied P and its aging of application on AB-DTPA extractable Se content

Application of P to the non-calcareous soil did not significantly affect the extractable amounts of Se, yet this available content varied significantly owing to the aging of the applied P (Fig 4A). In this concern, concentrations of Se increased progressively up to 48 h of application; thereafter, insignificant reductions occurred. Mostly, concentrations of Se in soil were too little, to the extent, that any detectable amounts of the applied P might be quite enough to compete with exchangeable Se.

There was no P_0 level to compare with, yet the effect of soil aging might endorse the above assumption. It is then thought that P competed with the poorly bound Se on the binding sites of the soil matrix (Schilling *et al*. 2011; Keskinen *et al*. 2013) to set it free (Peng et al. 2020); thus, its extractable concentrations increased significantly within the first 48h of application; while decreased thereafter to attain new levels towards equilibrium in soil. Interactions between Se and P on Se available content in soil were insignificant.

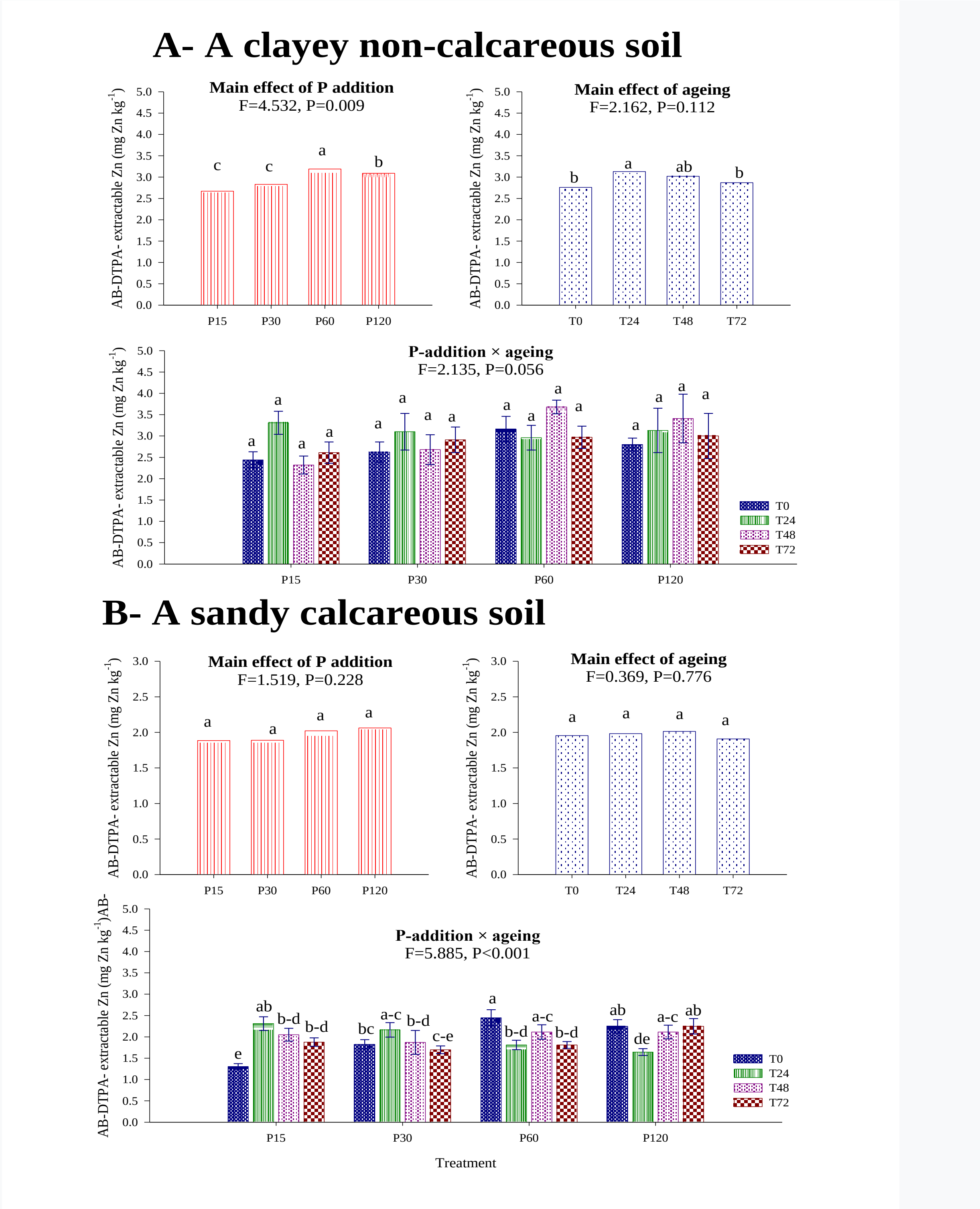

Fig. 3. AB-DTPA extractable Zn in the investigated soils as affected by increasing the rate of applied P and aging of its application. See footnote Fig 1. Similar letters indicate no significant variations among treatments.

Concerning Se in the calcareous soil, its AB-DTPA-extractable content was significantly affected by only the aging of the applied P while the effects of both the applied P dose as well as the interactions between P-dose and aging of its application were insignificant (Fig 4B). In this concern, the highest increases in AB-DTPA extractable-Se occurred within the first 24 h after P application then decreased significantly and again rose as if concentrations of AB-DTPA-Se underwent cycles of increases and decreases. The acceptable scenario for these findings is that the soil underwent successive oxidation-reduction reactions that affected P availability (Ahmed *et al*. 2013) and because Se was of low content in soil; thus its available content also changed. Generally, soil pH and redox potential are the two main factors affecting Se availability in soil (Dinh *et al.* 2017). In this concern, Se availability increased considerably in well-aerated alkaline soils, mainly as selenate (Tan *et al.* 2002; Li *et al*. 2017).

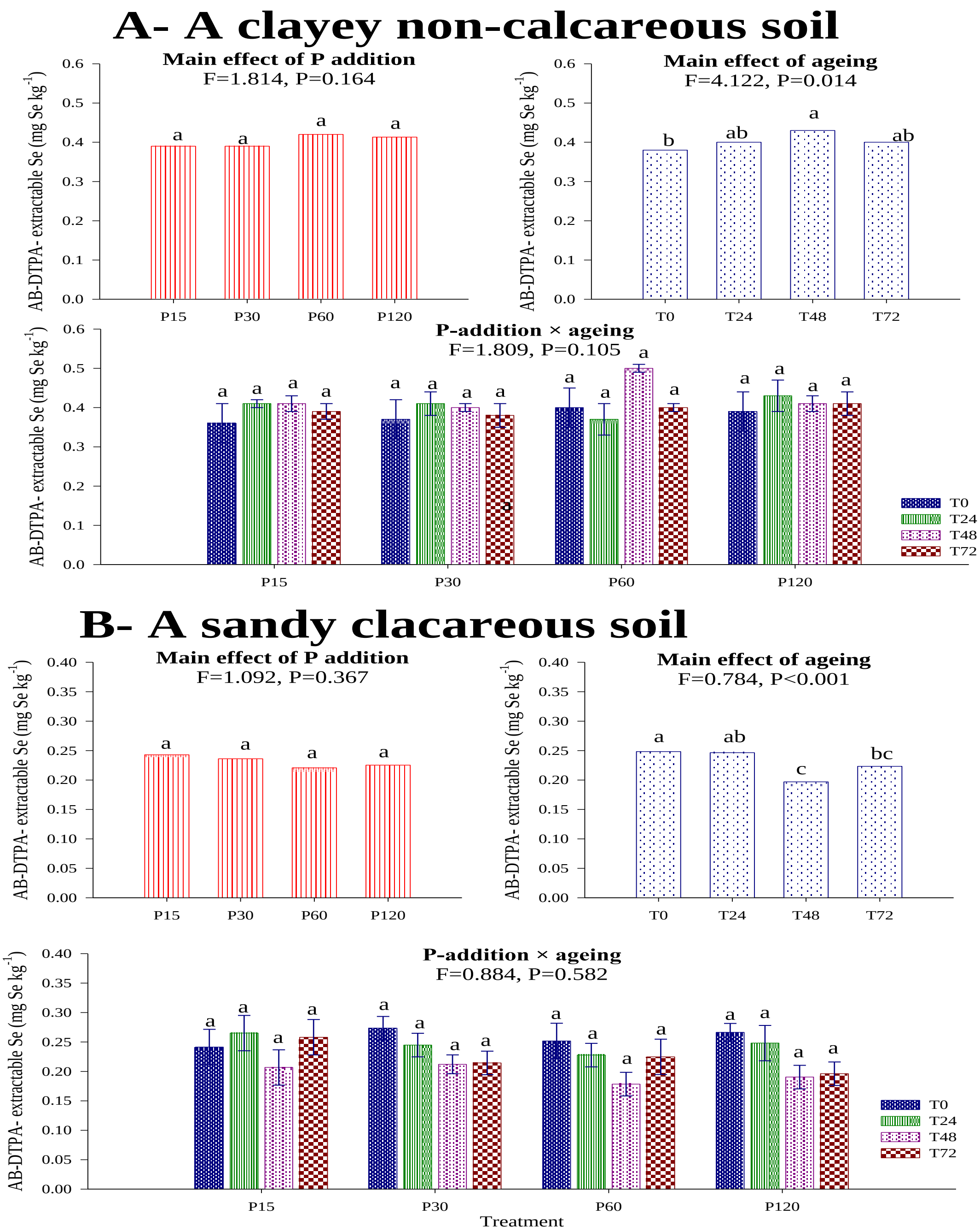

Fig 4. AB-DTPA-Se in the investigated soils as affected by increasing the rate of applied P and aging of its application. See footnote Fig 1. Similar letters indicate no significant variations among treatments.

3.5. Effect of dose of the applied P and its ageing of application on AB-DTPA extractable P content in soil

Results presented in Fig 5A reveal that the application of P significantly raised its AB-DTPA extractable amounts in the clayey non calcareous soil. Likewise, ageing of its application significantly affected this available content following the sequence: T_{72}>T_{48}>T_{24}>T_0. This probably indicates that P fixation needs longer time periods (>72h) to be noticeable in such a soil. In particular, the highest increases in AB- DTPA-P were detectable at T_{72} in soils treated with either: P_{30}, P_{60} or P_{120}, while T_{48} was quite enough to attain the highest increase in AB - DTPA-P in soil amended with P_{15}.

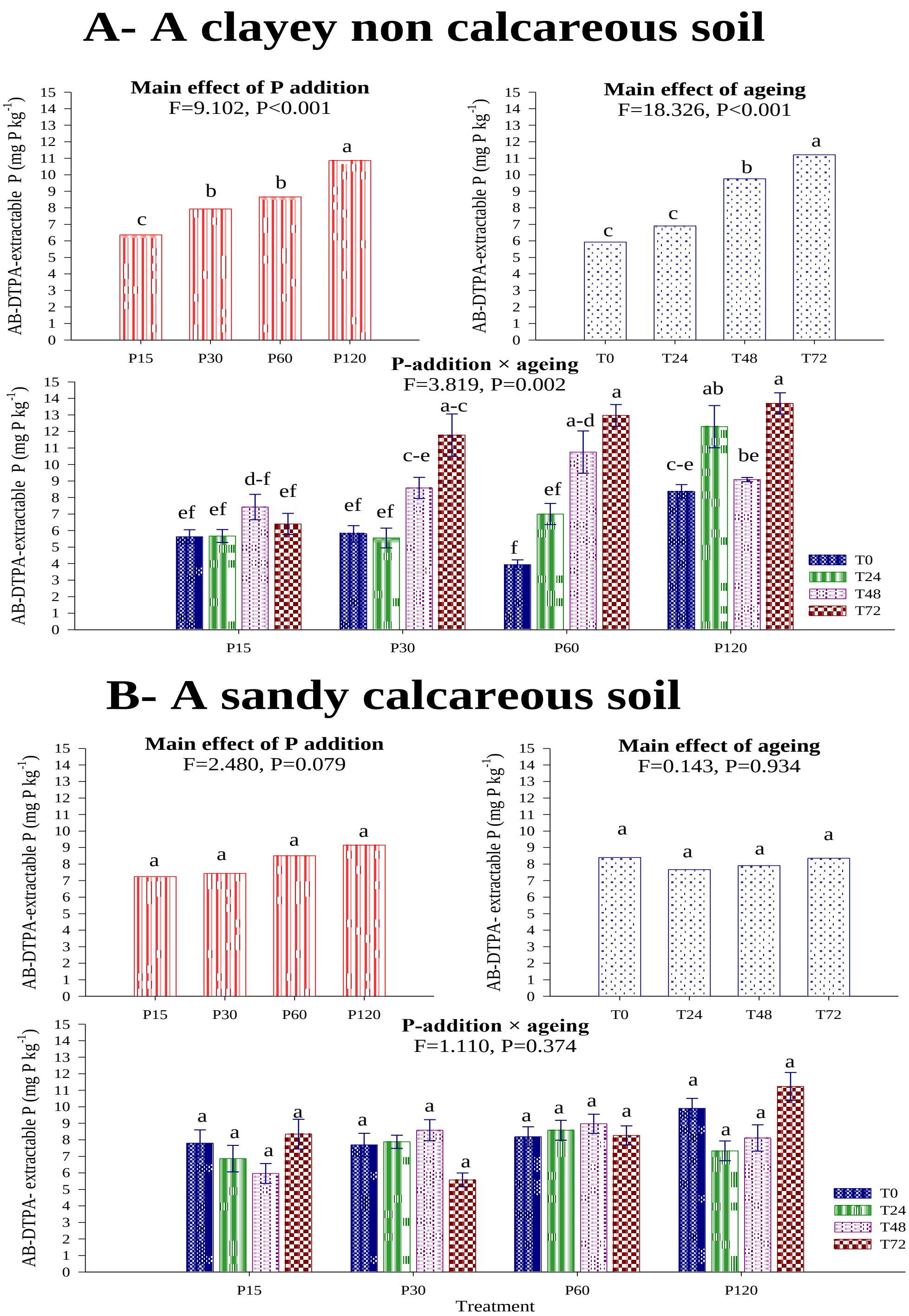

Fig 5. AB-DTPA extractable P in the investigated soils as affected by increasing the rate of applied Fe and the incubation period. See footnote Fig 1. Similar letters indicate no significant variations among treatments.

In the calcareous soil, AB-DTPA- extractable P was neither affected by the applied P dose nor aging of its application (Fig 5B). Moreover, the interactions between these two factors were of no significant impacts on AB-DTPA-P. This probably indicates rapid P sorption on $CaCO_3$ surfaces (Naeem *et al*. 2013). Also, the alkaline conditions of this soil significantly decreased P availability in soil (Jalali and Jalali 2016). In addition, Ca ion, which is found at high concentrations in the calcareous soil can immobilize P in the form of insoluble calcium phosphate (Wang *et al*. 2010; Wahid *et al*. 2020) within short time periods (Jalali and Ranjbar 2010). Initially, rapid adsorption of P occurred, then it changes into low-soluble salts (Jalali and Ranjbar 2010).

3. Conclusions

AB DTPA-P increased continuously in soil up to 72 h of P-application (the end of the investigation period). Such increases became noticeable with increasing the rate of applied-P. This almost indicates that P needs relatively longer time periods (>72h) to be fixed in soil. On the other hand, concentrations of AB- DTPA extractable- Fe and Se were not significantly affected by the dose of applied P when being added to the non-calcareous soil. In such a soil, interactionsbetween P and each of these two nutrients are thought to be slower than expected. On the other hand, AB-DTPA-extractable Mn and Zn increased significantly when P was applied at a rate of 60 mg P kg^{-1} or higher. These results validate partially the first hypothesis for only Mn and Zn

The highest increases in AB-DTPA-Mn owing to P applications were detected after 48 h while in case of AB DTPA- Zn, their contents flocculated in soil in the form of consecutive increases and reductions till the end of the investigation. These results could not therefore support the second hypothesis which indicated that micronutrients precipitate in the form of insoluble phosphate salts within the first 24 h of application.

Regarding the effect of ageing of the applied P on AB-DTPA extractable Se, this content increased significantly in soil up to 48h of application; thereafter, significant reductions occurred. These findings varifies partially the third assumption. In the calcareous soil, none of the hypothesis could be tested because this soil has very high affinity to immobilize P within a short time periods (<24 h); thus P interactions could not be monitored. Overall, the results of this study could effectively improve our knowledge about the possible interactions among added P and soil micronutrients/Se within the first 72 h of application. Yet, more time sequence analyses are needed to verify these reactions within this short time period.

4. Conflicts of interest

There are no conflicts to declare.

5. Formatting of funding sources

This research received no specific grant from any funding agency.

6. Acknowledgment

The authors would like to thank the staff of Soils and Water Department, Fac. Of Agriculture, Benha University, Egypt.

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