

## Research Article

# Temperature Effects on Phosphorus Release from a Biosolids-Amended Soil

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Received 10 July 2013; Revised 23 August 2013; Accepted 23 August 2013

Academic Editor: Leonid Perelomov

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This study was designed to evaluate the effects of temperature on the potential leachable P pool and distribution of chemical P forms in a biosolids-amended soil. A P-deficient Spodosol was incubated with seven biosolids and inorganic P fertilizer at 20 and 32°C for 90 days. Amendments were applied to provide a total P concentration of 112 mg kg<sup>-1</sup> soil, which correspond to a field application of ~224 kg P ha<sup>-1</sup>. Cumulative P mass leached during the 90 d study for any P source was <2% of the applied P, but greater cumulative P mass was released from the biological P removal and composted biosolids than from the heat-dried materials. Increasing temperature (20 to 32°C) generally decreased cumulative P mass leached, suggesting greater soil affinity to retain P at 32°C than at 20°C. In a static incubation experiment (no leaching), soil water-extractable P concentrations were reduced over time, but no temperature effect was observed. Similarly, P distribution among the various fractions was not affected by temperature. The relatively great ability of the soil to sorb P masked differences in biosolids properties and the potential impacts of temperature on P lability. Additional work using low P-sorbing soils is warranted.

## 1. Introduction

Biosolids can provide essential nutrients to plant and improve soil chemical, physical, and biological properties [1]. However, repeated biosolids applications based on plant N requirements supply P in excess of crop requirements, resulting in soil P accumulation. Environmental concerns associated with the buildup of soil P and potential losses of P movement to water bodies via surface runoff, vertical leaching, and erosion exist. Although P is typically immobile in most soils, coarse-textured soils are prone to P transport [2]. Florida Spodosols are particularly susceptible to P leaching due to the lack of reactive minerals, low Fe and Al oxides, and organic matter concentrations in surface horizons [3]. Moreover, poorly drained Spodosols associated with relatively shallow water tables intercepted by drainage ditches increase the potential risks of P edge-field losses.

Phosphorus lability in biosolids-treated soils depends on the forms of P is initially present in the biosolids and

the characteristics of the soil receiving the residual. The wastewater treatment processes strongly influence the chemistry and P pools in biosolids [4]. For instance, biological P removal (BPR) processes can increase P extractability and runoff losses [5, 6]. Conversely, biosolids treated with Al and Fe or heat-dried materials generally exhibit low extractable P concentrations [7–9]. According to [10], P lability from biosolids produced via BPR processes was similar to commercial P fertilizer, while in heat-dried materials containing high levels of Al and Fe less than 10% of total P was labile. However, differences in biosolids-P lability in response to biosolids treatment method appear to be less pronounced in P-enriched soils or soils that have high affinity to retain P [11].

Although biosolids have typically a wide range of physical and chemical properties, the vast majority of the P in biosolids is present as inorganic P [12, 13]. Research has shown that P lability in biosolids-treated soils is affected by the organic source as well as the soil P sorption characteristics and initial soil P levels [10, 11]. In addition, P forms

TABLE 1: Selected soil chemical properties.

Soil	Sand %	Organic matter g kg <sup>-1</sup>	pH	Total P	Mehlich-1 P	Oxalate -extractable			PSI <sub>ox</sub> <sup>1</sup>
						P	Fe	Al	
Millhopper	95	19	5.6	1339	5.7	585	2222	956	0.25

<sup>1</sup>Phosphorus saturation index (PSI) =  $[P \div (Al + Fe)]$  with P, Fe, and Al concentrations expressed as mmol kg<sup>-1</sup>.

and availability in biosolids-treated soils can be affected by environmental conditions, such as temperature and moisture content. Temperature can also increase the rate of reaction between soil and added P, resulting in a rapid decrease in soluble P [14]. Phosphorus availability increased in manure-treated soils when temperature increased from 10 to 20°C [15] and in a forest soil receiving P fertilizer [16]. Flooded sediments released much more P when incubated at 35°C than at 7°C [16], and temperature increased P release in flooded soils [17]. Fe-strip P concentrations decreased when biosolids-amended soils were incubated at 37°C than at 25°C, suggesting greater microbial immobilization of soluble P at a higher temperature [18]. Chemical mechanisms involved in P dynamics can also be affected by temperature. P extracted by NaHCO<sub>3</sub> was very sensitive to changes in temperature, increasing by ~3% per °C increase [17, 19]. Contrarily, [20] reported no differences in Olsen-extractable (available) P concentrations in calcareous soils incubated at temperatures varying from 5 to 25°C, possibly due the presence of Ca-based minerals that favored P sorption. It has been suggested that in some cases NaHCO<sub>3</sub> might not be a good indicator of soil P changes due to the increasing temperature [21]. Reference [22] found no significant effect of temperature on the amount of P released from swine slurry-treated soils; however, for soils receiving beef cattle manure, temperature increases increased P sorption in heavier-textured soils and decreased P retention in sandy soils. Phosphate adsorption increased with temperature (25 to 55°C) in five tropical soils, and P desorption was markedly reduced. Biosolids incubated at room temperature for 15 months exhibited greater concentrations of water-soluble P as compared to biosolids samples stored at -2°C [13]. The objective of this study was to investigate the effects of temperature on the potential leachable P pool and distribution of chemical P forms in biosolids-amended soils.

## 2. Material and Methods

**2.1. Soil and Biosolids Samples.** Composite samples of a sand soil (Millhopper soil series (loamy, siliceous, and hyperthermic Grossarenic Paleudults)) were collected from the 0 to 15 cm depth in Santa Fe Beef Research Unit, FL, USA (29°55'N, 82°30'W). Soil samples were air-dried and sieved to pass through a 2 mm screen sieve. Selected chemical characteristics are shown in Table 1. Seven different types of biosolids were collected at different locations and stored at 4°C until the experiment began. Chemical compositions of the various biosolids (Table 2) were similar to biosolids

produced nationwide [23]. Total P concentrations ranged from ~2% to 4%.

**2.2. Incubation Experiment.** An incubation study was designed to determine the potential P leachable pool from soils amended with biosolids and fertilizer. Appropriate amounts of biosolids and P fertilizer (triple super phosphate) were applied to provide a total P concentration of 112 mg kg<sup>-1</sup> soil, which correspond to a field application of ~224 kg P ha<sup>-1</sup> or 10 Mg ha<sup>-1</sup> of a typical biosolids containing between 2% and 2.2% of P. For each treatment, 100 g (eq. dry wt.) of soil were thoroughly mixed with biosolids in plastic bags. Samples were wetted to 80% of the field capacity. Biosolids-treated samples and control soils were incubated aerobically in filter chambers in the dark at 20 and 32°C for 90 days. Chambers containing treated samples were capped to reduce evaporation but aerated weekly by removing lids from containers for 2 hours to reestablish ambient conditions. Soil moisture contents were checked regularly by weighing the chambers, and water was added to maintain the samples at 80% of field capacity. Chambers were leached periodically, as described below, to determine potential P losses. Each treatment was replicated three times.

In a separate experiment, soils were treated with biosolids (at a P rate of 112 mg kg<sup>-1</sup>) and incubated in the dark at 20 and 32°C as described previously but were not subjected to leaching (static incubation). At 0, 15, 30, 45, and 90 days of incubation, subsamples were taken for P analysis. Changes in P forms over time were evaluated using a modification of the sequential extraction procedure developed by [24]. Water-extractable P concentrations were also determined in the treated samples to assess the effects of time and temperature on P extractability.

**2.3. Leaching Protocol.** Samples were leached with 60 mL of DDI water (~2 pore volume) at 15, 30, 60, and 90 days. Immediately after the discharge, a subsample was filtered through a 0.45 μm membrane filter. Filtered leachates were analyzed for total P using the potassium persulfate/sulfuric acid digestion (EPA method 365.1) [25], soluble reactive P (SRP) [26], pH, electrical conductivity (EC), and dissolved organic C (DOC) concentrations.

**2.4. Statistical Analysis.** Statistical analyses were performed using Proc Mixed [27]. Biosolids type and temperature were considered fixed effects, with replicates and their interactions being considered as random effects. Mean separation of treatment differences was by LSD. Treatments and their interactions were considered significant when *F*-test *P* values

TABLE 2: Selected properties of biosolids materials.

Biosolids	Treatment process	Class	pH	Solids %	C	Total			Oxalate-extractable			PSI					
						N	P	P	Al	Fe	P		Al	Fe			
ATAD <sup>1</sup>	Aerobically digested, biological P removal	A	6.3	3	350	55	40	23	3.2	8.3	2.8						
Disney compost <sup>1</sup>	Composted, biological P removal	A	5.7	70	420	28	15	11	7.0	20	0.6						
OC South Cake <sup>1</sup>	Anaerobically digested, biological P removal	B	8.1	15	410	66	23	23	5.0	4.4	2.8						
OC East Cake <sup>1</sup>	Aerobically digested, biological P removal	NA	5.9	16	430	70	20	17	1.3	3.4	5.0						
JEA Cake	Anaerobically digested, thickened	A	7.9	22	360	51	19	18	6.0	5.0	1.9						
Tampa pellets	Anaerobically digested, thermally dried	A	5.6	96	410	56	21	19	6.0	5.1	2.0						
WPB	Composted	A	5.8	67	420	25	19	19	3.0	3.0	3.7						

<sup>1</sup> Denotes biological P removal biosolids.

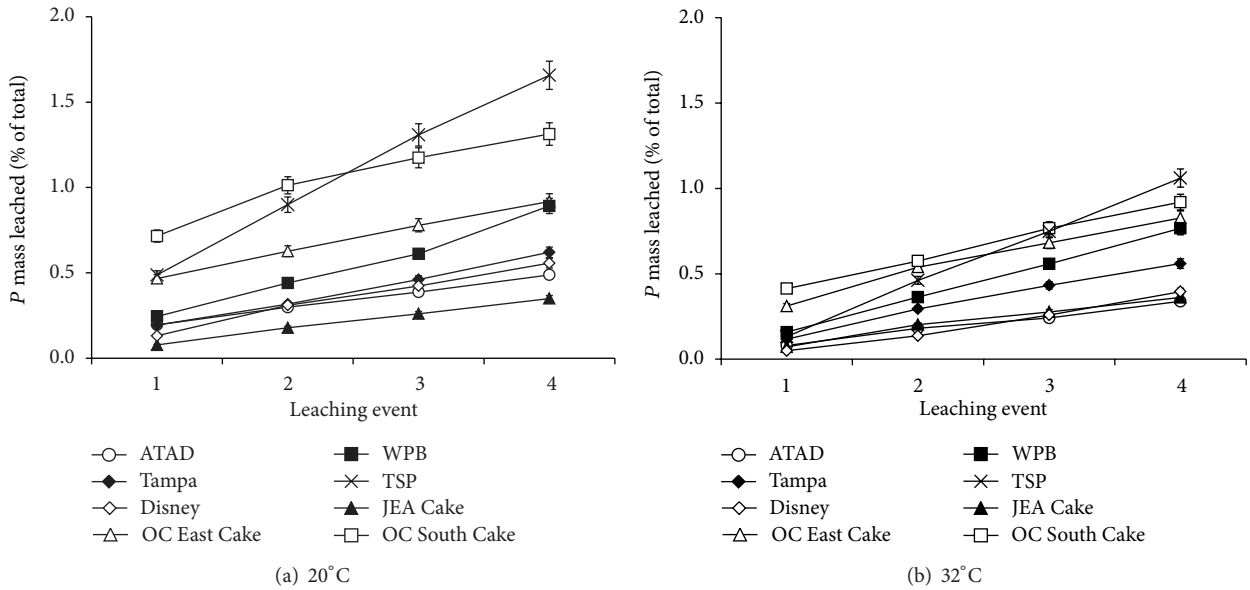


FIGURE 1: Cumulative P mass leached over the 90 d incubation. Leaching events 1, 2, 3, and 4 correspond to 15, 30, 60, and 90 d of incubation. Initial P mass applied 112 mg P kg<sup>-1</sup> soil P (224 kg P ha<sup>-1</sup>). Error bars represent one standard error. Data represent the average of three replicates. Error bars represent one standard error. Open symbols represent biological nutrient removal biosolids.

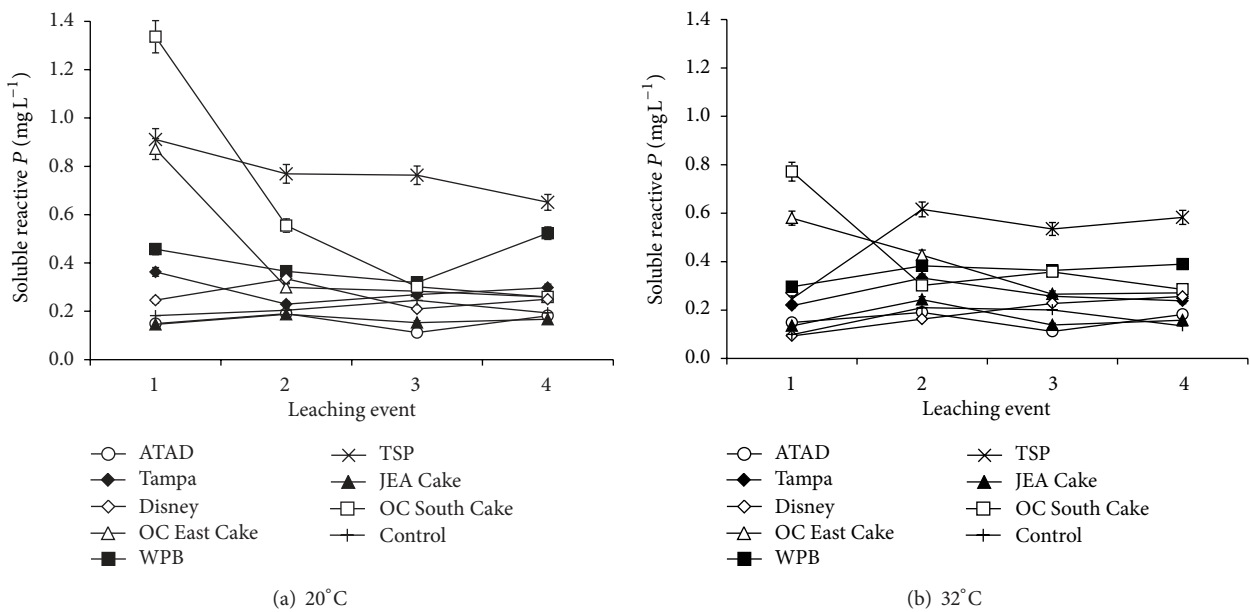


FIGURE 2: Leachate soluble reactive P (SRP) concentrations. Leaching events 1, 2, 3, and 4 correspond to 15, 30, 60, and 90 d of incubation. P rate 112 mg P kg<sup>-1</sup> (224 kg P ha<sup>-1</sup>). Error bars represent one standard error. Data represent the average of three replicates. Open symbols represent biological nutrient removal biosolids.

were <0.05. Interactions not discussed in the results and discussion section were not significant ( $P > 0.05$ ).

### 3. Results and Discussion

3.1. Phosphorus Leaching. After 90 d of incubation and a total of 8 PV of drainage, cumulative P mass leached was

greater for TSP-treated soils than biosolids-treated soils at both temperatures (Figure 1). Biosolids-treated soils leached ~20% to 87% less P than TSP-treated samples, suggesting that P availability in the biosolids was much smaller than in TSP. Greater cumulative P mass was released from two of the Cake materials (OC South and OC East) and from composted biosolids (WPB) as compared to the JEA Cake, Tampa, and Disney biosolids. This indicates that there was a significant

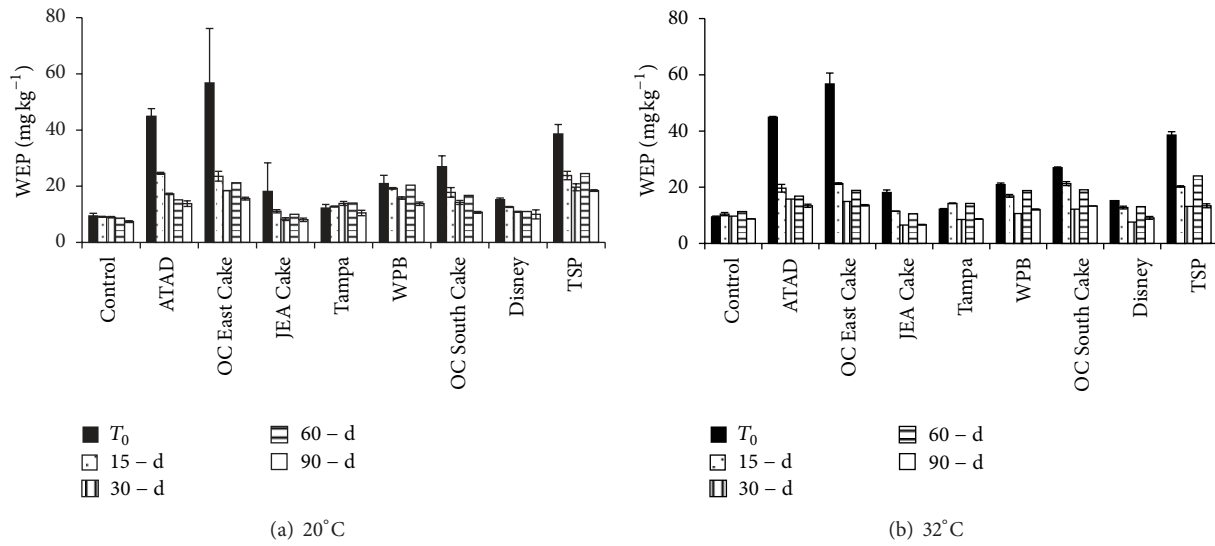


FIGURE 3: Changes in WEP concentrations as affected by incubation period and temperature.

TABLE 3: Leachate dissolved organic C concentrations.

Biosolids	Dissolved organic C (mg L <sup>-1</sup> )							
	1st leaching		2nd leaching		3rd leaching		4th leaching	
	20°C	32°C	20°C	32°C	20°C	32°C	20°C	32°C
Control	11	5	12	12	12	14	12	15
ATAD	39a <sup>1</sup>	10b	13	11	10	10	12	14
OC East Cake	32a	14b	15b	18a	11b	16a	12	13
JEA Cake	26a	11b	14	16	10b	14a	10b	14a
Tampa	38a	16b	16	18	12b	15a	12b	18a
WPB	35a	14b	22	20	21	17	26	23
OC South Cake	19a	10b	18	20	16	14	12	15
Disney	30a	7b	24a	12b	13	18	15	22
TSP	14a	3b	15	14	14	16	14	17

<sup>1</sup>Statistical analysis is valid within each leaching event. Means followed by different letters within biosolids source and leaching event are significantly different using the LSD procedure ( $P \leq 0.05$ ).

effect of biosolids sources on the amount of P leached. Greater cumulative P release from OC South compared to OC East material is due to the digestion process. While OC East is aerobically digested, the OC South is anaerobically digested which may result in nearly complete release of biologically fixed P [28]. Results observed in this current study are consistent with previous studies [9]. These authors concluded that P release from biosolids depends on the treatment by which the material is being produced.

Compared to the initial P load (112  $\mu\text{g P g}^{-1}$  soil or  $\sim 224 \text{ kg P ha}^{-1}$ ), cumulative P mass leached represented <2% of the applied P. At 20°C, between 0.3% to 1.3% of total applied P was leached throughout the 90 d incubation (for JEA and OC South Cake biosolids, resp.). This suggested the great affinity of the Millhopper soil to retain added P (mainly because of the considerable concentrations of oxalate-extractable Fe and Al) and the negligible potential for P leaching in this soil even when highly soluble P sources such as TSP are applied. Perhaps greater P rates or repeated

application of biosolids would increase the risks associated with P leaching if the sorption sites became significantly occupied.

The majority (80% to 100%) of the P leached at both temperatures was SRP, with no differences in P form among the biosolids sources. Large SRP concentrations were observed in the first leaching event (15 d) for the OC South and East Cake treatments especially at 20°C (Figure 2(a)). This suggests the presence of readily available forms of P in these materials, preferentially released in the first leaching event. TSP followed the same pattern at the lower temperature; however, at 32°C, SRP concentrations increased after the second leaching event (Figure 2(b)). The other biosolids did not show a clear trend, and, despite some fluctuations, SRP concentrations were, in general, almost constant during the various leachings.

In general, cumulative P mass leached was significantly smaller when biosolids were incubated at greater temperature (32°C). For instance, cumulative P mass released by OC South

TABLE 4: Soil P distributions among the various fractions at the beginning ( $T_0$ ) and after 90 days of incubation (90 d).

Biosolids	$H_2O$		KCl		NaOHP <sub>i</sub>		NaOHP <sub>o</sub>		HCl		Residual	
	$T_0$	90 d <sup>2</sup>	$T_0$	90 d	$T_0$	90 d	$T_0$	90 d	$T_0$	90 d	$T_0$	90 d
Control	9.5 (0.9) <sup>3</sup>	8.1 (0.7)	2.7 (0.2)	3.7 (0.3)	760 (68)	726 (61)	67 (6.0)	95 (8.0)	68 (6.0)	43 (3.6)	210 (19)	311 (26)
ATAD	45 (3.8)	14 (1.1)	3.9 (0.3)	10 (0.9)	683 (57)	799 (67)	66 (5.5)	72 (6.0)	71 (6.0)	46 (3.8)	320 (27)	249 (21)
OC East Cake	57 (4.3)	15 (1.1)	8.9 (0.7)	10 (0.8)	779 (59)	773 (59)	78 (6.0)	71 (5.3)	79 (6.0)	48 (3.7)	315 (24)	400 (30)
JEA Cake	18 (1.6)	7.4 (0.7)	3.3 (0.3)	6.3 (0.5)	764 (67)	763 (67)	51 (4.5)	73 (6.4)	90 (7.9)	48 (4.2)	214 (19)	242 (21)
Tampa	12 (1.0)	9.6 (0.8)	4.5 (0.4)	7.3 (0.6)	783 (65)	775 (65)	74 (6.2)	454 (3.8)	89 (7.5)	53 (4.4)	234 (19)	307 (26)
WPB	21 (1.8)	13 (1.1)	5.5 (0.5)	6.5 (0.5)	775 (65)	794 (66)	70 (5.9)	78 (6.5)	79 (6.6)	56 (4.7)	243 (20)	246 (21)
OC South Cake	27 (2.2)	12 (1.0)	6.9 (0.6)	9.3 (0.8)	761 (62)	762 (62)	77 (6.3)	56 (4.5)	83 (6.8)	48 (3.9)	271 (22)	339 (28)
Disney	15 (1.2)	9.5 (0.8)	3.3 (0.3)	5.4 (0.4)	786 (63)	853 (69)	35 (2.8)	63 (5.1)	86 (6.9)	55 (4.4)	319 (26)	257 (21)
TSP	39 (3.3)	16 (1.4)	6.2 (0.5)	6.8 (0.6)	739 (62)	823 (74)	54 (4.5)	67 (6.0)	74 (6.2)	49 (4.4)	276 (23)	156 (14)

<sup>1</sup>  $H_2O$  and KCl represent water-soluble and exchangeable P fractions, respectively; NaOHP<sub>i</sub> and NaOHP<sub>o</sub> represent the Al- and Fe-bound inorganic and organic P; HCl is Ca-bound P; Residual is mineral-associated inorganic P.

<sup>2</sup> Values represent average P concentration of samples incubated at 20 °C and 32 °C.

<sup>3</sup> Data in parenthesis are % of total P.

Cake decreased by 57% when treated soils were incubated at 32°C compared with release at 20°C. Exceptions were JEA Cake and Tampa biosolids, which exhibited no significant effect of temperature on P release. TSP-treated soils exhibited similar patterns and leached much less P when incubated at the greater temperature. It was not clear if the temperature effects on P leachability were due to the greater soil affinity to retain P at 32°C compared to 20°C or if biosolids-borne P availability was also affected by increased temperature. A number of studies document the impacts of temperature on soil P sorption reactions [29–32]. In general, high temperatures increase the rate of P transfer to strongly bond forms and thus decrease P concentration in solution. Therefore, the reductions in P leaching concentrations at 32°C observed in this current study can be attributed to the greater soil affinity to retain P in stable forms at a higher temperature.

**3.2. Changes in P Extractability and Distribution over Time.** Results from the static incubation (samples not subjected to leaching) revealed that, at both temperatures, WEP concentrations were significantly reduced over time (Figure 3). The decrease in P extractability was similar when samples were incubated at 20°C (Figure 3(a)) or 32°C (Figure 3(b)), suggesting that there was no significant effect of temperature on WEP concentrations. The reactions between biosolids-borne P with the soils matrix were favored with time, and thus, P extractability was considerably reduced. Similar results were also reported by [28, 32] who observed that P becomes less bioavailable with time due to P diffusion into soil sorption sites.

Overall P distribution among the various fractions was the same after 90 d of incubation (static incubation) (Table 4). NaOH-P (Al- and Fe-bound P) was the dominant fraction controlling P retention in the biosolids-treated soils, in agreement with other numerous studies [33–35]. The NaOH fraction is widely assumed to represent P associated with poor crystalline Al and Fe oxides and is the most important fraction controlling the P reactions in noncalcareous biosolids-treated soils.

**3.3. Biosolids Dissolved Organic C Release.** Large differences in DOC concentrations at 20°C and 32°C were observed in samples from the first leaching event (Table 3). The more easily degradable C fraction was likely leached in the first leaching event, the less available compounds remained in the treated soils. In the first leaching event, DOC release was, on average, ~3-fold greater at 20°C than at 32°C. Because complex processes control DOC release from biosolids-amended soils, it is difficult to distinguish whether greater mineralization of C compounds to CO<sub>2</sub> occurred at 32°C resulting in less DOC released or a larger proportion of complex, water-insoluble C was converted into labile, water-soluble C forms at 20°C. Numerous studies suggested that soil microbial activity is promoted at the greater temperature; thus, it is possible that microbes at 32°C were more efficient in complete utilization C from the various biosolids as an energy source. Reference [36] also observed greater biosolids mineralization rates when the material was applied in the

summer (higher temperature and rainfall) as compared to spring application. Leachate pH and EC data further support the hypothesis that greater biosolids mineralization rates occurred at 32°C than those at 20°C (data not shown). In subsequent leaching events, however, there was no clear evidence of temperature effect on DOC release. Some biosolids increased DOC release at 32°C (OC East Cake, JEA Cake, and Tampa), while others (ATAD, OC South Cake, Disney) did not show a clear pattern.

Dissolved C released clearly did not follow the same pattern as P leaching. This occurred because the mechanisms by which these elements are retained and mineralized in biosolids-treated soils are not related. Although the majority of the P present in the biosolids is inorganic P, our hypothesis was that P release increases as mineralization of biosolids occurs. However, our results suggested that the interaction between biosolids degradation and P availability is much more complex. For instance, OC East and South biosolids Cakes showed a decline in the amounts of P leached as a function of leaching event for both temperatures (Figure 2), but DOC concentrations in the leachates followed a different pattern (Table 3). Perhaps, the great affinity of the Millhopper soil for P played a major role by masking any relationship between DOC release and P leaching.

## 4. Conclusions

Negligible amounts (<2%) of the total P applied as either biosolids or TSP were leached from the Millhopper soil during the 90 d study (total of 8 pore volumes of drainage). The appreciable amounts of oxalate-extractable Al and Fe oxides in the soil apparently retained most of the applied P and prevented leaching. The relatively great ability of the soil to sorb P masked differences in P solubility among the various sources and the potential impacts of temperature on biosolids-P availability. Additional work using other soils is warranted because this study focused on a Spodosol with relatively high P-sorbing capacity. Studies on other sandy soils in Florida reveal much greater P mobility [35].

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