

Immobilization of Phosphorus in Biosolids-Amended Soils by Water Treatment Residual Nanoparticles

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ABSTRACT

Phosphorous mobility in soil environments is largely controlled by P sorption and desorption reactions. This study was designed to evaluate the effects of water treatment residual nanoparticles (nWTRs) at different rates on P mobility in biosolids-amended soils. Sorption and desorption batch experiments were performed on two different soils amended with biosolids at a rate of 3% and 3 rates of nWTRs (0.10, 0.20, and 0.30%). The sorption data showed that nWTRs increased the amount of P sorbed by the biosolids-amended soils with the effect increases as the nWTR application rate increases suggesting that more sorption sites were added on the soil surface as a result of nWTRs addition. The modeling of sorption equilibrium data showed that Langmuir model fit the data much better than Freundlich, Elovich, Kiselev Hill-de Boer, Fowler-Guggenheim, and Temkin models, with relatively higher R² values and smaller standard error of estimates (SE). Whereas, the power function and first order kinetics models provided much better fit for the P adsorption kinetics as evidenced by higher coefficient of determination (R²) and lower SE values. Application of nWTRs with different rates to the clay soil drastically reduced the percentage of desorbed P to 6, 4, and 1% from clay soil and to 12, 7 and 4% from sandy soil at 0.10, 0.20 and 0.30% application rates, respectively. The lack of similarity between adsorption and desorption due to the hysteresis is likely a result of binding to Al/Fe hydroxides. Fourier transmission infrared (FTIR) results indicate the crucial role of surface hydroxyl groups in P retention onto nWTRs.

Keywords: phosphorus- mobility- sorption-nanoparticles.

INTRODUCTION

Phosphorus behavior in soils differs from soil to soil according to the variation and abundance in solid phase and their reactivity. The behavior of inorganic P in soil can be best explained by adsorption-desorption reactions (Barrow, 1980). Soils with excess P concentrations are a water quality concern because even a small amount of P (1 to 2 kg/ha/yr) transported into lakes can accelerate eutrophication. Eutrophication increases the cost of purifying surface water and restricts water usage by recreational and industrial sectors (Novak and Watts, 2004).

The availability of P added to crops, among other factors, depends on the rate at which it is converted to

less soluble forms in soils. The addition of P fertilizer to soils resulted in a series of chemical reactions with soil compounds that decrease its solubility such as P adsorption on clay minerals, Fe-Al oxides and CaCO₃ and precipitation of Ca phosphates.

The nanotechnology-based soil amendments enable better control over the conditions for timing of plant nutrients release.

Water treatment residuals (WTRs) are by-product of water purification systems in which undesirable attributes of the raw water such as turbidity, color and dissolved solids are removed by a variety of physical and chemical processes. In recent years, more attention has focused on beneficial reuse of WTRs in land application (Elkhatib and Mahdy, 2008; Mahdy et al., 2009, 2012, 2013). Recent research has focused on using WTRs as cost-effective materials to reduce soluble phosphorus (P) in soils, runoff, and land-applied organic wastes (manures and biosolids).

Recently, Elkhatib et al., (2015) have created nanoparticles from water treatment residuals (nWTRs). Because of their amorphous nature, nanoparticles have a large and highly reactive surface area (> 105 m² g⁻¹) (Makris et al., 2004) where tremendous quantities of P can be adsorbed (Ippolito et al., 2003; Novak and Watts, 2004), along with many other oxyanions such as AS⁵⁻, As³⁻, ClO⁴⁻, and Pb (Makris et al., 2006a,b).

We hypothesize that the application of nWTRs in biosolid-amended agricultural soils would substantially increase P adsorption on amorphous nanoparticles and further decrease runoff to ground water. The objectives of this study were to: make a systematic investigation of adsorption-desorption reactions of P, at various initial concentrations, in different soils amended with different rates of biosolids and nWTRs, and study effects of nWTRs on the kinetics of P adsorption in biosolids and the biosolid-amended agricultural soil at 25⁰ C.

MATERIALS AND METHODS

Soils, biosolids and water treatment residuals (WTRs)

Two soil types were selected for this study: clay soil (*Typic torrifluvent*, from Kafr El-Dawar, Elbohera Governorate, Egypt), and sandy soil (*Typic*

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torripsamment, from El-Bostan ,Elbohera Governorate, Egypt). Soils were collected from a depth of 0–30 cm at each sampling location. Air-dried soil samples were ground and subsequently sieved (<2 mm). The experimental biosolids were obtained from the General Organization Sanitary (GOS) in Alexandria City (Station 9), Egypt, air-dried ground and sieved (<2 mm) prior to use (Makris and Harris 2005). The original WTRs were obtained from a local drinking water treatment plant (in Alexandria), Egypt. The plant uses aluminum sulfate for flocculation. The WTR samples were collected, transported to the laboratory and air-dried. Sub-samples were ground and passed through two different sieves having 2 mm and 51 μ m of pore diameters.

Subsamples <51 μ m in diameter of 6g were milled using Fritsch planetary mono mill Pulverisette 6 classic line equipped with 80 ml stainless steel grinding bowl and 150 g of 1 mm steel grinding balls (Elkhatib et al., 2015).

The general physiochemical properties of the soils, biosolids, and WTR were determined according to standard methods (Tan, 1996) and compiled in Table (1).

Stabilization experiment

To ensure chemical equilibrium between soil/amendments, incubation experiment was conducted. Recommended rate of biosolids (3%), recommended rate of traditional WTR (2%), and 3 rates of nWTR (0.10, 0.20, 0.30%) were applied, either separately or combined, to *Typic torrifluent*, and *Typic torripsamment* soils in Egypt. The various soil mixes were moistened, thoroughly mixed, and equilibrated at 80 % of their water holding capacity (WHC) for 30 d at room temperature (25°C). This experiment was conducted in a completely randomized design in a laboratory environment, and each treatment was triplicate. After the incubation period, soil samples were air dried, crushed to pass a 2-mm sieve, and stored until chemical analysis.

Table 1. Some physical and chemical characteristics of studied soils, WTRs and biosolids

Characteristics	Units	Clay	Sandy	WTRs	Biosolids
pH		8.13 \pm 0.05	7.69 \pm 0.05	7.45 \pm 0.06	6.69 \pm 0.03
EC	dSm ⁻¹	2.66 \pm 0.11	3.84 \pm 0.12	1.67 \pm 0.04	11.25 \pm 0.12
CaCO ₃	g kg ⁻¹	57.90 \pm 0.60	2.40 \pm 0.30	-	-
Sand	g kg ⁻¹	596.4 \pm 4.20	868.2 \pm 5.10	-	-
Silt	g kg ⁻¹	141.3 \pm 1.50	25.10 \pm 0.30	-	-
Clay	g kg ⁻¹	262.30 \pm 3.70	106.70 \pm 2.20	-	-
Texture		S.C.L	L.S	-	-
O.M	g kg ⁻¹	8.50 \pm 0.15	1.00 \pm 0.04	57.00 \pm 2.00	450.00 \pm 1.67
KCl-Al	mg kg ⁻¹	1.03 \pm 0.04	0.13 \pm 0.02	28.18 \pm 1.03	4.22 \pm 0.13
Olsen-P	mg kg ⁻¹	24.75 \pm 0.25	2.89 \pm 0.14	24.00 \pm 2.00	48.60 \pm 1.62
CEC	Cmol(+)kg ⁻¹	39.13 \pm 0.98	8.70 \pm 0.20	34.78 \pm 0.34	73.57 \pm 0.51
Total Elements:					
N	g kg ⁻¹	-	-	4.20 \pm 0.13	32.00 \pm 1.56
P	g kg ⁻¹	-	-	1.90 \pm 0.15	4.60 \pm 0.12
K	g kg ⁻¹	-	-	2.20 \pm 0.21	1.90 \pm 0.08
Al	g kg ⁻¹	-	-	38.01 \pm 0.93	3.10 \pm 0.23
Ni	mg kg ⁻¹	25.01 \pm 0.02	14.00 \pm 0.11	9.40 \pm 0.07	108.00 \pm 1.01
Pb	mg kg ⁻¹	35.08 \pm 0.17	14.00 \pm 0.11	76.00 \pm 0.17	143.00 \pm 0.64
Cu	mg kg ⁻¹	30.22 \pm 0.79	43.21 \pm 0.22	49.00 \pm 0.02	128.00 \pm 0.44
Cd	mg kg ⁻¹	3.30 \pm 0.18	2.10 \pm 0.11	3.00 \pm 0.02	4.00 \pm 0.15
DTPA-Extractable Metals:					
Ni	mg kg ⁻¹	8.92 \pm 0.04	5.13 \pm 0.05	2.49 \pm 0.07	12.12 \pm 0.24
Pb	mg kg ⁻¹	6.13 \pm 0.02	2.18 \pm 0.08	1.58 \pm 0.04	62.13 \pm 0.22
Cu	mg kg ⁻¹	9.09 \pm 0.03	3.13 \pm 0.05	1.20 \pm 0.1	11.83 \pm 0.15
Cd	mg kg ⁻¹	0.33 \pm 0.02	0.18 \pm 0.02	0.09 \pm 0.02	0.72 \pm 0.04

Table 2. Equilibrium model constants, determination coefficient and standard error of estimate for P adsorption by the clay soil treated with biosolids, WTRs and nWTRs

Models	Parameter	clay					
		Untreated	Biosolids(B)	B+WTR	B+0.1nWTR	B+0.2nWTR	B+0.3nWTR
Freundlich $q_e = K_F C_e^{1/n}$	K_F (mL g ⁻¹)	1.28	3.82	11.73	8.88	30.48	96.03
	1/n	1.218	1.134	1.097	1.202	0.964	0.782
	R ²	0.96	0.84	0.65	0.78	0.86	0.92
	SE	0.117	0.226	0.333	0.261	0.187	0.133
Langmuir $q_e = q_{max} / (K_L C_e + 1/K_L C_e)$	q_{max} (μg g ⁻¹)	714.286	1250	1428.571	1428.571	1666.66	3333.333
	K_L (L mg ⁻¹)	3.10 X10 ⁻³	3.90X10 ⁻³	0.0146	0.0136	0.0261	0.0286
	R ²	0.99	0.88	0.91	0.74	0.96	0.99
	SE	0.0016	0.0024	0.0015	0.0022	0.0008	0.00013
Elovich $q_e / q_m = K_E C_e \exp(-q_e / q_m)$	q_{max} (μg g ⁻¹)	1111.111	1428.571	1428.571	1666.667	5000	10000
	K_E (Lmg ⁻¹)	2.18X10 ⁻³	3.32 X10 ⁻³	7.31 X10 ⁻³	6.96 X10 ⁻³	4.69 X10 ⁻³	6.48 X10 ⁻³
	R ²	0.46	0.31	0.48	0.53	0.18	0.07
	SE	0.260	0.448	0.559	0.431	0.392	0.408
Temkin $\theta = RT/\Delta Q \ln K_0 C_e$	ΔQ (Jmol ⁻¹)	7.14	8.32	5.57	5.09	6.74	14.92
	K_0 (L g ⁻¹)	19.04	13.99	8.89	9.46	5.47	2.27
	R ²	0.92	0.83	0.55	0.65	0.68	0.73
	SE	0.110	0.140	0.382	0.329	0.276	0.134
Fowler–Guggenheim(FG) $K_{FG} C_e = \theta / (1 - \theta) \exp(2 \theta w / RT)$	W(kJmol ⁻¹)	3.40	3.25	4.25	3.97	2.45	1.45
	K_{FG} (Lmg ⁻¹)	2.94X10 ⁻³	3.51 X10 ⁻³	5.68X10 ⁻³	6.39X10 ⁻³	0.0130	0.0182
	R ²	0.99	0.84	0.72	0.79	0.49	0.34
	SE	0.111	0.387	0.642	0.491	0.500	0.426
Kiselev $k_1 C_e = \theta / (1 - \theta) + k_n \theta$	k_1 (L mg ⁻¹)	0.002	0.0026	0.0133	0.0139	0.0336	0.0335
	kn	11.05	9	1.248	1.25	2.74	1.81
	R ²	0.83	0.68	0.48	0.55	0.74	0.85
	SE	0.0077	0.0146	0.0705	0.0626	0.0903	0.107
Hill–deBoer $K_1 C_e = \theta / (1 - \theta) \exp(\theta / (1 - \theta) - K_2 (RT\theta /$	K_1 (Lmg ⁻¹)	1.53 X10 ⁻³	2.70X10 ⁻³	4.48X10 ⁻³	5.01X10 ⁻³	0.0106	0.0150
	K_2 (kJmol ⁻¹)	20.62	14.82	15.87	15.21	11.19	9.61
	R ²	0.94	0.99	0.88	0.91	0.80	0.80
	SE	0.790	0.212	0.697	0.559	0.564	0.503

Phosphorus sorption kinetics

Kinetic retention using the batch method described by Amacher et al. (1988) was done on the highest concentration of P (240 mg l⁻¹) to quantify adsorption isotherms for P by untreated, WTRs and nWTRs treated soils at 25°C at 0.25, 0.50, 1, 2, 4, 8, 16, and 24 hrs.

Phosphorus sorption-desorption study

The phosphorus retention capacities of each natural (i.e., the control soil) and the WTRs / nWTR- amended soils were determined. Traditional batch equilibration technique was used, in which a solution of known initial element concentration will be shaken with a given mass of the soil for a specified time interval. Differences in element sorption between original waste materials and the amended soils were quantified. Soil-amendments mixtures were shaken with 0, 20, 40, 60, 120, and 240 mg l⁻¹ P in 20 ml of 0.01 M KCl (as a back ground solution) on a reciprocating shaker for the equilibrium time from kinetics experiment. The mixtures were continuously shaken and then centrifuged at 5000 ×g for 15 minutes prior to sampling. At the end of this time

periods, final P concentrations in solution were measured.

Desorption or release experiments were conducted to assess the release of P as well as the extent of hysteresis behavior by the untreated and WTRs or nWTRs treated soils. Sequential dilutions was initiated immediately after the last adsorption step for 240 mg P L⁻¹ initial concentrations of WTRs or nWTR-soil mixture for the treatments of 0.10, 0.20, and 0.30% nWTR to soil. Each desorption step was carried out by replacing the supernatant, followed by adding 30 mL of 0.01 M KCl background solution and shaking for 12 h. Six desorption steps were carried out. The fraction of P desorbed from each soil was calculated based on the change in concentration in solution (before and after desorption). The amount of P released/desorbed was calculated from the difference between concentrations of the supernatant and that of the amount initially sorbed at each desorption step.

Adsorption isotherm models

Sorption equilibrium data were fitted to the following well known adsorption isotherm models in order to determine the adsorption parameters:

$$\text{Freundlich (Freundlich,1906)} \quad q_e = K_F C_e^{1/n} \quad (1)$$

$$\text{Langmuir [Langmuir,1916]} \quad q_e = q_{\max} (K_L C_e / (1 + K_L C_e)) \quad (2)$$

$$\text{Temkin [Temkin,1941]} \quad \theta = RT/\Delta Q \ln K_0 C_e \quad (3)$$

Fowler–Guggenheim[Fowler and Guggenheim, 1939]

$$K_{FG} C_e = \theta / (1 - \theta) \exp(2 \theta w / RT) \quad (4)$$

$$\text{Kiselev[Kiselev,1958]} \quad k_1 C_e = \theta / (1 - \theta) (1 + k_n \theta) \quad (5)$$

$$\text{Hill–de Boer [Hill,1946; De Boer,1953]} \quad K_1 C_e = \theta / (1 - \theta) \exp(\theta / (1 - \theta) - K_2 \theta / RT) \quad (6)$$

$$\text{Elovich(Elovich and Larinov, 1962)} \quad q_e / q_{\max} = K_E C_e \exp(-q_e / q_{\max}) \quad (7)$$

Where:

q_e (mg g^{-1}) = P adsorbed per gram of adsorbent, C_e (mg L^{-1}) = equilibrium P concentration in solution, K_F = a constant related to adsorption capacity of the adsorbent ($\text{mg}^{1-(1/n)} \text{L}^{1/n} \text{g}^{-1}$),

n = a constant, q_{\max} (mg g^{-1}) is the maximum adsorption capacity of the adsorbent, K_L (L mg^{-1}) = Langmuir constant related to the free energy of adsorption, θ = fractional coverage, R = the universal gas constant ($\text{kJ mol}^{-1} \text{K}^{-1}$), T = the temperature (K), ΔQ = ($-\Delta H$) the variation of adsorption energy (kJ mol^{-1}), and K_0 = Temkin constant (L mg^{-1}), K_{FG} = Fowler–Guggenheim constant (L mg^{-1}), w = the interaction energy between adsorbed molecules (kJ mol^{-1}), k_1 = Kiselev constant (L mg^{-1}), k_n = a constant of complex formation between adsorbed molecules, K_1 = Hill–de Boer constant (L mg^{-1}), and K_2 (kJ mol^{-1}) = a constant related to the interaction between adsorbed molecules. A positive K_2 means attraction between adsorbed species and negative value means repulsion, K_E (L mg^{-1}) is the Elovich equilibrium constant.

Kinetics modeling

Four kinetic models were applied to the sorption data to understand the effect of time on the sorption process. The four equations are as follows:

$$\text{First-order rate model [Laidler, 1965]} \quad \ln(q_0 - q) = a - kat \quad (8)$$

$$\text{Elovich model [Elkhatib et al., 1988]} \quad qt = (1/\beta) \ln(\alpha\beta) + (1/\beta) \ln t \quad (9)$$

$$\text{Parabolic Diffusion Model [Laidler, 1965]} \quad q = a + kdt/2 \quad (10)$$

$$\text{Power Function model [Elkhatib, 1992]} \quad q = ka C_0 t/m \quad (11)$$

Where:

q or q_t = P adsorbed (mg kg^{-1}) at time t , q_0 = P adsorbed (mg kg^{-1}) at equilibrium, k_a = apparent sorption rate coefficient, α = the initial adsorption rate ($\text{mg g}^{-1} \text{min}^{-1}$), β = a constant related to the extent of surface coverage (mg g^{-1}), a = a constant; k_d = apparent diffusion rate coefficient, q = adsorbed P (mg kg^{-1}), C_0 = initial P concentration (mg L^{-1}), t = reaction time (min), k_a = sorption rate coefficient (min^{-1}), and $1/m$ = constant.

RESULTS AND DISCUSSION

Characterization and elemental composition of nWTR

The study of Elkhatib et al.,(2015) indicated that the major elements for produced water treatment residual particles are Fe (49.65 %), Si (21.77 %), Cr (7.67 %), Ca (6.9 %), and Al (6.3 %) with small amounts (lower than 2 %) of K, Ti, Mn, and traces (lower than 0.5 %) of S, Ni, Mg, and Zn. Also, they revealed that X-ray diffraction analysis suggested that amorphous iron, aluminum (hydr) oxides, and silicon oxide dominated all nWTR, with no apparent crystalline iron–Al (hydr) oxides. The abundant iron and aluminum in water treatment residual nanoparticles could have great influence on P sorption-desorption in soils-biosolids-nWTR mixture. The nWTR has a specific surface area of $129.0 \text{ m}^2 \text{g}^{-1}$ and has a total pore volume of $0.051 \text{ cm}^3 \text{g}^{-1}$. The specific surface area of nanoscale WTR sample is 2-3 times higher than bulk WTR and therefore, a large enhancement of its reactivity is anticipated.

Phosphorus sorption isotherms for the biosolids amended soils as affected by WTRs and different rates of nWTRs

The effect of nWTRs addition on P sorption by the two biosolids-amended soils studied is shown in Figure (1). In biosolids-amended soils studied, the P sorption isotherms for the clay, and sandy soils were S type and changed from S type to H type isotherm when biosolids-amended soils treated with 0.1-0.3% nWTRs (Fig.1). Various sorption studies suggest that WTRs can retain from 1740 to 37,000 mg P kg^{-1} and that sorbed P is not readily desorbed. Due to nWTRs porosity and amorphous nature and the presence of Al and Fe (hydr) oxides, and high specific surface area, nWTRs have the propensity to adsorb tremendous quantities of anions. Anion sorption onto WTRs should be a function of the WTRs particle size, charge, and surface area.

The nWTRs additions increased the amount of P sorbed by the biosolids-amended clay, and sandy soils due to more sorption sites and aluminum hydroxides added on the soil surface as a result of nWTRs and biosolids additions (Ippolito et al., 2003). The quantity

of P sorbed by the soils followed the trend clay > sandy (Figure 1). These results are in agreement with those of Novak and Watts (2004) who found that WTRs addition increased phosphorus sorption by soil. Mahdy et al. (2007, 2009) reported that addition of WTRs with high rates (30 and 40 g kg⁻¹) to different soil types significantly decreased extractable P in all studied soils. Sorbed P with nWTRs is becoming stable and immobilized over long periods (Makris et al., 2004). Additionally, strong P retention has been explained by reaction with hydrous oxide surfaces, which replaces singly coordinated OH-groups and then undergoes a rearrangement into a more stable binuclear bridge-type bond between cations (Bohn et al., 1985).

Modeling sorption isotherms

The data of P sorption onto biosolids-amended two soils treated with WTR or nWTR was fitted to seven isotherm models for reliable prediction of adsorption parameters including maximum sorption capacity. The parameters of the models tested are presented in Tables (2 and 3). The determination coefficients (R^2) of Freundlich, Langmuir, Elovich, and Kiselev models were significant ($p < 0.05$), but the R^2 values of Hill-de Boer, Fowler-Guggenheim, and Temkin models were not significant. However, SE values of Freundlich, Elovich, Hill-de Boer, Fowler-Guggenheim, and Temkin models were much higher than the SE values of Kiselev and Langmuir models. This indicates the low predictive capability of these models to describe P sorption (Tables 2 and 3). Therefore, Hill-de Boer, Fowler-Guggenheim, and Temkin models were not analyzed further.

Phosphorus sorption conformed to the Langmuir model over the entire concentration range for studied soils with R^2 0.99 ($p > 0.05$). The R^2 and the SE values of Langmuir models for all sorbent-P systems are presented in (Tables 2 and 3). The low values of SE of Langmuir model indicate the goodness of fit of Langmuir model which suggested the homogeneous and monolayer mode of adsorption (Fig.2). The calculated Langmuir q_{max} values for all studied biosolids-amended soils treated with WTR, nWTR ranging from 400 to 1666.7, from 1250 to 3333.3 $\mu\text{g P g}^{-1}$ for sandy, clay soils, respectively (Tables 2 and 3).

The calculated q_{max} values of Langmuir model are in good agreement with the experimental values. Previous studies reported that the adsorption capacity of bulk WTR varied from 0.30 mg P g⁻¹ to 3.5 mg P g⁻¹ depending on the WTR and test conditions (Yang et al. 2006). The bulk WTR used in this study can be seen to have a comparable P adsorption capacity. However, the calculated q_{max} of biosolids-nWTR-amended soils was 4 times higher than q_{max} of biosolids-WTR-amended

soils. The very high P sorption capacity of nWTR-treated soils suggests that the nWTR is a superior sorbent for P (Makris et al., 2004; Li et al., 2010; Kim et al., 2011).

The equilibrium data were modeled using the linearized form of Kiselev model by plotting $1/[C_e(1-\theta)]$ versus $1/\theta$. The simulated isotherm curves of the Kiselev expression are shown in Fig.(3).

The Kiselev model was also successful in describing P adsorption on the two studied biosolids-amended soils treated with WTR or nWTR due to its low SE and high R^2 values (Tables 2, and 3). The equilibrium constant of the formation of complex between adsorbed molecules, k_n , is positive which indicates formation of complexes between the adsorbed phosphate molecules.

Sorption Kinetics

The Kinetics of P sorption on the two studied soils at 240 ppm P concentration and a temperature of 25 °C are illustrated in Figure (4).

Kinetics studies are important to get insight into the rate of the adsorption process. The effects of contact time (15 min to 24 h) on phosphate adsorption kinetics by the two un-amended and biosolids-amended soils treated with WTRs and nWTRs were evaluated. Phosphorus adsorption by the un-amended soils was biphasic, consisting of a rapid sorption phase followed by a much slower sorption phase. The sorption of P on untreated soils was initially fast with >60% of initially P sorbed in the first 450 min.

Kinetics of P sorption by bulk WTR in biosolids-treated soils revealed that approximately >80 % of P was adsorbed within the first 120 min and slowly preceded to 100% sorption by the end of the 24h period (Figures 4), but kinetics of P by nWTR in biosolids-treated soils revealed that approximately 95% of P was adsorbed within the first 90 min and slowly preceded to 100% sorption by the end of the 24h period (Figures 4). Similar biphasic P kinetic sorption data were obtained for the Fe-WTR (Makris et al., 2004). The slow stage has been attributed to diffusion into micropores or aggregates of particles (Torrent et al., 1992).

The kinetically driven P adsorption data for the two studied soils treated with biosolids, WTR and nWTR were fitted to first-order, Elovich, Intraparticle diffusion and, Power Function models. The conformity between experimental data and the model predicted values were expressed by the coefficient of determination (R^2) and the standard error of estimate (SE) values (Tables 4, 5). The first order model best described P adsorption on nWTR as the R^2 values of first order model were quite high (significant at $p < 0.01$) and SE values were the lowest (Tables 4, 5 and Fig.5).

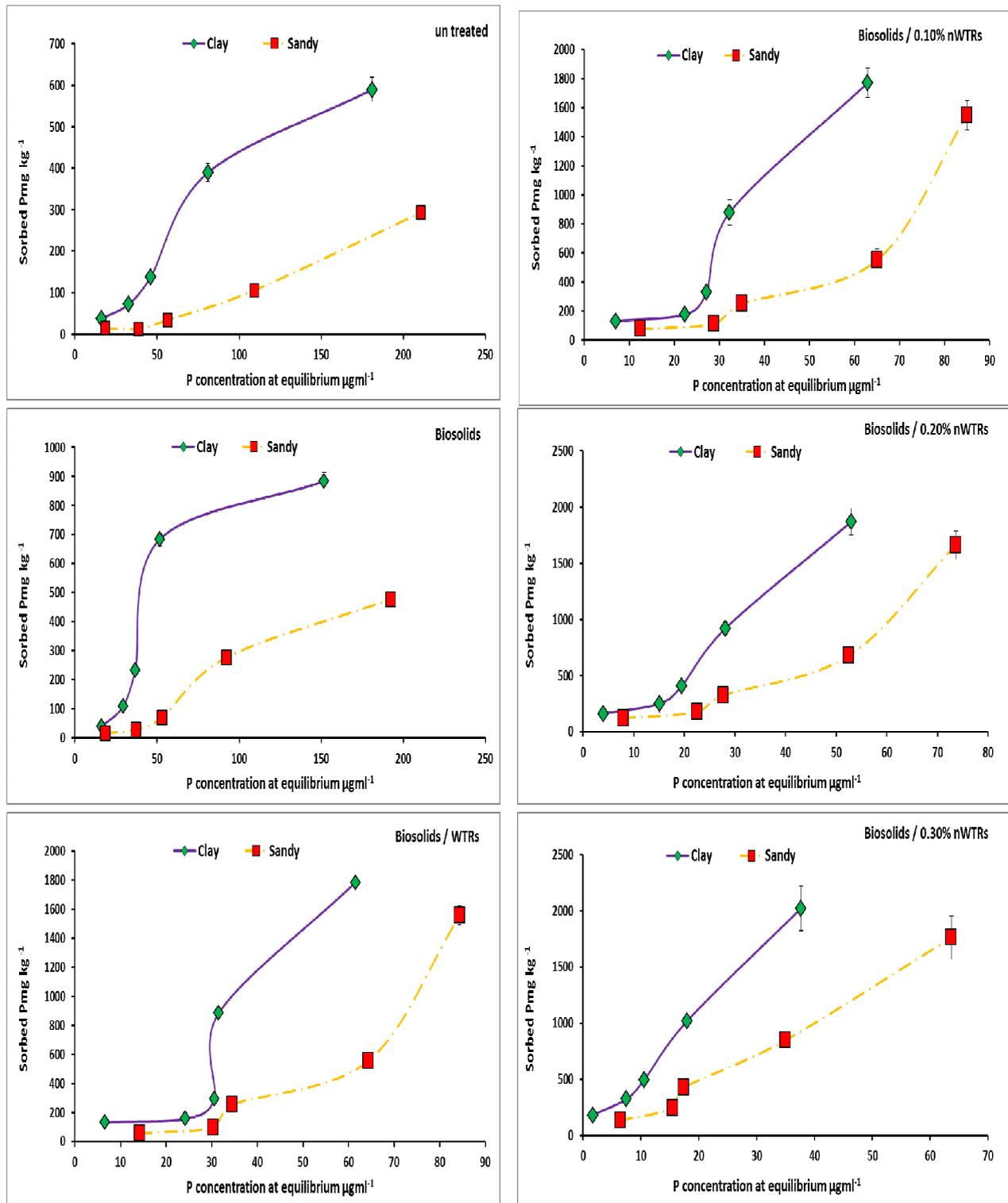


Fig 1. Phosphorus sorption isotherms for two studied soils amended biosolids as affected by different rates of nWTRs. Error bars in all figures represent the standard error of the mean. When no error bars are present, the standard error was too small to be presented as the scale of the diagram

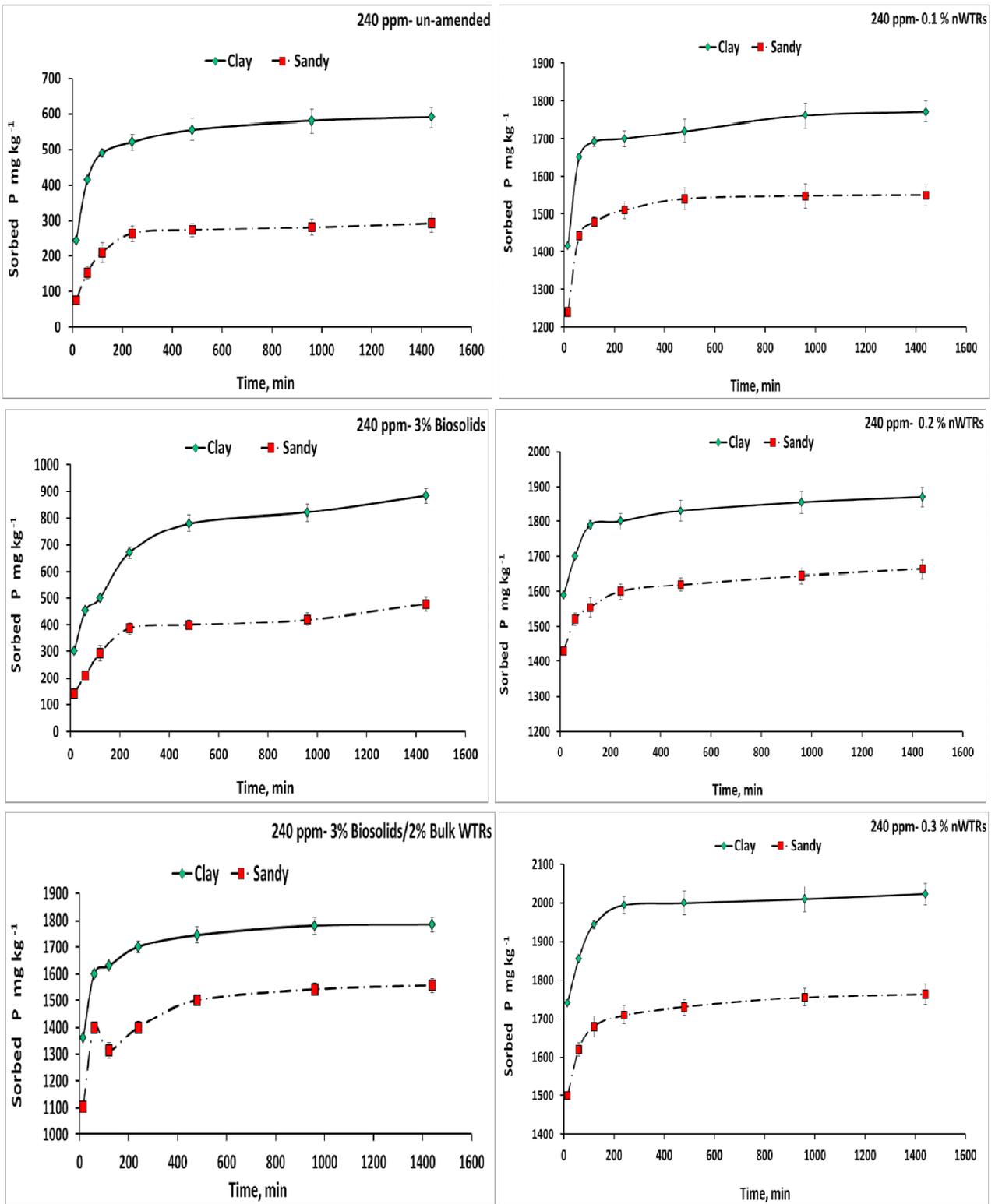


Fig 2. Kinetics of P sorption on the two studied soils at 298K and 240 ppm P concentration

Table 3. Equilibrium model constants, determination coefficient and standard error of estimate for P adsorption by the clay soil treated with biosolids, WTRs and nWTRs

Models	Parameter	sandy					
		Untreated	Biosolids (B)	B+WTR	B+0.1nWTR R	B+0.2nWTR TR	B+0.3nWTR R
Freundlich $q_e = K_f C_e^{1/n}$	$K_f (mL g^{-1})$	1.001	1.178	2.933	1.103	7.83	14.10
	$1/n$	1.058	1.138	1.825	1.536	1.155	1.1498
	R^2	0.997	0.93	0.92	0.89	0.88	0.97
	SE	0.026	0.146	0.190	0.200	0.183	0.087
Langmuir $q_e = q_{max} (K_L C_e / (1 + K_L C_e))$	$q_{max} (\mu g g^{-1})$	357.1429	400	454.5455	833.3333	1000	1666.667
	$K_L (L mg^{-1})$	2.99×10^{-3}	5.59×10^{-3}	7.71×10^{-3}	6.63×10^{-3}	0.0160	0.0105
	R^2	0.99	0.91	0.90	0.999	0.83	0.99
	SE	0.0022	0.0038	0.0027	0.00026	0.0017	0.00004
Elovich $q_e / q_m = K_E C_e \exp(-q_e / q_m)$	$q_{max} (\mu g g^{-1})$	833.333	909.0909	1000	1111.111	2000	5000
	$K_E (L mg^{-1})$	1.35×10^{-3}	1.85×10^{-3}	4.09×10^{-3}	4.42×10^{-3}	4.98×10^{-3}	3.78×10^{-3}
	R^2	0.51	0.42	0.86	0.87	0.60	0.52
	SE	0.072	0.281	0.299	0.232	0.283	0.173
Temkin $\theta = RT / \Delta Q$ $\ln K_0 C_e$	$\Delta Q (kJ mol^{-1})$	8.22	5.30	1.53	3.16	4.12	5.87
	$K_0 (L g^{-1})$	21.01	20.83	19.18	17.02	10.72	7.86
	R^2	0.87	0.85	0.68	0.64	0.66	0.85
	SE	0.129	0.214	0.892	0.507	0.430	0.180
Fowler–Guggenheim(FG) $K_{FG} C_e = \theta / (1 - \theta) \exp(2 \theta w / RT)$	$W (kJ mol^{-1})$	2.97	3.424	4.07	3.36	2.54	2.64
	$K_{FG} (L mg^{-1})$	2.81×10^{-3}	3.42×10^{-3}	5.59×10^{-3}	5.01×10^{-3}	9.38×10^{-3}	0.0102
	R^2	0.97	0.90	0.93	0.91	0.75	0.86
	SE	0.139	0.313	0.294	0.265	0.358	0.1993
Kiselev $k_1 C_e = \theta / (1 - \theta) + k_n \theta$	$k_1 (L mg^{-1})$	0.0026	0.0062	0.0133	0.0066	0.0156	0.0261
	kn	5.31	0.968	1.38	1.29	0.147	4.36
	R^2	0.86	0.62	0.48	0.65	0.73	0.50
	SE	0.0074	0.024	0.050	0.0248	0.0344	0.138
Hill–deBoer $K_1 C_e = \theta / (1 - \theta) \exp(\theta / (1 - \theta) - K_2 (RT \theta /$	$K_1 (L mg^{-1})$	1.31×10^{-3}	2.43×10^{-3}	4.38×10^{-3}	3.63×10^{-3}	7.1×10^{-3}	8.72×10^{-3}
	$K_2 (kJ mol^{-1})$	20.86	16.04	14.80	14.89	13.97	10.91
	R^2	0.93	0.97	0.96	0.97	0.93	0.96
	SE	0.793	0.417	0.372	0.329	0.484	0.209

Table 4. Equilibrium model constants, determination coefficient and standard error of estimate for P adsorption by the sandy soil treated with biosolids, WTRs and nWTRs

Models	Parameter	clay					
		Untreated	Biosolids(B)	B+WTR	B+0.1nWTR	B+0.2nWTR	B+0.3nWTR
Elovich	$\alpha mg g^{-1} min^{-1}$	2.76×10^2	2.47×10^2	5.36×10^7	1.46×10^{10}	2.55×10^{12}	1.52×10^{13}
	$B mg g^{-1}$	0.0138	7.51×10^{-3}	0.011	0.015	0.0168	0.016
	R^2	0.92	0.979	0.92	0.83	0.93	0.89
	SE	37.99	34.31	45.94	54.52	27.96	37.38
First order	$K_d min^{-1}$	-0.003	-0.002	-0.004	-0.003	-0.002	-0.002
	$a \mu g g^{-1}$	5.329	6.153	5.682	5.209	5.153	4.912
	R^2	0.93	0.92	0.98	0.87	0.89	0.77
	SE	0.383	0.29	0.27	0.46	0.390	0.678
Parabolic diffusion	$K_d \mu g g^{-1} min^{-1/2}$	8.0769	16.38	9.941	7.380	6.756	6.733
	$a \mu g g^{-1}$	336.41	327.7	1474	1536	1652	1814
	R^2	0.69	0.89	0.697	0.59	0.73	0.65
	SE	74.82	76.79	89.75	84.8	56.88	67.7
Power function	$K_a min^{-1}$	180.76	124.68	1223.77	1328.92	1476.73	1602.51
	$1/m$	0.1781	0.2655	0.0561	0.0426	0.0343	0.0362
	R^2	0.85	0.98	0.90	0.80	0.92	0.90
	SE	0.0575	0.03	0.014	0.015	0.007	0.008

Elovich and parabolic diffusion models did not fit satisfactorily the adsorption data. The R^2 values of both models were low (not significant at $p < 0.05$) and their SE values were quite high (Tables 4, 5).

Also, power function model was best described P adsorption on nWTR as the R^2 values of power function model were quite high (significant at $p < 0.01$) and SE values were the lowest (Tables 4, 5 and Fig.6).

The lower SE values of power function model than those of Elovich and parabolic models indicating that power function model predictive capability to describe sorption data is accurate (Tables 4 & 5). Therefore, based on SE values, Power function and first order models are the most suitable model to describe P chemisorption by nWTR suggesting that the adsorbate can be bound to different binding sites on the adsorbent.

Table 5. Kinetics model constants, determination coefficient and standard error of estimate for P adsorption by bulk WTR and nWTR-biosolids treated clay soil

Models	Parameter	clay					
		Untreated	Biosolids (B)	B+WTR	B+0.1nWTR	B+0.2nWTR	B+0.3nWTR
Elovich	$\alpha \text{mg g}^{-1} \text{min}^{-1}$	2.76×10^2	2.47×10^2	5.36×10^7	1.46×10^{10}	2.55×10^{12}	1.52×10^{13}
	$B \text{mg g}^{-1}$	0.0138	7.51×10^{-3}	0.011	0.015	0.0168	0.016
	R^2	0.92	0.979	0.92	0.83	0.93	0.89
	SE	37.99	34.31	45.94	54.52	27.96	37.38
First order	$K_d \text{min}^{-1}$	-0.003	-0.002	-0.004	-0.003	-0.002	-0.002
	$a \mu\text{g g}^{-1}$	5.329	6.153	5.682	5.209	5.153	4.912
	R^2	0.93	0.92	0.98	0.87	0.89	0.77
	SE	0.383	0.29	0.27	0.46	0.390	0.678
Parabolic diffusion	$K_d \mu\text{g g}^{-1} \text{min}^{-1/2}$	8.0769	16.38	9.941	7.380	6.756	6.733
	$a \mu\text{g g}^{-1}$	336.41	327.7	1474	1536	1652	1814
	R^2	0.69	0.89	0.697	0.59	0.73	0.65
	SE	76.79	76.79	89.75	84.8	56.88	67.7
Power function	$K_a \text{min}^{-1}$	180.76	124.68	1223.77	1328.92	1476.73	1602.51
	$1/m$	0.1781	0.2655	0.0561	0.0426	0.0343	0.0362
	R^2	0.85	0.98	0.90	0.80	0.92	0.90
	SE	0.0575	0.03	0.014	0.015	0.007	0.008

Table 6. Kinetics model constants, determination coefficient and standard error of estimate for P adsorption by bulk WTR and nWTR-biosolids treated sandy soil

Models	Parameter	Sandy					
		Untreated	Biosolids (B)	B+WTR	B+0.1nWTR	B+0.2nWTR	B+0.3nWTR
Elovich	$\alpha \text{mg g}^{-1} \text{min}^{-1}$	1.06×10^2	1.72×10^2	1.87×10^6	5.18×10^9	1.076×10^{13}	1.52×10^{13}
	$B \text{mg g}^{-1}$	0.0206	0.0135	0.011	0.016	0.020	0.016
	R^2	0.93	0.96	0.88	0.84	0.98	0.89
	SE	23.32	26.72	60.77	48.92	11.93	25.87
First order	$K_d \text{min}^{-1}$	-0.002	-0.001	-0.003	-0.004	-0.002	-0.003
	$a \mu\text{g g}^{-1}$	4.832	5.463	5.765	5.087	5.085	5.112
	R^2	0.798	0.76	0.94	0.94	0.92	0.93
	SE	0.588	0.3997	0.344	0.482	0.294	0.352
Parabolic diffusion	$K_d \mu\text{g g}^{-1} \text{min}^{-1/2}$	5.48	8.89	10.78	6.63	5.89	6.28
	$a \mu\text{g g}^{-1}$	120.82	169	12.3	1350	1467	1564
	R^2	0.72	0.84	0.66	0.57	0.82	0.72
	SE	47.44	53.84	89.94	79.18	37.97555	54.39
Power function	$K_a \text{min}^{-1}$	44.29	75.49	965.38	1127.46	1324.34	1398.30
	$1/m$	0.286	0.264	0.069	0.051	0.032	0.034
	R^2	0.86	0.94	0.86	0.85	0.98	0.92
	SE	0.087	0.051	0.021	0.016	0.004	0.007

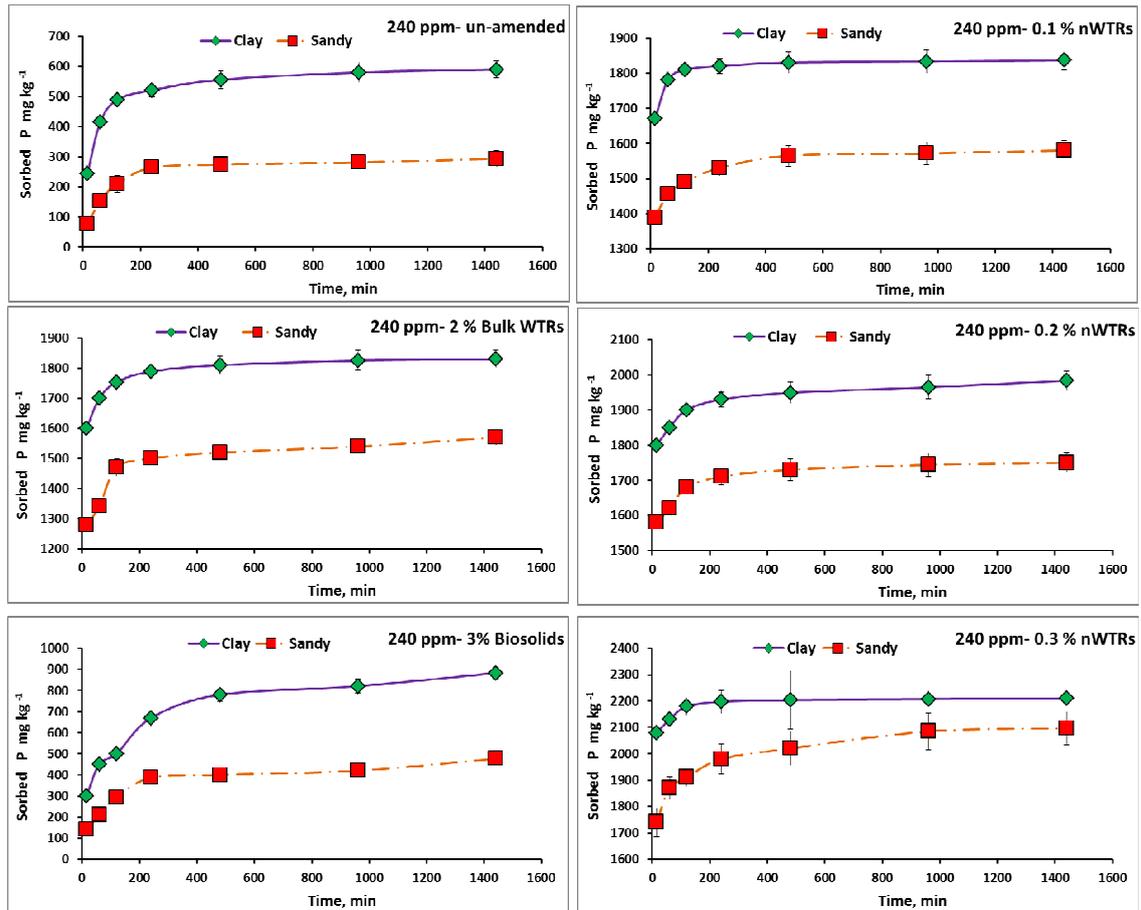


Fig 3. Kinetics of P sorption on two studied soils at 298K and 240 ppm concentration

The mobility of P in soil can be assessed from desorption studies. The selection of treatment technique is based on its availability in the liquid phase. Desorption which is the reversibility to sorption process plays significant role in determining chemical mobility of P in soil (Seybold and Mersie., 1996).

In the present study the results shows that desorption of P percentage from the biosolids- treated clay soil was about 10-18 %. However, addition of bulk WTR reduced the desorption percentage to 6-8%. Similarly, nWTRs application with different rates drastically reduced the percentage of desorbed P to 6, 4, and 1% at 0.10, 0.20 and 0.30% application rate, respectively (Figure 7). On the other hand, the desorption percentage of P in biosolids-sandy soil was 85% and higher than that of clay soil. Adding of 2% WTR reduced the desorption percentage of P in biosolids-treated sandy soil to 12-17%. WTR nanoparticles significantly reduced the desorption percentage to 12, 7 and 4% at 0.10, 0.20 and 0.30% application rate, respectively.

Hysteresis is manifested by an increase in the difference between the adsorption and desorption isotherm slopes and has been well documented (Carrizosa et al., 2001). Conceptually, the lack of similarity between adsorption and desorption due to the hysteresis is likely a result of binding to organic matter and mineral particles, particularly Al/Fe hydroxides and clay minerals (Neville et al., 2000).

Desorption Kinetic

The Kinetics of P desorption on the two studied soils at 120 and 240 ppm concentrations and a temperature of 25 °C are illustrated in Figures (8).

Kinetics studies are important to get insight into the rate of the desorption process. The effects of contact time (6 to 36 h) on phosphate desorption kinetics by the two un-amended studied soils were evaluated. Phosphorus desorption from the un-amended soils was much higher than that of WTR or nWTRs-amended studied soils. The order of P desorption from studied soils were: sandy > Clay soils (Fig. 8).

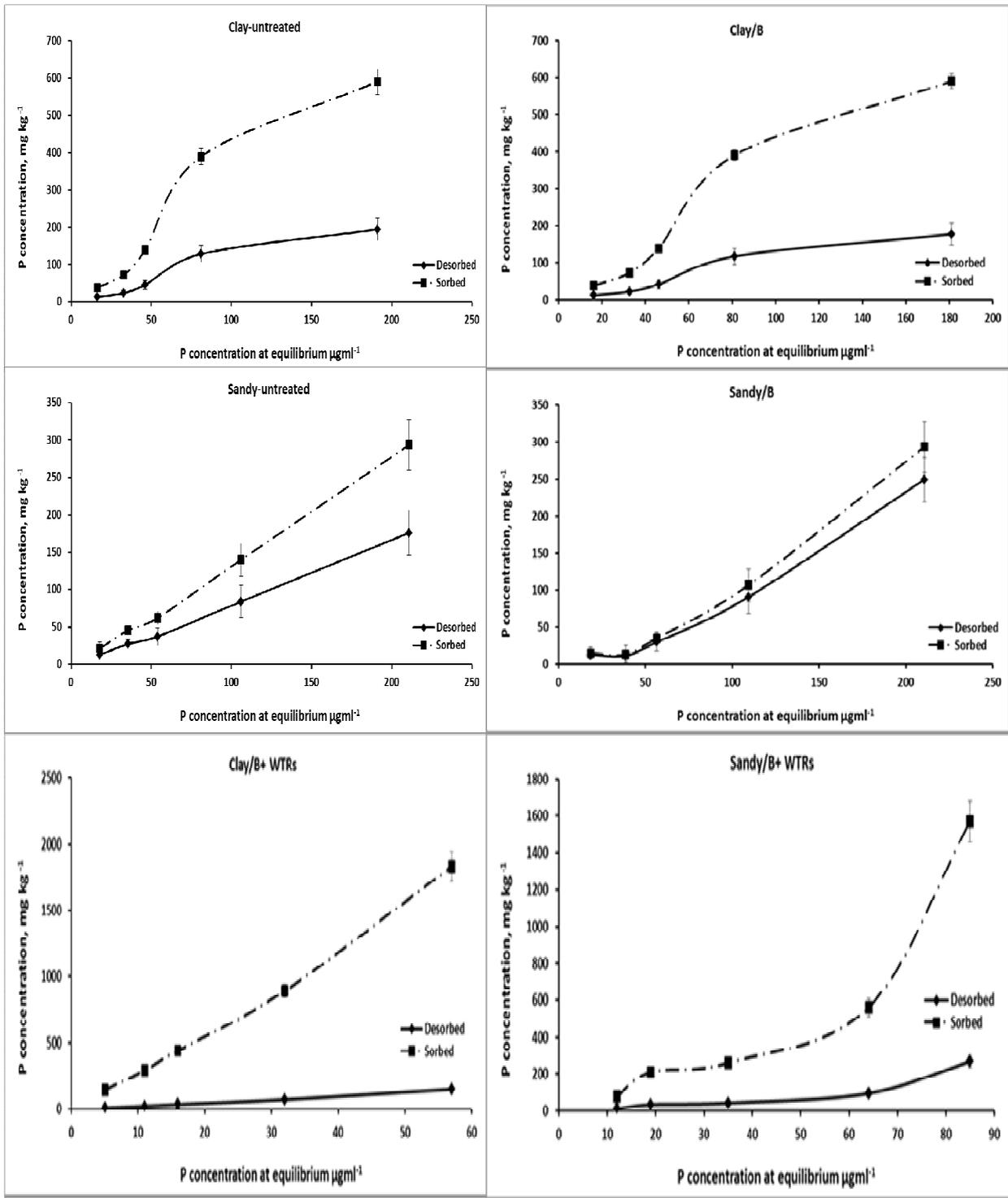


Fig 4. Sorption and desorption of P in biosolids-WTRs- treated and untreated clay and sandy soils

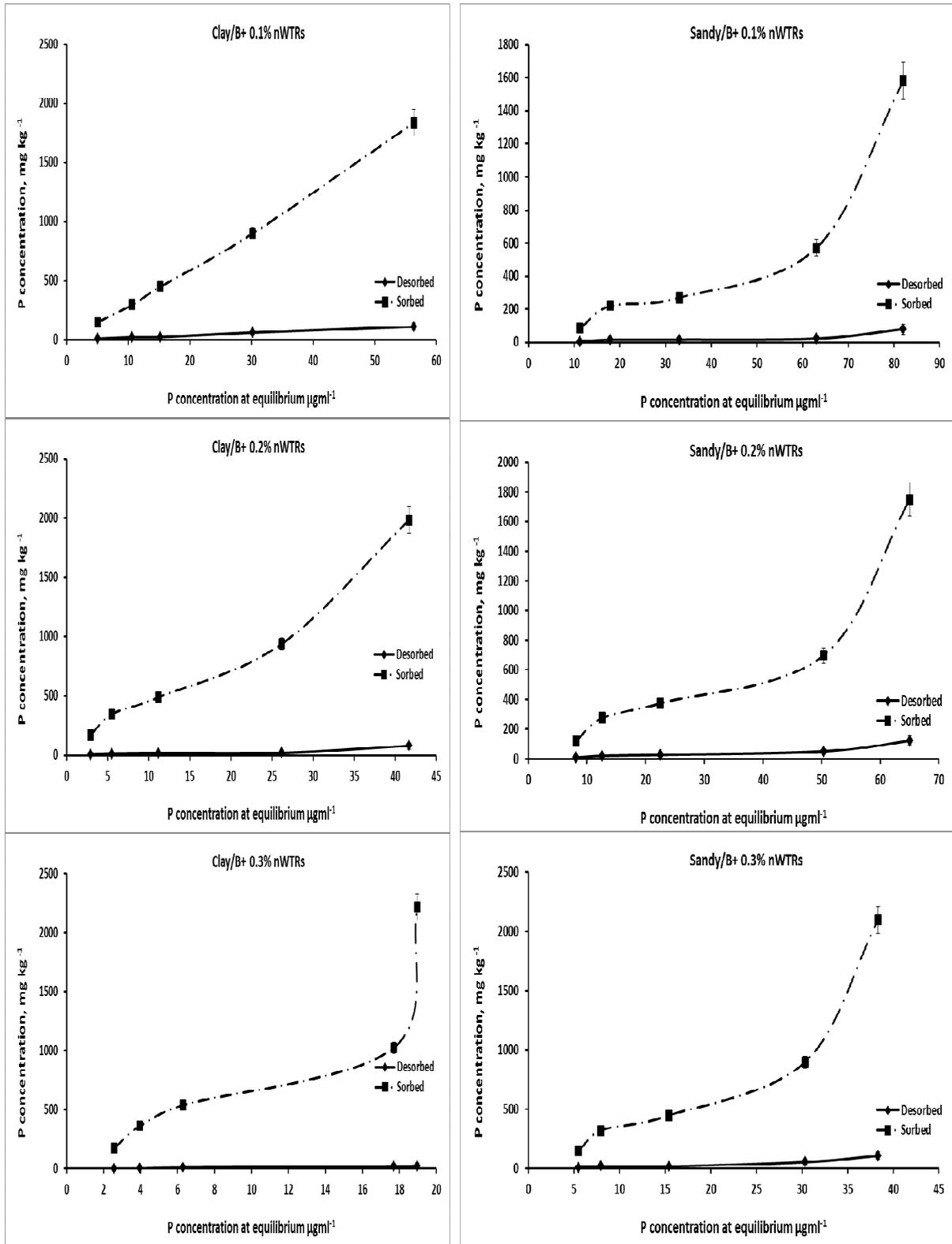


Fig 5. Sorption and desorption of P in biosolids-nWTRs- treated clay and sandy soils

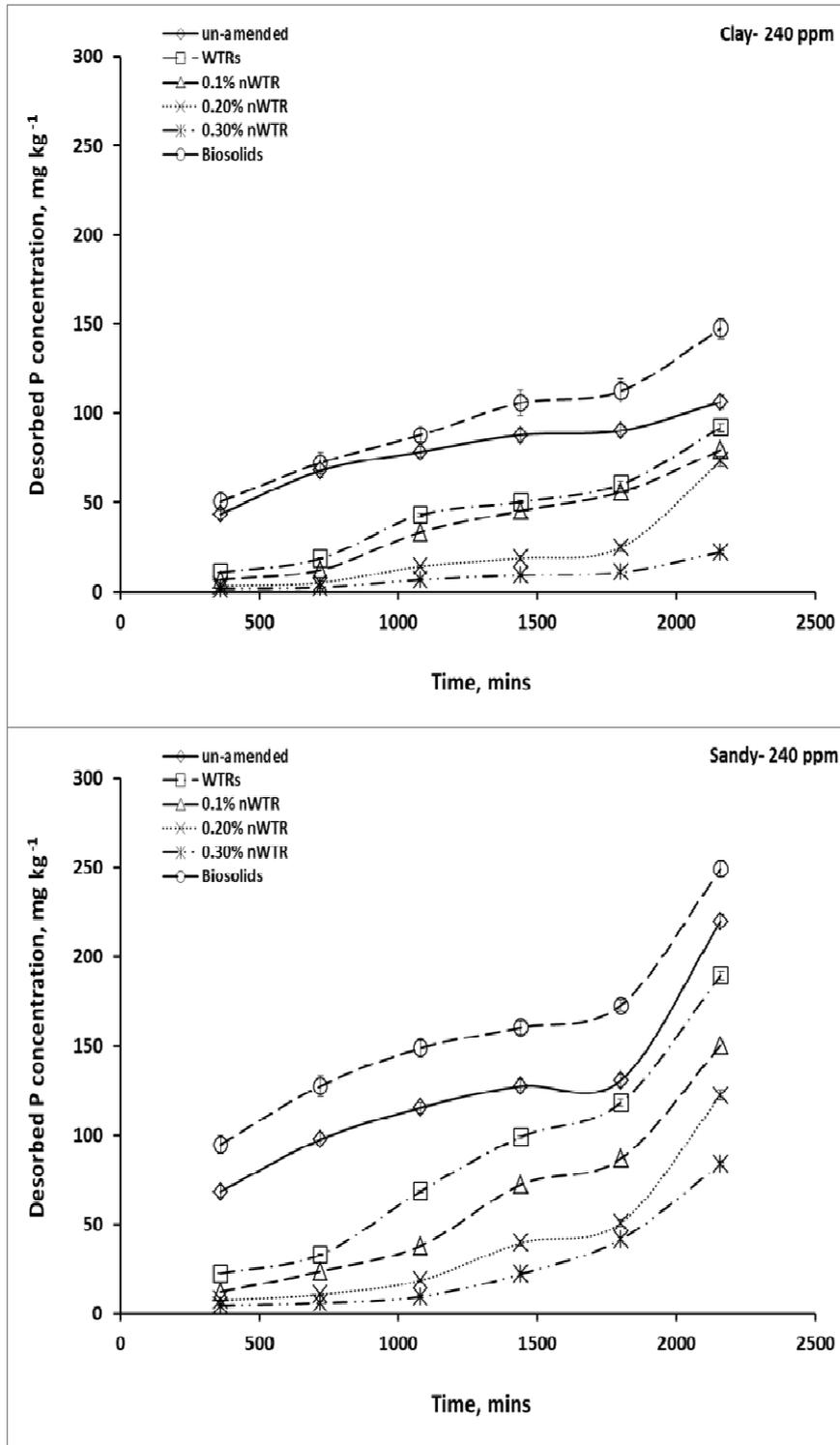


Fig 6. Kinetics of P desorption from two studied soils at 298K and 240 ppm concentration

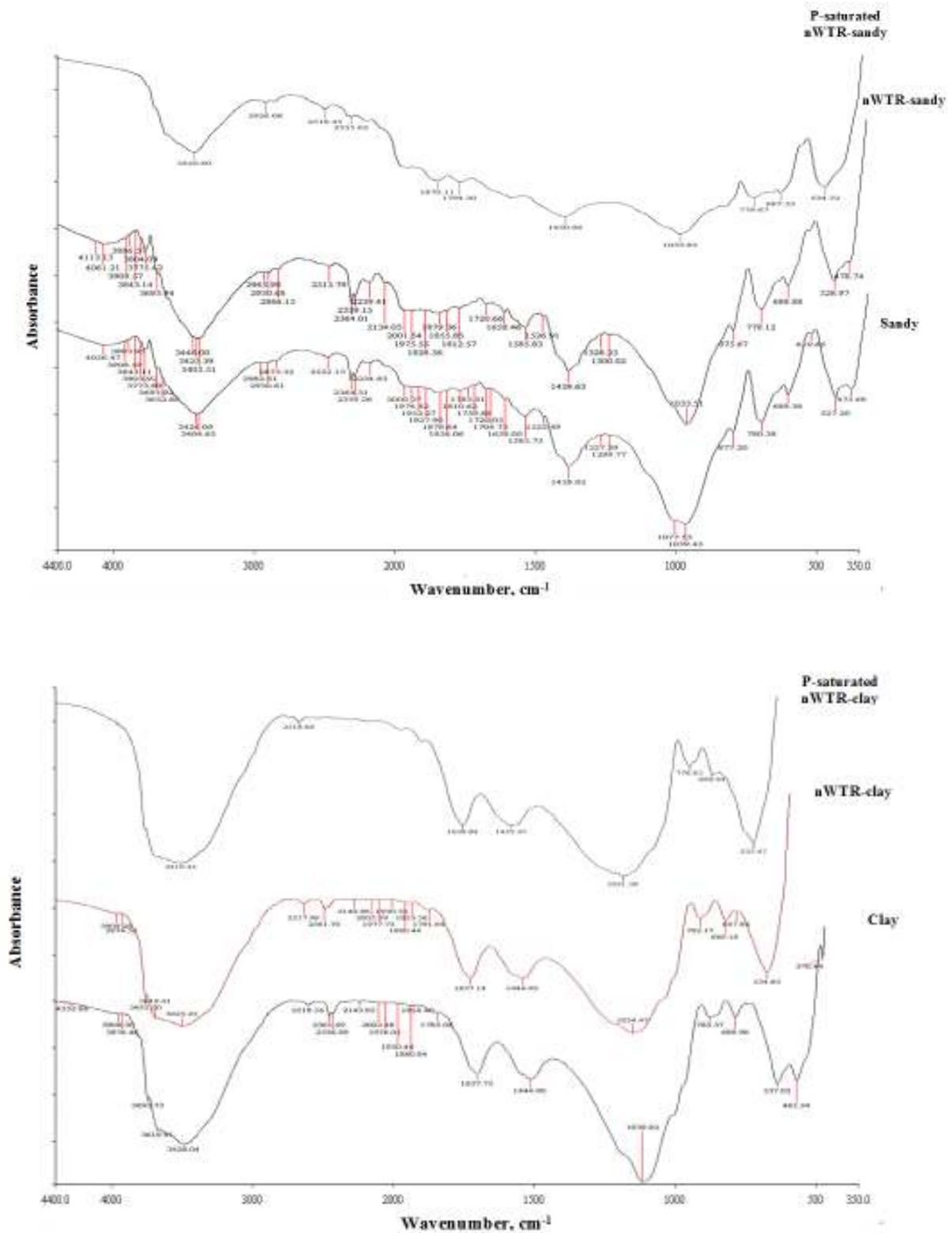


Fig 7. FTIR spectrum of two studied soils amended with nWTR before and after P adsorption

Kinetics of P desorption from bulk WTR and/or nWTR in biosolids-treated soils revealed that only 2-6% of sorbed P is readily release after 36 hrs of contact time.

In general, the orders of P desorption from untreated and treated soils were: Biosolids > untreated > treated soils sandy > clay soils (Fig. 8). The slow desorption rate has been attributed to diffusion into micropores or aggregates of particles and strongly sorbed P due to presence of amorphous aluminum and iron oxides present in nWTR-biosolids-treated soils (Torrent et al., 1992; Elkhatab et al., 2015).

Fourier transmission infrared spectroscopy (FTIR)

The FTIR analysis was carried out to further investigate P adsorption mechanism onto nWTR amended soils. The FTIR spectra of clay soil before and after nWTR application are presented in Fig. 2. The spectrum of clay soil displays various absorbance bands in the 400-4000 cm^{-1} region, which are centered at 3428, 2517, 1637, 1444, 1030, 783, 689, 537, and 465 cm^{-1} . These main absorption bands are attributed to OH stretching, water association with clay, CO_3^{2-} stretching vibration, stretching vibrations of Si-O groups, presence of quartz mixtures, Al-O, Al-OH vibrations and Al-O-Si and Si-O-Si bending vibrations respectively (Vlaev et al. 1989; Madejova 2003; Janik et al. 2007 a, b; Spence and Kelleher 2012; Lu et al. 2012; Bruckman et al. 2013). Comparison of FTIR spectrum of clay soil with FTIR spectrum of clay soil amended with 0.30% nWTR (Fig. 9) shows a shift of Si-O stretching vibrations band at 1030 cm^{-1} to higher wave number and disappearance of Si-O-Si vibration band at 465 cm^{-1} . It is important to note, however, that Al-O-Si vibration band has been broaden and shifted to lower frequency at 534 cm^{-1} as a result of increasing Al content in the sample due to nWTR addition. On P saturated nWTR-clay soil, the OH stretching bands at 3909 and 3876 cm^{-1} disappeared which demonstrated the involvement of OH group in P retention by nWTRs amended soil.

The FTIR spectrum of the sandy soil (Fig. 9) displays prominent bands at 3773, 3404, 1439, 1039, 780, 527 and 473 cm^{-1} which are attributed to OH stretching, H bonded water, $\gamma\text{-Al}_2\text{O}_3$, CO_3^{2-} stretching vibration, quartz mixture, Al-O-Si and Si-O-Si bending vibrations respectively (Vlaev et al. 1989; Madejova 2003; Janik et al. 2007 a, b; Bermudez 2010; Spence and Kelleher 2012; Lu et al. 2012; Bruckman et al. 2013). Application of 0.30% nWTR to the sandy soil resulted in disappearance of OH stretching band at 3773 cm^{-1} and the shift of H bonded water band at 3404 cm^{-1} to higher wave numbers.

The retention of P ions influences the FTIR spectrum of nWTR amended sandy soil as follows: 1) reduction and shift of the H bonded water band (3404 cm^{-1}) and $\gamma\text{-Al}_2\text{O}_3$ band (1439 cm^{-1}) to lower wave numbers which suggests inner-sphere adsorption of P ions to surface functional groups on the nWTR (Bruckman et al. 2013). 2) Disappearance of Si-O-Si bending vibrations band which indicates the interaction of Si-O-Si of nWTR with P ions.

The FTIR spectra of calcareous and nWTR-calcareous soil mix before and after P adsorption are shown in Fig. 4. The spectrum of calcareous is characterized by absorption bands near 3000-3700 cm^{-1} , due to hydrogen-bonded water and hydroxide ion; and a strong band between 1430 and 1500 cm^{-1} , due to the CO_3^{2-} stretching vibration. The changes in FTIR spectrum of 0.30% nWTR amended calcareous soil as a result of P adsorption is evident. Most of the prominent bands has been diminished, flattened and shifted to lower wave numbers. Consequently, the adsorption mechanism between nWTR-calcareous adsorbents and the phosphate may result from the ion exchange between the hydroxyl on the adsorbent surface and phosphate in the solution in addition to adsorption on surface of carbonate-rich minerals present in calcareous soil.

CONCLUSIONS

Addition of nWTR caused an overall increase in P sorption in clay and sandy soils treated and un-treated with biosolids due to more sorption sites and aluminum hydroxides added on the soil surface as a result of nWTRs and biosolids additions. Phosphorus desorption from the un-amended soils was much higher than that of WTR or nWTRs-amended studied soils. The order of P desorption from studied soils were: sandy > Clay soils. Kinetics of P desorption from bulk WTR and/or nWTR in biosolids-treated soils revealed that only 2-6% of sorbed P is readily release after 36 hrs of contact time. In conclusion, the addition of nWTR increased the proportion of immobilized phosphate in the studied soils and becomes more and more irreversible with the progress of P absorption that occurs by penetrating from outer layer to the inner layer of the subject granular. Thus, potentials for loss of P to water resource can be reduced.

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المخلص العربي

تقييد حركية الفوسفور في الاراضي المعاملة بالحمأة بواسطة الحبيبات النانومترية لمخلفات تنقية مياه الشرب

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فوسفور/ لتر وأشارت النتائج ان زيادة معدل اضافة الحبيبات النانومترية لمخلفات تنقية مياه الشرب أدت الى زيادة الكمية المدمصة من الفوسفور وكذلك خفض الكمية المتحررة من الأراضى. وأشارت نتائج تحليل ال FTIR الى إرتباط المجاميع الفعالة فى مخلفات تنقية مياه الشرب النانومترية بالفوسفور مكونة معقد بين الفوسفور والمجاميع الفعالة مما يترتب عليه إختفاء هذه المجاميع الفعالة فى كل الاراضى المدروسة بعد اضافة مخلفات تنقية مياه الشرب النانومترية مما يوضح الدور الحيوى الذى تلعبه مجاميع الهيدروكسيل السطحية فى مسك الفوسفور على سطح التربة وعدم تحرره عند إضافة الحبيبات النانومترية لمخلفات تنقية مياه الشرب.

الكلمات المفتاحية: الفوسفور - حركية - امتصاص -

الحبيبات النانومترية

أجريت هذه الدراسة بهدف معرفة تأثير اضافة مخلفات تنقية مياه الشرب النانومترية على تفاعلات ادمصاص وحركية الفوسفور فى الأراضى المعاملة بمخلفات معالجة مياه الصرف الصحى(الحمأة). أجريت الدراسة على نوعين مختلفين من الأراضى المصرية (رملية - طينية). وأشارت النتائج إلى أن الأرض الطينية لديها قابلية أعلى لادمصاص الفوسفور من الأراضى الرملية وكان منحنى الادمصاص للأراضى موضع الدراسة من النوع S. استخدمت معادلة Freundlich و Langmuir و Elovich و Temkin و Fowler-Guggenheim و Hill-de Boer بنجاح فى وصف أدمصاص الفوسفور. كما استخدمت معادلات Elovich و Parabolic diffusion و modified Freundlich والدرجة الاولى بنجاح فى وصف حركية وتحرر الفوسفور المدمص على الأراضى المدروسة عند التركيز ١٢٠ و ٢٤٠ مليجرام