



Effect of organic and inorganic phosphorus fertilizers on phosphorus availability and its leaching over incubation time

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Abstract

The use of organic and inorganic phosphorus (P) fertilizers in agricultural soils is very common, and few studies have been conducted to study the effect of different P sources on relative P extractability (RPE) and leaching using different P extractants and degree of P saturation (DPS), over a long period of time. Thus, this study was conducted to investigate the effect of incubation time and different P sources on RPE, DPS, and to predict the concentration of P leached from soil using different P extractants. In order to achieve these goals, nine sewage sludges (SSs), two biochars, animal manure (AM), poultry manure (PM), wheat residue (WR), diammonium phosphate (DAP), and triple superphosphate (TSP) were added to the soil as much as 100 mg P kg⁻¹ in a 163 days incubation experiment. On average across all amendments and incubation periods, Mehlich-3 extractable P (M3EP) gave the highest mean RPE (42.9%, SE = 7.1%), with water-extractable P (WEP) the lowest (4.6%, SE = 0.93%), and Olsen-extractable P (OEP) (38.3%, SE = 6.3%) in between. Among SSs and based on average across of all incubation periods, soils treated with Shiraz and Takestan SSs were the least soluble source of P, while the highest soluble source of P were soils treated with Kermanshah and Tehran SSs. The results indicated that soil samples taken 16 days following the addition of amendments should reflect agronomic and environmental purposes aiming to assess available and the potential P loss from agricultural soils. The split line model perfectly fitted to the relation between OEP and M3EP ($r = 0.93$). The DPSs were calculated and the P leaching rate was estimated. Based on OEP, the soils treated with TSP and DAP were at high risk, the medium risk was for soils treated with Kermanshah, Saveh, Tehran, Rasht, Sanandaj, and Isfahan SSs, and PM. Control soil, and soils treated with WR were at no risk, and the soils treated with Arak, Shiraz, and Takestan SSs, ABC, WBC, and AM were classified as low risk.

Keywords Phosphorus fertilizers · Phosphorus extractors · Degree of phosphorus saturation · Phosphorus loss potential

Introduction

Phosphorus (P) content starts from a small amount to over 1 g kg⁻¹ in the surface and subsurface of the soil (Prasad and Power 1997). About 30–60% of the total P is organic P in most soils, and its range differs depending on warm and cold regions of the world (Gregory and Nortcliff 2013). Inorganic

P generally bound with calcium (Ca) in neutral to alkaline soils, whereas in acidic soils, iron (Fe) and aluminum (Al) are compound with P. The demand for P use is primarily covered by application of inorganic fertilizers, animal manure, biowaste, and sewage sludge (SS) in agricultural soils, and accumulation of these inputs in soil may build up to 1000 kg P ha⁻¹ year⁻¹ (Van der Perk 2013).

Sewage sludge containing 8% of P is recognized as an essential resource of the P nutrient used in agricultural land (Klingmair et al. 2015). Another source of P application in soils is biochar, a solid, rich in carbon material produced by the pyrolysis of biomass at temperature ≤ 700 °C under no or low oxygen condition (Lehmann and Joseph 2015). Sewage sludge and biochar can improve soil physical and chemical characteristics and increase soil agricultural productivity and enhance P availability in soils (Huang et al. 2012; Schneider and Haderlein 2016; Sohi et al. 2010). Animal manures are also used in agricultural land which are derived from livestock

The original article was revised: The correct presentation of the equations in Table 6 footnote is presented in this paper.

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feces and urine. Manure contains different forms and content of P and about 60 to 90% of total P in manure is inorganic P (Sharpley and Moyer 2000).

Several studies have evaluated the effect of organic residuals on P release (Jalali 2009; Jalali and Ranjbar 2011; Jalali et al. 2018). Also, there are studies that reported the effect of the application of SS and biochar on availability of P (Frišták and Soja 2015; Lemming et al. 2017a, b); however, there was no particular study which used these various types of organic and inorganic fertilizers to assess efficiency and P leaching risk.

The objectives of this study were (1) to examine whether different SSs, biochars, manures, and plant residue when applied at the same rate are different in their effects on P efficiency using three different P extractants and degree of P saturation (DPS) over a period of 163 days; (2) to find relationship among different P extractants, (3) to predict the concentration of P leached using linear and split line, and (4) to prepare a risk category for soil treatments.

Materials and methods

Soil sampling and analyses

The soil sample was collected from topsoil (0–30 cm) of the Azandarian region located in the Hamadan province (34° 29' 35" N, 48° 42' 52" E, and altitude of 1775 m). The soil sample was air-dried and sieved through a 2-mm sieve. Soil pH (1:5 soil to distilled water ratio and, 30 min shaking), electrical conductivity (EC) (1:5 soil to distilled water ratio and, 30 min shaking), cation exchange capacity (CEC) (sodium acetate and ammonium acetate method), organic matter (OM) (Walkley-Black method), CaCO₃ (back titration method), and soil texture (hydrometer method) contents were determined according to the methods of Rowell (1994). Total P, sodium (Na), potassium (K), Ca, magnesium (Mg), and Fe were measured by weighing 0.2 g soil in a porcelain crucible and placed in a furnace at 550 °C for 1 h, then the soil transferred to Erlenmeyer flask and 25 ml 1 N HCl was added to the flask and boiling for 15 min. Once cooled, the solution transferred to a 50 ml volumetric flask and made up to the mark by distilled water (Andersen 1976). Phosphorus was determined by the colorimetric method (Murphy and Riley 1962), using a UV-visible spectrophotometer. Sodium and K were measured by flame photometer, Jenway-PFP 7. Calcium and Mg were measured by complexometric titration method (Rowell, 1994). Iron was measured using atomic absorption spectrophotometry, Varian, spectra 220. Water-extractable P (WEP), Olsen-extractable P (OEP), and Mehlich-3 extractable P (M3EP) were extracted by method described in incubation experiments and chemical analysis section. The results of soil analyzes are presented in Table 1.

Table 1 The physicochemical properties of Azandarian soil

Soil parameters	Azandarian soil
pH	7.8
EC ^a dS m ⁻¹	0.098
CEC ^b meq 100 g ⁻¹	4.7
OM ^c %	0.46
CaCO ₃ %	1.57
WEP ^d mg kg ⁻¹	11.08
OEP ^e mg kg ⁻¹	29.85
M3EP ^f mg kg ⁻¹	32.82
Total P mg kg ⁻¹	657.2
Total Na mg kg ⁻¹	788.4
Total K mg kg ⁻¹	2527.9
Total Ca mg kg ⁻¹	1667.8
Total Mg mg kg ⁻¹	12,551.8
Total Fe mg kg ⁻¹	15,568.9
Clay %	20.0
Sand %	70.8

^aElectrical conductivity, ^bcation exchange capacity, ^corganic matter, ^dwater-extractable P, ^eOlsen-extractable P, ^fMehlich-3 extractable P

Fertilizers

Nine SSs that came from various cities of Iran were used (Arak, Isfahan, Kermanshah, Rasht, Sanandaj, Saveh, Shiraz, Takestan, and Tehran). These SSs results from municipal wastewater treatment plants, but the SSs of Arak and Saveh results from a treatment plant receiving both municipal and industrial wastewater (Feizi et al. 2019). The procedures to acquire SSs in wastewaters is settlement tank, waste stabilization pond, constructed wetland, percolating filter, and anaerobic digestion. Two biochars were used in this study as the fertilizer; the almond shell biochar (ABC) and walnut shell biochar (WBC) were produced with pyrolysis at 500 and 400 °C, respectively, for 2 h. More information and characteristics of SSs and two biochars are presented in Feizi et al. (2019) and Hemati Matin et al. (2020a), respectively. Animal manure (AM), poultry manure (PM), and wheat residue (WR) were collected from local farms.

The diammonium phosphate (DAP), triple superphosphate (TSP) were used as the synthetic P fertilizers in the studied area. The pH and EC of fertilizers were analyzed in distilled water, and with the ratio of 1:10, total P were analyzed as described in the previous section (0.2 g soil, 1 h, 550 °C, boiling with 25 ml 1 N HCl for 15 min) using the method of Andersen (1976); WEP, OEP, and M3EP were extracted by the methods described in incubation experiments and chemical analysis section, and the results are given in Table 2.

Table 2 Fertilizers characteristics

Fertilizers	pH	EC dS m ⁻¹	WEP mg kg ⁻¹	OEP mg kg ⁻¹	M3EP mg kg ⁻¹	Total P mg kg ⁻¹	Total Na mg kg ⁻¹	Total K mg kg ⁻¹	Total Ca mg kg ⁻¹	Total Mg mg kg ⁻¹	Total Fe mg kg ⁻¹	C/P %	C/N %
SS of Arak	7.4 [†]	4.6 [†]	4.40	138.90	233.94	8652.0	1382.3	724.7	35,070.8	14,165.2	16,200.6	24.1	10.3 [†]
SS of Isfahan	7.3 [†]	3.4 [†]	122.33	950.46	1741.52	10,128.0	1880.0	1469.1	29,266.3	11,887.7	12,098.0	25.8	9.4 [†]
SS of Kermanshah	6.1 [†]	4.5 [†]	497.12	2559.27	2693.88	12,204.0	1203.9	1848.9	59,038.4	24,361.2	13,092.0	17.4	8.7 [†]
SS of Rasht	7.4 [†]	1.9 [†]	215.21	792.75	1491.60	15,564.0	1476.2	1967.1	6826.1	15,304.0	15,736.3	10.9	7.4 [†]
SS of Sanandaj	6.7 [†]	2.3 [†]	165.03	699.15	1256.64	10,299.0	1424.6	2030.0	15,185.6	39,021.4	14,785.8	24.9	7.8 [†]
SS of Saveh	8.9 [†]	2.9 [†]	99.02	703.80	1427.80	7479.0	6450.6	2686.4	9853.3	14,910.1	13,451.3	41.4	17.7 [†]
SS of Shiraz	7.3 [†]	1.9 [†]	17.85	166.80	707.78	9972.0	1333.0	571.3	115,027.8	18,962.8	10,874.0	21.3	10.5 [†]
SS of Takestan	6.8 [†]	5.1 [†]	6.56	255.30	356.09	4586.0	1844.8	2266.4	23,767.3	19,101.6	15,763.2	49.7	10.2 [†]
SS of Tehran	7.5 [†]	1.5 [†]	556.32	1413.30	2500.68	14,109.0	1563.1	1992.2	10,214.4	12,443.2	11,405.0	19.7	8.3 [†]
ABC	6.5 ^{††}	0.4 ^{††}	40.03	63.09	76.72	300.2	809.5	3476.1	2618.4	1825.5	92.8	66.2	161.0
WBC	5.6 ^{††}	0.5 ^{††}	79.72	99.49	127.68	578.2	713.3	5264.2	8464.7	1924.0	135.3	99.4	118.0
AM	10.0	2.3	243.56	526.65	465.60	1328.7	1171.1	5616.3	3369.3	6559.9	13,152.5	34.8	28.7
PM	7.5	2.5	1549.50	3193.25	7341.60	21,348.0	3023.2	18,966.6	44,124.6	13,041.6	1927.0	18.2	11.5
WR	6.3	0.4	0.1	30.34	60.65	590.6	882.3	2623.5	10,325.5	6948.8	3509.0	924.4	124.4
DAP	8.3	67.5	-	-	-	-	-	-	-	-	-	-	-
TSP	2.8	18.6	89,875.00	105,110.00	130,480.00	184,483.3	3098.4	344.9	6131.8	10,357.5	2790.0	-	-

[†] Measured by Feizi et al. (2019)B8, ^{††} Measured by Hemati Matin et al. (2020a)

ABC, Almond shell biochar; WBC, walnut shell biochar; AM, animal manure; MP, poultry manure; WR, wheat residue; DAP, diammonium phosphate; TSP, triple superphosphate; WEP, water-extractable P; OEP, Olsen-extractable P; M3EP, Mehlich-3 extractable P

Incubation experiments and chemical analysis

Plastic pots were prepared and included treatments with the addition of SS of Arak, SS of Isfahan, SS of Kermanshah, SS of Rasht, SS of Sanandaj, SS of Saveh, SS of Shiraz, SS of Takestan, SS of Tehran, ABC, WBC, AM, PM, and WR at a rate of 100 mg P kg⁻¹ (approximately at an equal rate of 260 or 300 kg P ha⁻¹ for the top 20 cm soil depth having 1.3 or 1.5 g cm⁻³ bulk density) to each pot which already contains 1 kg Azandarian soil. The application rate of P used in the current study was comparable with other studies. Lemming et al. (2017b) added the SS and TSP at the rate of 80 mg P kg⁻¹. Siddique and Robinson (2003) amended the soils with AM and SS at the rate of 100 mg P kg⁻¹. In the study performed by Huang et al. (2012), they amended the soil with SS at the rates of 150 and 300 mg P kg⁻¹. Azandarian soil without any amendments considered control soil. Each treatment was replicated three times. After mixing soil with amendments, the soil moisture was brought to field capacity by distilled water and then placed them at 25 °C and incubated for 163 days. The pots were inspected every week, and soil moisture was maintained at field capacity. A certain amount of soil (about 40 g) were taken from days 2, 9, 16, 23, 37, 58, 86, 121, and 163 (at same soil moisture level) to represent the short and long incubation time. The soil samples from each incubation time let 1 week to air dry, crushed, and passed through 2 mm sieve.

Soil pH was measured and samples were then extracted with three P extractants for available P. Water-extractable P was determined in 1:5 soil to solution ratio, and half an hour shaking (Rowell 1994), also by this method, we measured the pH of solutions. Olsen-extractable P was measured by shaking 1 g soil with 20 ml sodium bicarbonate 0.5 M (pH 8.5) for half an hour (Olsen and Sommers 1982). Mehlich-3 extractable P was obtained by shaking 1 g soil with 8 ml Mehlich-3 solution (0.2 M CH₃COOH + 0.25 M NH₄NO₃ + 0.015 M NH₄F + 0.13 M HNO₃ + 0.001 M EDTA) for 5 minutes (Mehlich 1984). Phosphorus in the various extracts was determined colorimetrically using the ascorbic acid method (Murphy and Riley 1962). Relative P extractability (RPE) for WEP, OEP, and M3EP was determined for all treated soils over all incubation periods to indicate the ability of each treatment to increase soil P (Leytem and Bjorneberg 2009). The RPE for WEP, OEP, M3EP in amended soils was calculated as (Leytem and Bjorneberg 2009):

$$\text{Relative P extractability} = \frac{\text{Soil } P_{\text{amended}} - \text{Soil } P_{\text{control}}}{P_{\text{applied}}} \times 100$$

where soil P_{amended} is the WEP (or OEP, M3EP) at each sampling time for soil treated with SSs, ABC, WBC, AM, PM, WR, DAP, or TSP; soil P_{control} is the WEP (or OEP, M3EP) for the soil in control at each sampling time; and P_{applied} is the amount of P applied with each treatment.

Phosphorus sorption index

Phosphorus sorption index (PSI) is a single point isotherm which is first introduced by Bache and Williams (1971) and determined by weighing 1 g soil, equilibrating with a 20 ml solution of 75 mg P l⁻¹, containing 10 mM CaCl₂ as background electrolyte, for 18 h at room temperature. The PSI was calculated according to Wang et al. (2016) and Jalali and Jalali (2017). Degree of P saturation demonstrating the extent of P accumulating in soils related to the sorption capacity of P. There are many ways to calculate DPS, and in this study, we used the equations presented by Jalali and Jalali (2017).

$$\text{DPSWEP} = \frac{P_{\text{WEP}}}{P_{\text{WEP}} + \text{PSI}} \times 100 \quad (1)$$

$$\text{DPSOEP} = \frac{P_{\text{OEP}}}{P_{\text{OEP}} + \text{PSI}} \times 100 \quad (2)$$

$$\text{DPSM3EP} = \frac{P_{\text{M3EP}}}{P_{\text{M3EP}} + \text{PSI}} \times 100 \quad (3)$$

where the units of parameters are mg kg⁻¹.

Data analysis

The relationship between different P extraction methods was described by the split line model using SigmaPlot (2011) program. All experiments were carried out in triplicates.

Results and discussion

Soil and organic and inorganic properties

The pH, EC, CEC, and OM of the soil are 7.8, 0.098 dS m⁻¹, 4.7 cmol_c kg⁻¹, and 0.46%, respectively. The total P content was 657.2 mg kg⁻¹. Other soil properties and P extracted with different extractions are shown in Table 1. Main SSs chemical properties are reported in Table 2, indicating high variation in OM, pH, EC, and total P. The highest content of total P (except TSP and DAP) was attributed to the PM, SS of Rasht, and SS of Tehran, while ABC, WBC had the lowest total P. Because of the very low P content, the biochars as well as the WR tend to be highly unlike option of P amendment. Approximately 800–900 t ha⁻¹ of ABC may be applied to supply the 100 mg P kg⁻¹, which is approximately 3 times the amount applied by Hemati Matin et al. (2020a) in similar conditions. Biochars derived from almond and walnut shells

have already been used by Hemati Matin et al. (2020a) at a rate of 2.5–10% to test sorption-desorption, fractionation, and P release in two soils. Hemati Matin et al. (2020b) indicated that increasing the application rates of ABC and WBC reduces leaching and stabilizes heavy metals in contaminated soils. Though these rates of application are not feasible on the field scale and are not practical in the natural environment, they are widely used in related studies to explain the benefits or disadvantages of higher application rates (Hemati Matin et al. 2020a). The aim was to pick a broad range of amendments to investigate the impact on P availability of different SSs, biochars, manures, and plant residues with a wide range of P content when added at the same rate.

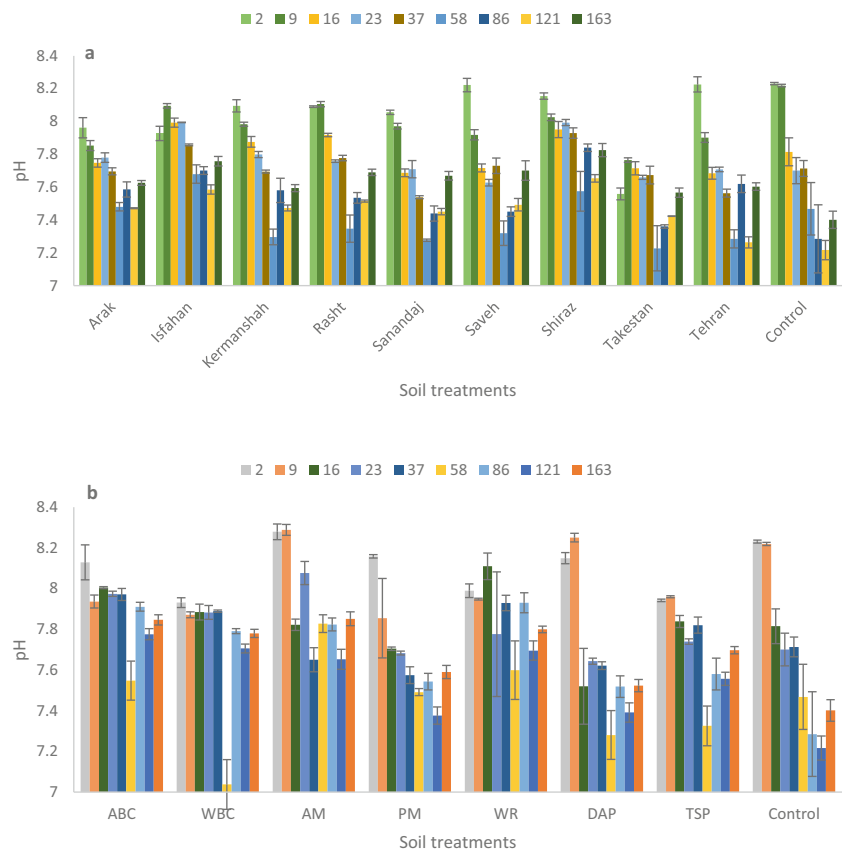
The M3EP of organic and inorganic P sources gave higher values and a larger range (2.7–70.7% of total P) than OEP (1.6–57.0% of total P), and WEP (0.05–48.7% of total P). The WEP contents of the SSs samples ranged from 0.05 to 4% of total P, while the content of TSP extracted by WEP was 48.7%, indicating low solubility of the SSs.

In general, different P fertilizers vary widely in their solubility, which induces the response of crops to P fertilizers to vary under the same soil and crop conditions (Chien et al. 1990). The low water-soluble P in TSP used in the current study can be clarified according to the following factors. The percentage of water-soluble P in P fertilizers depends on

some factors including the nature of the phosphate rock ore used to manufacture P fertilizers and the method of determining the solubility of P fertilizers (e.g., the time of extraction and the ratio of solid to the solution used to extract P). When producing P fertilizers, water-insoluble complexed P fractions such as Fe-P, Al-P, Ca-P, and Mg-P can form. The variation in water solubility of P in TSP is the result of rock components, primarily Fe and Al, which react with P during the production process to produce water-insoluble P compounds (Johnston and Richards 2003). Chien et al. (2011) suggested that single superphosphate (SSP), TSP, monoammonium phosphate (MAP), and DAP categorized as water-soluble P fertilizers are 85–90% water-soluble and the remaining P is citrate-soluble. They also indicated that some sources of low quality SSP could contain as little as 50–60% water-soluble P.

On the other hand, for TSP, water-soluble P is usually determined using 1:250 fertilizer to water ratio and 1 h shaking time (AOAC 1999). Johnston and Richards (2003) analyzed two US and two UK TSP products according to the AOAC procedure and found that water-soluble P was in the range of 84 to 95%. In the current study, the water solubility of TSP was measured using 1:5 ratio and 30 min shaking to be comparable with other amendments. Therefore, it appears that increasing the ratio and shaking time will also tend to increase the water-soluble P derived from TSP.

Fig. 1 Changes in pH of soil treatments. **a** SS treatments. **b** other treatments. Error bars show standard errors of the means ($n = 3$). Almond shell biochar (ABC), walnut shell biochar (WBC), animal manure (AM), poultry manure (PM), wheat residue (WR), diammonium phosphate (DAP), triple superphosphate (TSP)



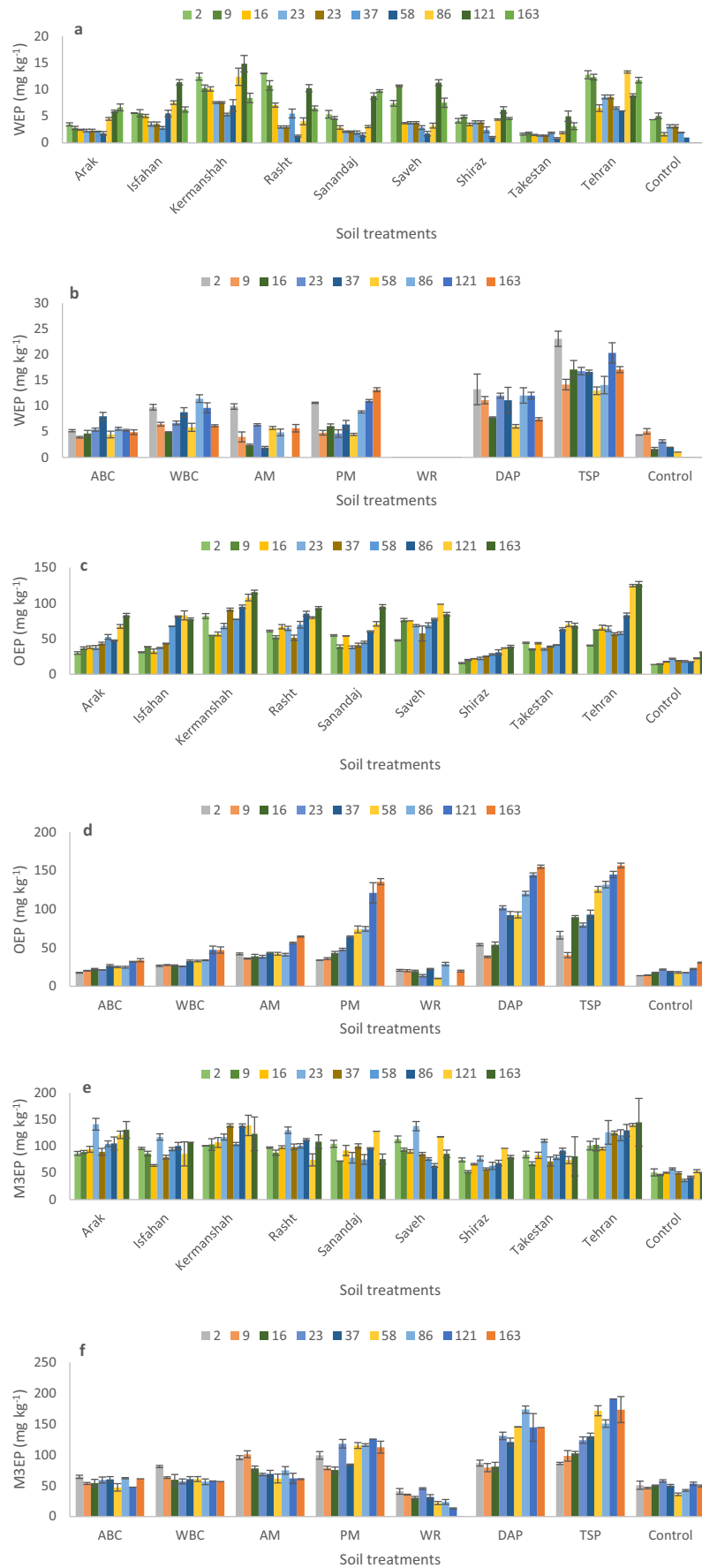


Fig. 2 Changes in P content of soil amended with different treatments. **a**, **b** WEP. **c**, **d** OEP. **e**, **f** M3EP. Error bars show standard errors of the means ($n = 3$). Almond shell biochar (ABC), walnut shell biochar (WBC), animal manure (AM), poultry manure (PM), wheat residue (WR), diammonium phosphate (DAP), triple superphosphate (TSP), water-extractable P (WEP), Olsen-extractable P (OEP), Mehlich-3 extractable P (M3EP)

pH and phosphorus availability in soil treatments

Changes in pH over 163 days of incubation of soil fertilized with different P sources are shown in Fig. 1. Generally, the Figure shows that the soil pH decreased over the 163 days of incubation for all treatments. Control treatment with dramatic pH decline at day 163 (from 8.23 to 7.40) indicates the lowest value among other treatments. Lemming et al. (2017b) studied the effect of 197-day incubation on soil fertilized with SS and TSP, at a P level of 80 mg kg⁻¹ soil, and reported the decrease in soil pH for all treatments. The pH decline in amended soils could be due to the nitrification and creation of protons during this procedure. The SSs utilized in this investigation has a carbon to nitrogen (C/N) proportion of about 10 (except SS of Saveh) (Table 2), indicating that nitrification is probably going to happen in the amended soils, because of the buildup of this N-rich residues.

Table 3 The statistically analyzed of 17 treatments × 3 extractants as a factorial completely randomized design (each value, mg kg⁻¹ represent the average of nine incubation time)

Treatments	WEP	OEP	M3EP
SS of Arak	3.5 ^{qr}	48.4 ^{lm}	107.1 ^{bcd}
SS of Isfahan	5.9 ^{qr}	54.6 ^{kl}	92.4 ^{def}
SS of Kermanshah	9.8 ^{opqr}	83.1 ^{efgh}	119.2 ^{bc}
SS of Rasht	6.8 ^{pqr}	69.3 ^{hijk}	100.1 ^d
SS of Sanandaj	4.4 ^{qr}	55.3 ^{jkl}	91.4 ^{defg}
SS of Saveh	5.8 ^{qr}	72.7 ^{hij}	96.0 ^{de}
SS of Shiraz	3.9 ^{qr}	26.6 ^{no}	70.6 ^{hijk}
SS of Takestan	2.1 ^{qr}	48.9 ^{lm}	82.5 ^{efgh}
SS of Tehran	9.6 ^{opqr}	75.8 ^{fghi}	120.7 ^{ab}
ABC	5.2 ^{qr}	24.8 ^{nop}	56.6 ^{ijkl}
WBC	7.7 ^{pqr}	33.3 ^{mn}	61.4 ^{ijkl}
AM	4.5 ^{qr}	44.6 ^{lm}	74.5 ^{fghi}
PM	7.7 ^{pqr}	69.9 ^{hijk}	102.7 ^{cd}
WR	0.0 ^f	17.2 ^{nopqr}	26.9 ^{no}
DAP	10.3 ^{opqr}	94.7 ^{de}	122.9 ^{ab}
TSP	16.9 ^{nopqr}	103.0 ^{cd}	136.4 ^a
Control	1.9 ^{qr}	19.4 ^{nopq}	48.5 ^{lm}

ABC, Almond shell biochar; WBC, walnut shell biochar; AM, animal manure; PM, poultry manure; WR, wheat residue; DAP, diammonium phosphate; TSP, triple superphosphate; WEP, water-extractable P; OEP, Olsen-extractable P; M3EP, Mehlich-3 extractable P

Figure 2 shows the changes in P content extracted with WEP, OEP, and M3EP in different treatments. The P content derived by WEP was so small in WR treatment that it could not be detected. The contents of P in the control treatment were undetectable after 58 days of incubation. Among the treatments, the P content derived from WEP was lowest over the incubation period in SS of Takestan, SS of Arak, and SS of Shiraz (on average, 2.1, 3.5, and 3.9 mg kg⁻¹, respectively). Not surprisingly, the TSP and DAP treatments showed on average the highest P release (16.9 and 10.3 mg kg⁻¹, respectively) (Fig. 2a, b). The statistically analyzed treatments are presented in Table 3. These results indicate that the extractability of P from amendments is dependent on the characteristics of the P source rather than the total amounts of P in amendments (Table 2).

Although the WEP of treated soils decreased dramatically over time, the content of P in OEP, increased from day 2 to day 163 at all treatments (Fig. 2c, d). This trend may be due to the mineralization of organic P in amended soils, or a slow dissolution or desorption of organic P compounds (Lemming et al. 2017a). Similar results were also reported by Case et al. (2016) conducted on sludge-amended soil and biosolids-amended soil, respectively. Clearly, the decrease in pH over time (Fig. 1) may enhance the solubility of Ca-P compounds lead to an increase in P availability in soil (Gahoonia et al. 1992). The OEP in the soils treated with the TSP and DAP was significantly higher than other treatments and in soils treated with WR, ABC, SS of

Table 4 Correlations between amendment properties and available P from treated soils (on average over all incubation times) using different extractants

Available P extracted from treated soils			
Amendment properties	WEP	OEP	M3EP
pH	-0.561 [*]	-0.262 ^{ns}	-0.204 ^{ns}
EC	0.701 ^{**}	0.658 ^{**}	0.614 [*]
WEP	0.749 ^{**}	0.559 [*]	0.468 ^{ns}
OEP	0.758 ^{**}	0.572 [*]	0.481 ^{ns}
M3EP	0.761 ^{**}	0.578 [*]	0.486 ^{ns}
Total P	0.784 ^{**}	0.628 [*]	0.547 [*]
Total Na	0.242 ^{ns}	0.508 ^{ns}	0.367 ^{ns}
Total K	0.022 ^{ns}	0.032 ^{ns}	-0.046 ^{ns}
Total Ca	-0.118 ^{ns}	-0.130 ^{ns}	0.045 ^{ns}
Total Mg	-0.067 ^{ns}	0.292 ^{ns}	0.356 ^{ns}
Total Fe	-0.275 ^{ns}	0.204 ^{ns}	0.336 ^{ns}
C/P	-0.585 [*]	-0.527 [*]	-0.705 ^{**}
C/N	-0.243 ^{ns}	-0.702 ^{**}	-0.784 ^{**}

** Correlations significant at $p = 0.01$, * correlations significant at $p = 0.05$, ^{ns} not significant

WEP, water-extractable P; OEP, Olsen-extractable P; M3EP, Mehlich-3 extractable P

Table 5 Pearson correlation between different time of incubation with three P extractant methods

Extractant methods	Incubation time	2	9	16	23	37	58	86	121
WEP	9	0.862**							
	16	0.920**	0.807**						
	23	0.887**	0.756*	0.866**					
	37	0.844**	0.694*	0.871**	0.904**				
	58	0.843**	0.627 ^{ns}	0.860**	0.913**	0.827**			
	86	0.797**	0.700*	0.801**	0.837**	0.794**	0.866**		
	121	0.759*	0.770**	0.877**	0.666*	0.726*	0.684*	0.751*	
	163	0.795**	0.657**	0.784**	0.680*	0.677*	0.721*	0.740*	0.807**
OEP	9	0.666*							
	16	0.818**	0.806**						
	23	0.771**	0.678*	0.829**					
	37	0.827**	0.571 ^{ns}	0.771**	0.917**				
	58	0.747*	0.565 ^{ns}	0.832**	0.876**	0.921**			
	86	0.802**	0.618 ^{ns}	0.843**	0.919**	0.925**	0.954**		
	121	0.702*	0.652*	0.811**	0.904**	0.912**	0.922**	0.928**	
	163	0.725*	0.571 ^{ns}	0.794**	0.886**	0.916**	0.911**	0.917**	0.975**
M3EP	9	0.836**							
	16	0.805**	0.850**						
	23	0.693*	0.740*	0.775**					
	37	0.674*	0.800**	0.874**	0.731*				
	58	0.560 ^{ns}	0.706*	0.737*	0.797**	0.863**			
	86	0.568 ^{ns}	0.691*	0.750*	0.750*	0.912**	0.942**		
	121	0.629 ^{ns}	0.682*	0.797**	0.724*	0.877**	0.889**	0.837**	
	163	0.590 ^{ns}	0.745*	0.789**	0.838**	0.880**	0.962**	0.921**	0.896**

** Correlations significant at $p = 0.01$, * correlations significant at $p = 0.05$, ^{ns} not significant

WEP, water-extractable P; OEP, Olsen-extractable P; M3EP, Mehlich-3 extractable P

Shiraz, WBC, and control soil were significantly lower than the other treatments (Table 3). Figure 2e, f shows the P content of M3EP over incubation time. In most treated soils, the M3EP increased with incubation time. For example, the M3EP from soils treated with SS of Arak increased from 87 mg kg⁻¹ after 2 days to 131 mg kg⁻¹ after 163 days. In addition, M3EP in soils treated with TSP and DAP was significantly higher, and soil treated with WR was significantly lower than the other treatments (Table 3).

Considering the mean extractable P under different treatments and incubation times by various methods, the lowest amount of P was extracted by WEP, while the highest amount of P was extracted by M3EP (Table 3), which may be related to the acidity of this extracting solution. Phosphorus bound with Ca can be dissolved following the addition of the acid solution. The same results were also reported by Ige et al. (2006).

Phosphorus availability and amendments properties

The bioavailability of P depends on organic C, Ca, Mg, Fe, Al, and P forms, and presence of organic anions in soil and

manure (Wang et al. 2016; Kahiluoto et al. 2015). The SSs used in our experiment had a relatively high content of Fe (Table 2). Hence, it could be speculated that the addition of these SSs to soil may lead to the formation of less soluble Ca-P and Fe-P compounds (Smith et al. 2002). The correlations were performed between properties of amendments and available P from treated soils (on average across all incubation times) using different extractants (Table 4). There was not any significant correlation between total Na, K, Mg, Ca, and Fe content in amendments and available P, but there were positive correlations between EC, total P, WEP, OEP, and M3EP in amendments with available P extracted from treated soils using different extractants. But the correlations between WEP, OEP, and M3EP in amendments with available P extracted by M3EP from treated soils were not significant (Table 4).

Carbon to N ratio of amendments may affect P availability in soil (Sharpley and Smith 1989). In this study, there was a significant negative correlation between C/N in amendments and OEP (-0.702) and M3EP (-0.784) in treated soils. There was also a significant negative correlation between C/P in amendments and WEP (-0.585), OEP (-0.527), and M3EP (-0.705),

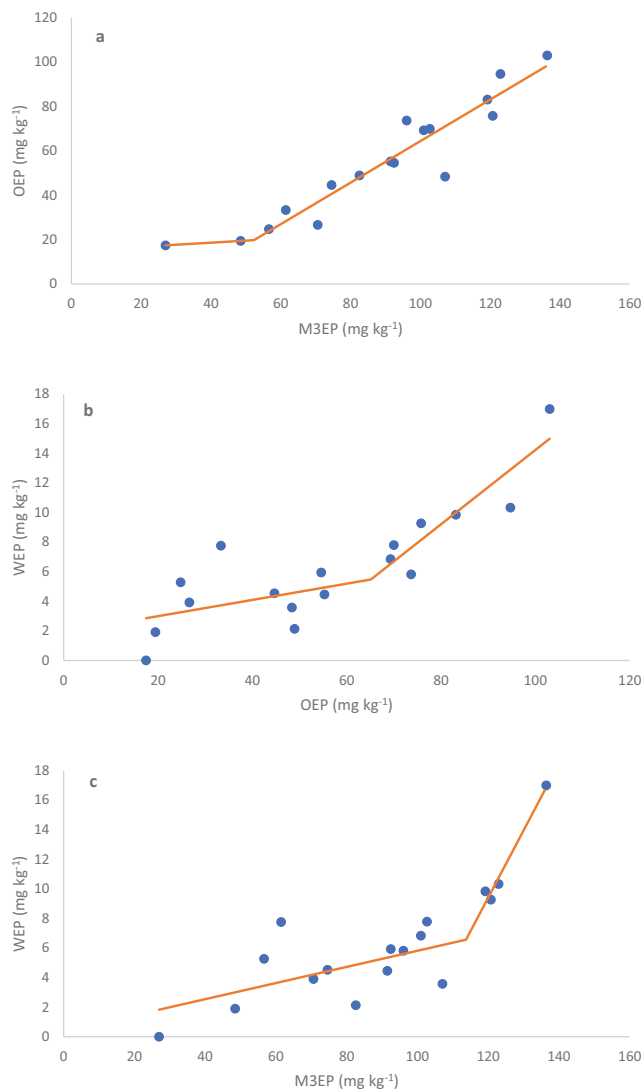


Fig. 3 Relationship between **a** OEP and M3EP, **b** WEP and OEP, and **c** WEP and M3EP by split line model. Each point is the average of nine incubation time. Water-extractable P (WEP), Olsen-extractable P (OEP), Mehlich-3 extractable P (M3EP)

in treated soils suggesting that WR, SS of Saveh, SS of Takestan, and AM were expected to mineralize at a lower rate than the other amendments during incubation. Thus it seems that the solubility of P in these amendments when applied to the soil depends on their available and total P, C/N, and C/P.

Relative P extractability of added P

The RPE of added P varied with different amendments, incubation periods, and extractants. The average (across all amendments and incubation periods) M3EP gave the highest mean RPE values (42.9%, SE = 7.1%), with WEP the lowest (4.6%, SE = 0.93%), and OEP (38.3%, SE = 6.3%) in between. Unlike PM, the RPE values in AM were 28.2% and 33.7%, respectively, after 2 and 163 days of incubation. The explanation why

these two treatments differ in behavior could be due to the high C/N and C/P values in AM compared with PM (Table 2).

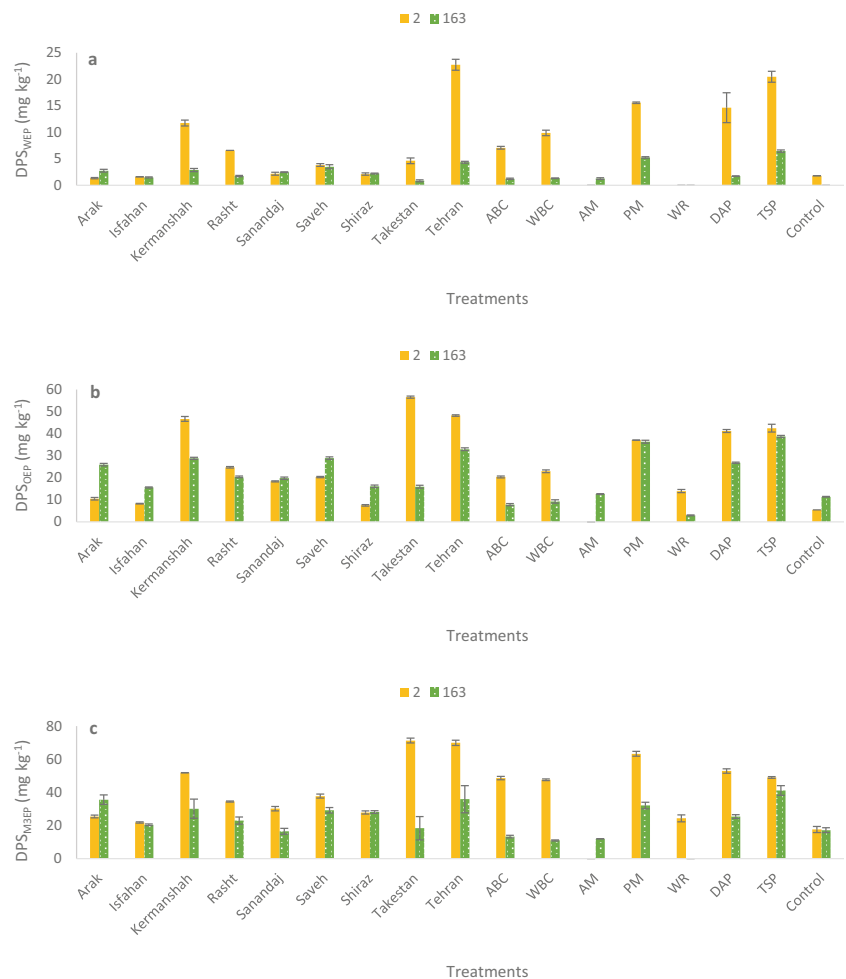
On average 3.4%, 5.4%, and 8.1% of WEP, OEP, and M3EP were recovered for ABC over all incubation times, while 7.9%, 63.7%, and 70.7% of WEP, OEP, and M3EP were extracted for Kermanshah’s SS. Throughout the 163 days, the SSs (except Shiraz’s SS) retained a higher RPE. Following addition of SSs, organic compounds will be added to the soil, preventing P adsorption in the SSs-treated soils (Kahiluoto et al. 2015). The immediate increase in OEP with SS of Kermanshah, SS of Tehran, SS of Saveh, SS of Rasht, and SS of Isfahan may be due to the presence of soluble P forms in these amendments. Net P mineralization can be expected with the application of all SSs, and PM, because the P content in all of them (Table 2) was higher than the critical level of 0.25%, suggested for net P mineralization (Nziguheba et al. 1998). The low P content in the WR (0.06%), ABC (0.03%), WBC (0.06%), and AM (0.13%) would result in net P immobilization. Iyamuremye et al. (1996) also found increases in the resin P, bicarbonate inorganic P, and NaOH extractions with manure and alfalfa residues, both with greater than 0.24% P, whereas wheat straw with 0.09% P generally did not increase these fractions.

Parham et al. (2002) studied the effects of long-term application of animal manure and chemical fertilizer on soil P. They indicated that the M3EP was significantly lower in soil treated with manure than soil treated with chemical fertilizer which was close to our study. Similar to present study, Garg and Bahl (2008) observed that incubating soil fertilized with organic manure for 60-day increased OEP, and the highest amount of extractable P observed in PM treated soil than crop residue.

Relationships of available P between various incubation times

The average (across all amendments and incubation periods) *r* (0.811) obtained from linear relationships between OEP in different incubation times was higher but not significantly than WEP (0.788) and M3EP (0.781) (Table 5). These results proved that when these three methods were used to determine P, changes of available P over time were stable. It is important to know the best time to take samples following the addition of P amendments which should reflect plant-available P and P losses from the soil. Considering the values of *r* for OEP over 16 days with a mean of 0.848, M3EP with a mean of 0.797, and WEP with a mean of 0.813 compared with the other incubation periods can be used as an indicator of P availability. Thus, it can be concluded that all methods are suitable to be used in extracting P from the soil after 16 days of application of P amendments. Therefore, 16 days of extraction can provide a better estimate of the solubility of P in various sources of P. The implications of the results are important from agronomic and environmental aspects for soil testing

Fig. 4 DPS_{WEP} (a), DPS_{OEP} (b), and DPS_{M3EP} (c) of the soil incubation experiment for all treatments on day 2 and day 163. Error bars show standard errors of the means. Almond shell biochar (ABC), walnut shell biochar (WBC), animal manure (AM), poultry manure (PM), wheat residue (WR), diammonium phosphate (DAP), triple superphosphate (TSP), degree of phosphorus saturation (DPS), water-extractable P (WEP), Olsen-extractable P (OEP), Mehlich-3 extractable P (M3EP)



after the addition of these amendments to the soil. Thus, soil samples for extracting available P can be taken 16 days after the addition of amendments which can be considered to represent plant-available P and P losses from the soil. Wang et al. (2016) indicated that the 2-week incubation using M3EP is suitable for determining P availability following the addition of various manures across soil types.

Relationship between P extractability in treated soils

Figure 3 shows the relationships between different extractants by fitting the split line model to the data. Each point reflects an average of nine incubation time. Although some P losses due to the surface runoff or leaching occur in water, our results indicate that OEP due to the high correlation of this extractant with WEP has higher accuracy ($P < 0.001$) for the estimation of P in water, after adding amendments to the soil. Sharpley (1997) indicated that M3EP was significantly correlated with dissolved P, which will be lost from soils by runoff. Results indicated that we could calculate the content of P from one extraction by another one using these relationships, and also showed that the content of WEP, OEP, and M3EP began to increase sharply

after a change point. Other researches also found such relations (Jalali and Jalali 2016, 2017; Wang et al. 2015; Zang et al. 2013). It should be noted here that substantial amounts of P lost during runoff or leaching events are particulate or colloid-bound and not water-solubilized and recorded by several researchers (Weaver and Summers 2014; Hua and Zho 2020). Phosphorus that is attached to particles and colloids may be lost and leads to P leaching from soils.

Jalali and Jalali (2017) suggested the relationship between WEP and OEP with leached P by distilled water can be expressed by the split line model, and found the change point of 27.4 and 61.5 mg kg^{-1} , for WEP and OEP, respectively. This change point means that if we extracted soil P by any of these extractants and the value obtained was higher than these change points, P leaching from soil will continue with a higher slope and increase the risk of P leaching. On average of all incubation time, none of the treatments showed the values higher than change point of 27.4 mg kg^{-1} for WEP. For OEP of SS of Tehran (75.7 mg kg^{-1}), SS of Saveh (73.6 mg kg^{-1}), PM (69.9 mg kg^{-1}), and SS of Rasht (69.2 mg kg^{-1}) were higher than 61.5 mg kg^{-1} , the change point for OEP, other treatments were lower than the change point.

Table 6 The concentration of P in leachate predicted for different treatments and WEP, OEP, M3EP, and DPS_{M3EP} by linear and split line model

Treatments	Linear			Split line								
	Predicted concentration of P leached by distilled water (mg l ⁻¹)			Predicted concentration of P leached by 10 mM CaCl ₂ (mg l ⁻¹)			Predicted concentration of P leached by distilled water (mg l ⁻¹)			Predicted concentration of P leached by 10 mM CaCl ₂ (mg l ⁻¹)		
	WEP ¹	OEP ²	M3EP ³	WEP ⁴	OEP ⁵	M3EP ⁶	WEP ⁷	OEP ⁸	DPS _{M3EP} ⁹	WEP ¹⁰	OEP ¹¹	DPS _{M3EP} ¹²
SS of Arak	0.248	1.930	0.454	0.087	0.963	0.963	0.514	1.662	0.388	0.422	0.862	0.369
SS of Isfahan	0.654	2.276	0.391	0.103	1.105	1.105	0.810	2.143	0.393	0.533	1.044	0.366
SS of Kermanshah	1.328	3.865	0.506	0.130	1.755	1.755	1.301	4.138	0.412	0.716	1.801	0.359
SS of Rasht	0.811	3.093	0.428	0.109	1.439	1.439	0.924	2.765	0.662	0.575	1.291	0.481
SS of Sanandaj	0.400	2.314	0.387	0.093	1.120	1.120	0.625	1.979	0.343	0.464	1.000	0.387
SS of Saveh	0.634	3.283	0.406	0.102	1.517	1.517	0.795	3.104	0.376	0.527	1.424	0.373
SS of Shiraz	0.305	0.719	0.297	0.090	0.468	0.468	0.556	0.854	0.316	0.438	0.532	0.398
SS of Takestan	-0.001	1.959	0.348	0.078	0.975	0.975	0.332	1.597	0.639	0.355	0.827	0.487
SS of Tehran	1.296	3.455	0.513	0.128	1.588	1.588	1.278	3.526	1.124	0.708	1.624	0.319
ABC	0.540	0.616	0.237	0.099	0.425	0.425	0.727	0.806	0.646	0.502	0.510	0.520
WBC	0.968	1.091	0.257	0.116	0.620	0.620	1.039	1.028	0.321	0.618	0.609	0.396
AM	0.413	1.723	0.314	0.094	0.878	0.878	0.634	1.351	0.375	0.467	0.741	0.374
PM	0.974	3.132	0.435	0.116	1.455	1.455	1.043	3.404	0.640	0.620	1.573	0.361
WR	-0.369	0.193	0.109	0.063	0.252	0.252	0.064	0.598	0.239	0.255	0.447	0.429
DAP	1.412	4.510	0.522	0.133	2.019	2.019	1.362	5.581	0.399	0.739	2.490	0.364
TSP	2.549	4.973	0.580	0.178	2.209	2.209	2.192	6.306	0.489	1.049	2.776	0.531
Control	-0.040	0.317	0.202	0.076	0.303	0.303	0.304	0.667	0.275	0.344	0.448	0.415

¹ $y = 0.1725x - 0.3687$, ² $y = 0.0557x - 7661$, ³ $y = 0.0043x - 0.0067$, ⁴ $y = 0.00676x - 0632$, ⁵ $y = 0.0228x - 0.1404$, ⁶ $y = 0.0018x - 1535$,
⁷ $\frac{(0.253) \times (27.4-x) + (3.51) \times (x-1.5)}{27.4-1.5}$, ⁸ $\frac{(0.274) \times (61.2-x) + (1.75) \times (x-4.298)}{61.2-4.298}$, ⁹ $\frac{(0.183) \times (48.4-x) + (0.476) \times (x-3.357)}{48.4-3.357}$, ¹⁰ $\frac{(0.325) \times (27.6-x) + (1.55) \times (x-1.5)}{27.6-1.5}$,
¹¹ $\frac{(0.272) \times (64.5-x) + (0.971) \times (x-4.298)}{64.5-4.298}$, ¹² $\frac{(0.452) \times (50.2-x) + (0.328) \times (x-3.357)}{50.2-3.357}$

x, average content of extractable P or DPS across all incubation time; y, predicted concentration of P leached. *ABS*, Almond shell biochar; *WBC*, walnut shell biochar; *AM*, animal manure; *PM*, poultry manure; *WR*, wheat residue; *DAP*, diammonium phosphate; *TSP*, triple superphosphate; *DPS*, degree of phosphorus saturation; *WEP*, water-extractable P; *OEP*, Olsen-extractable P; *M3EP*, Mehlich-3 extractable P

Degree of phosphorus saturation in different treatments

On average across all amendments and incubation times, the DPS was in the order of M3EP > OEP > WEP. Figure 4 shows the DPS which is calculated for the incubation period of 2 and 163 days in all treatments. Obviously, the DPS_{WEP} was lower than DPS_{OEP} and DPS_{M3EP}, and DPS_{M3EP} on average had the highest DPS percentage. Zang et al. (2013) used two rates of pig manure and two rates of superphosphate. They reported that application of these fertilizers had a great effect on DPS, and suggested that this high DPS is an important factor which is affecting accumulation and mobilization of P. Ajiboye (2003) reported that the DPS, in bio-fertilizer and MAP (8–16%) was lower than that of pig and cattle (11 to 55%), and heavily dependent on the rate of added P in the soil.

Jalali and Jalali (2017) related the concentration of P in leachate with DPS_{M3EP} by split line model. They found the change point of 48.4 and 50.2% for P leached with distilled

water and 10 mM CaCl₂ solutions, respectively. However, there is still concern about SS of Kermanshah, SS of Rasht, PM, and DAP treatments, where the mean values were near the change points and continuous application of these SSs and synthetic P fertilizer in the future will increase the risk of P leaching in the studied area.

Prediction leaching of phosphorus

Jalali and Jalali (2017) found the relationship between P concentration in leachates and different soil extraction P methods and DPS. They found that there was a linear correlation between the soil leached with either distilled water or 10 mM CaCl₂ solution and soil extractable P. They also found the relationship among the leached P concentration with former solutions and extractable P and DPSs by split line model. We used these relations to predict the concentration of P in leachate in different treated soils. The concentration of P in leachate predicted for different

treatments and different extraction P methods and DPS by linear and split line model are presented in Table 6. In split line model M3EP, DPS_{WEP} , and DPS_{OEP} used in the study of Jalali and Jalali (2017) were not significantly related to the P concentration in leachate, so in Table 6 we show only the predicted values for WEP, OEP, and DPS_{M3EP} . Predicted values showed that the concentration of P in leachate was lower in 10 mM $CaCl_2$ than that of the distilled water, except for M3EP in the linear model which was on average 0.74 mg l^{-1} higher in 10 mM $CaCl_2$. The highest P concentration in leachate in the linear model was observed in soils treated with TSP, DAP, SS of Kermanshah, and SS of Tehran for WEP, OEP, when the soil was leached with distilled water. The concentration of P is also predicted by the split line model (Table 6). Like the linear model, WEP and OEP concentrations were lower in 0.01 mM $CaCl_2$ solution than that of the distilled water.

In order to check the P losses potential in different treatments, we used classification which suggested by Jalali and Jalali (2017). They used change points which resulted from the relationship between WEP, OEP, and DPS_{M3EP} with P leached by distilled water, and then they classified them into four risk categories. Table 7 shows the range of

each extractable or DPS and potential P losses from soil treatments. Considering WEP, none of the treatments (average of nine incubation time) categorized as a high risk (Table 7). Most treated soils were at low and medium risk.. Based on DPS_{M3EP} , none of the treatments were at high risk of P leaching, soils treated with SS of Tehran and TSP at medium risk, soils treated with SS of Shiraz, WBC, WR, and control soil at no risk, and other treatments were at low risk category. Even though the application rate of P used in the current study was comparable with other studies, the current application rate could be high from a farmer point of view. Under field conditions, the proposed categories for each treatment may not be matched. Therefore, if the model predicts certain treatments for a high concentration of P in the leachates, this does not mean that equivalent concentrations of P will occur in water percolating into groundwater.

Conclusion

Upon the addition of different P sources in soil, extractable P increased, which was varied with P sources. The smallest

Table 7 Soil treatments risk category of P leaching potential based on Jalali and Jalali (2017)

Soil extractables or DPS	P range (mg kg^{-1})	Treatments
WEP		
No risk	≤ 5	SS of Arak, SS of Sanandaj, SS of Shiraz, SS Takestan, AM, WR, control
Low	5–15	SS of Isfahan, SS of Kermanshah, SS of Rasht, SS of Saveh, SS of Tehran, ABC, WBC, PM, DAP
Medium	15–31	TSP
High	> 31	-
OEP		
No risk	≤ 20	WR, control
Low	20–50	SS of Arak, SS of Shiraz, SS of Takestan, ABC, WBC, AM
Medium	50–85	SS of Isfahan, SS of Kermanshah, SS of Rasht, SS of Sanandaj, SS of Saveh, SS of Tehran, PM
High	> 85	DAP, TSP
DPS_{M3EP}		
No risk	< 25	SS of Shiraz, WBC, WR, control
Low	25–50	SS of Arak, SS of Isfahan, SS of Kermanshah, SS of Rasht, SS of Sanandaj, SS of Saveh, SS of Takestan, ABC, AM, PM, DAP
Medium	50–75	SS of Tehran, TSP
High	> 78	-

ABS, Almond shell biochar; WBC, walnut shell biochar; AM, animal manure; PM, poultry manure (PM); WR, wheat residue; DAP, diammonium phosphate; TSP, triple superphosphate; DPS, degree of phosphorus saturation; WEP, water-extractable P; OEP, Olsen-extractable P; M3EP, Mehlich-3 extractable P

extractable P was with soils treated with WR and SS of Shiraz, while the greatest was with TSP, DAP, Tehran and Kermanshah SSs, and Arak and Rasht SSs, and PM were intermediate. Since all three extractant methods had a high correlation coefficient, they provide a strong index of P availability in this calcareous-amended soil. The results suggested that the availability of P from various amendments is different, which should be taken into account in their management following addition to the soil. Results of calculating DPSs showed that the highest DPS values were observed at soils treated with TSP, SS of Tehran, and PM and the lowest were at soils treated with WR, and SS of Shiraz, and control soil. The linear and split line models predicted the highest concentration of P in leachate for soils treated with TSP, DAP, SS of Kermanshah, and SS of Tehran. Among SSs, the soils treated with Shiraz, Sanandaj, and Takestan showed the lowest P release, and among other fertilizers, PM showed higher release and then WBC and ABC, respectively. The results can be used to predict leaching of P due to the addition of organic and inorganic P sources, which is essential to reduce P losses to the surface and groundwater. In general, higher loads of biochar and wheat amendments should be applied due to the low P content which is not recommended. We acknowledge the fact that if the model predicts a high concentration of P in the leachates and meets standards of water quality, this does not mean that comparable P concentrations would occur in water percolating into groundwaters. The current application rate might be high from a farmer's perspective. Had the procedure for P been conducted at the usual rate, there would have been less leaching of P and so the concentration of P in the leachate would probably have been different.

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