



Bioavailability, mobility and leaching of phosphorus in a Mediterranean agricultural soil (ne Spain) amended with different doses of biosolids

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Abstract The precipitation of sparingly soluble calcium phosphate in calcareous soils decreases the bioavailability of macronutrients, which makes their addition by way of fertilisers necessary. Sludge resulting from treating urban wastewater does not only provide significant amounts of phosphorus, but also helps lower the pH, thus increasing its bioavailability. The loss of part of soil nutrients due to irrigation or rain can contaminate groundwater. In order to assess the movement of phosphorus, an experiment was conducted on percolation columns, to which different doses of wastes were applied. The pH decreased by as much as 0.89 units, as well as the assimilable and soluble P, in intervals of 20 cm of depth, obtaining maximum values of 254 mg P kg⁻¹ and 1455 µg P kg⁻¹ respectively, and the P present in the leached water collected, which did not surpass 95 µg PL⁻¹. The intent was to learn which was the

majoritarian inorganic formed crystalline phase that immobilised the movement of phosphorus through the percolation column. The results obtained by the diffraction of X-rays are not conclusive, although they point to the formation of octacalcium phosphate. The diffractograms of the studied samples have similar diffraction lines to those of apatites.

Keywords pH · Sewage sludge · Mediterranean soil · Phosphorus · P mobility · Leachates

Introduction

In recent years, the need to treat wastewater has entailed the start-up of a large number of treatment plants. This has led to a great increase in the production of sewage sludge, and to a search for uses for this by-product is its use as an amendment for agricultural soils, due to the fact that its addition makes it possible to improve their physical, chemical and microbiological properties (Neumann et al. 2017). However, its addition to soil is not devoid of environmental risks or drawbacks resulting from the possible presence of organic compounds that are hardly degradable, hazardous metals (Núñez et al. 2002), microorganisms, an excess of nutrients and the mobility of ions or potentially contaminating substances (Galán et al. 2008; Bech et al. 2008; Jordán

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et al. 2017). In this sense, the use of sewage sludge as fertiliser could cause issues due to the contamination of waters with nitrates and phosphates, as the surface runoff can contaminate surface water or even carry said ions, passing through deep geological strata until reaching aquifers (Almendro et al. 2001, 2003). The sludge applied to soils, if not handled properly, can cause the eutrophication of lakes and surface runoff water currents, soil erosion and the contamination of the subsoil with water nitrates from the leaching of N released by the sludge. Eutrophication can take place in soils where organic waste is usually applied as fertiliser. The surface runoff can thus contaminate surface waters or even carry the nitrates and phosphates, passing through deep geological strata (Jordán et al. 2017).

The phosphate can be retained in the soil by adsorption and precipitation, with the magnitude of this process depending on several factors, including the pH of the solution, anionic competitiveness and the presence of hydrated oxide and iron, and aluminium hydroxides such as goethite (α -FeOOH) and gibbsite (γ -Al(OH)₃), respectively (Jordán et al. 2009). In the case of acidic soils governed by clays with permanent charges (montmorillonite, vermiculite, illite, etc.), several authors have confirmed the formation of iron and aluminium phosphates. In these soils, the decrease in pH following the appliance of sewage sludge makes it possible to break down the structure of the clay minerals and, as a result, Al and Fe are released, which react with the phosphate, creating fairly insoluble compounds. The clay surfaces of these soils are not reactive and have modest amounts of P, making the reactions with Fe and Al the ones which immobilise P. On the other hand, the tropical soils governed by clay minerals with variable charges (kaolinite, Fe and Al oxides, allophane, imogolite, etc.) are stable even at low pH values, and only when the pH of the soil reaches values lower than 5 do Fe and Al release themselves into the solution of the soil and can react with the phosphate. In this case, the immobilisation of P is linked to the high reactivity or affinity for P of the surfaces of the clays that are present in the soil. This process retains significant amounts of P in a wide range of pH values. Less soluble calcium phosphates are formed in the case of alkaline soils. Over time, several of these final forms of phosphate can also dissolve and seep into the solution of the soil, from where they can be absorbed by plants or take part in

reactions such as the aforementioned (Yan et al. 2018). In the literature consulted, there is limited information on the mobility of this element in soils with pH > 8 and high contents of calcium carbonate. However, relevant works have been found regarding phosphorus adsorption–desorption kinetics (Akhtar et al. 2002; Abdala et al. 2015). Furthermore, similar experiments have been developed to assess the potential mobility of heavy metals in soils that had sludge applied to their surface (Navarro-Pedreno et al. 2003) and to study the ability to retain nutrients from wastewater (Almendro et al. 2003), among others.

The main goal of this paper is to verify, through a controlled study in a greenhouse, the possibility of movement of P through the unsaturated zone, due to the resulting risk of aquifer contamination. The results of this study will make it possible to define protocols for the use of sewage sludge in Mediterranean crops high in carbonates and with a pH > 8, both from dry farming and irrigated land, because, until now, only the behaviour of legislated heavy metals and the mobility of nitrogenated compounds have been taken into account by the region's managers and relevant authorities.

Materials and methods

The experiment was conducted under controlled environmental conditions inside a greenhouse (a temperature of 25 °C and 50% of relative humidity). The experimentation was based on a study using percolation columns (OECD 2004). To do so, a pipe with a diameter of 10.5 cm was cut into 20 cm-long pieces, creating 40 columns measuring 80 cm in height. Each column consisted of four differentiated sections of 20 cm each: 0–20 cm, 20–40 cm, 40–60 cm and 60–80 cm, similar to those used by Almendro et al. (2001, 2003), who used them to study the movement of nitrogenous substances through the profile of the soil. These sections allowed us to conduct a differentiated study of the mobility of P through the soil (Lewis and Sjöström 2010). A receptacle with a conveniently adapted lid was placed at the end of the columns in order to collect the water that had permeated the soil (leachates).

Three treatments were conducted by applying different amounts of sludge (T₄₀: 40,000 kg ha⁻¹, T₈₀: 80,000 kg ha⁻¹ and T₁₂₀: 120,000 kg ha⁻¹), as

Table 1 Sewage sludge composition (dry matter)

Parameter	Unit	Value	C.I	Parameter	Unit	Value	C.I
Humidity	%	83	3	Oxidsable C	%	20.2	3.0
Org M. _{500 °C}	%	61.2	4.9	Org. M. oxid	%	34,1	5.0
Al	g/kg	12,23	1.15	Mg	g/kg	5.69	0,30
As	mg/kg	1.0	0.5	Mn	mg/kg	153	10
Ba	mg/kg	539	25	N	g/kg	42.15	1.07
Ca	g/kg	58.02	3.09	Na	g/kg	9.75	1.30
Cd	mg/kg	39.1	0.4	Ni	mg/kg	292	55
Cr	mg/kg	29	5	P	mg/kg	2375	256
Cu	mg/kg	415	52	Pb	mg/kg	89	6
Fe	g/kg	42.35	1.55	Sr	mg/kg	453	36
Hg	µg/kg	4	0.2	Ti	mg/kg	20	7
K	g/kg	1.47	0.34	Zn	mg/kg	2163	12

well as a blank test or control treatment (T_0). The sludge used in this experiment, whose properties are in Table 1, comes from the wastewater treatment plant of a ceramic industrial area of the province of Castellón, near the town of L’Alcora, in the district of L’Alcalatén. The residue was applied to the surface and mixed with the soil, simulating the action of ploughing, creating a homogeneous mixture of the sludge with the first 20 cm of soil. This mixture was placed on the upper part of each percolation column, over the bottom 40 cm of untreated soil, preparing 10 columns for each treatment.

The soil used to carry out this experiment, whose properties can be seen in Table 2, corresponds to agricultural soil located in an experimental field dedicated to the cultivation of almond trees and seasonal vegetables in the north of Castellón province (Fig. 1). This soil has been structurally altered by the action of the machinery in charge of adapting the terrain. Therefore, there is no clear distinction between horizons, and it has similar physical–chemical properties throughout its profile. This makes it a young xeric soil that has been strongly modified by anthropic action, meaning it could thus be classified as an anthrosol (Staff 2010).

In order to create the greatest parallelism possible between the real conditions of an irrigated Mediterranean crop and the experiment, the soils included in the columns were subjected to a weekly supply of water (80 mm). The supplying of water was conducted with a device that simulates a flood irrigation system, which covers the surface and then percolates through the soil. A total 12 irrigations were conducted during

the experiment, which equates to the total supply of irrigation water during the vegetable growing season.

Four soil and leached water samplings were conducted every 60 days. The pH and assimilable and soluble P were analysed in each one. The pH was measured in soil suspended in deionised water, at a proportion of 1:2.5 (m/V). The Burriel-Hernando method (Almedro et al. 2003) was used to analyse the assimilable phosphorus in the soil, due to its high sensitivity for calcareous soils with an alkaline pH (extracting solution comprised of CaCO_3 , MgCO_3 , H_2SO_4 and acetic acid, pH between 3.2 and 3.3). The soluble phosphorus was measured with an extraction with deionised water (1:5, m/V). Once the extraction was conducted, the amount of phosphorus was measured by forming a phosphomolybdic compound that is reduced in the presence of ascorbic acid, in an acidic medium, and then measuring it with colorimetry at 825 nm (Almendro et al. 2003). Leached waters were analysed with the same method.

The ANOVA test was used to assess the statistical significance of the results, analysing both the effect of the treatments and the depth for the analysed parameters (*, **, *** indicate significance at levels of $p = 0.05$, 0.01 and 0.001 respectively; ns: not significant). The confidence interval (C.I.) is also shown at $p = 0.05$ to justify the difference among treatments.

X-ray diffraction was used to identify the formed crystalline phases of phosphorus. For the XRD analysis, step-scanned data were collected between 2θ angles of 4° and 60° at a rate of $1.7^\circ/\text{min}$ using a SIEMENS D5000 diffractometer. This device was operated at 30 kV and 20 mA using $\text{CuK}\alpha$ radiation

Table 2 Soil physical and chemical properties

Parameter		Value	Parameter		Value
<i>Texture</i>			<i>Mineralogy</i>		
Sand	%	26	Soil	Quartz	Calcite
Silt	%	35	Clay fraction	Quartz/Illite	Kaol/Chl
Clay	%	39	<i>Total elements</i>		
pH		8.70	Al	g/kg	18.55
E.C	μS/cm	80.5	B	mg/kg	50.0
CaCO ₃	%	71	Be	μg/kg	49.8
C. oxidisable	g/kg	1.0	Ca	g/kg	235.7
Org M. oxid	g/kg	3.6	Cd	μg/kg	388
N Kjeldahl	g/kg	0.4	Cr	mg/kg	21.6
P	mg/kg	15.72	Fe	g/kg	10.097
<i>Extracted ammonium acetate</i>			K	g/kg	3.178
Ca	g/kg	5.230	Li	mg/kg	10.1
K	g/kg	0.167	Mg	g/kg	4.452
Mg	g/kg	0.255	Mn	mg/kg	167.2
Na	g/kg	0.080	Mo	mg/kg	1.8
<i>Extracted DTPA</i>			Na	g/kg	0.405
Cu	mg/kg	0.39	Ni	mg/kg	18.5
Fe	mg/kg	0.52	P	mg/kg	96.5
Mn	mg/kg	1.05	Sr	mg/kg	400.4
Zn	mg/kg	0.21	Zn	mg/kg	37.3

and graphite monochromator. Some samples were treated with HOAc-NaOAc buffer (pH 5.0) to remove carbonates and with H₂O₂ to remove organic matter. The calcite and dolomite carbonates have diffraction lines that overlap with apatite. Furthermore, the clay fraction was separated by sedimentation (Gee and Bauder 1986).

Results and discussion

A decrease in pH is observed with an increased appliance of sludge (Table 3). This decrease is more noticeable where the sludge was applied, in the first 20 cm of soil, where the pH of the control treatment (8.70) decreases by as much as 0.89 units in the treatment with the highest amount of sludge (7.81). Treated soils tend to gradually increase their pH, due to the effect of the sludge decreasing over time.

A significant increase in assimilable phosphorus was tested in the surface horizon in the presence of sludge (Table 4), increasing from 16.08 mg/kg⁻¹ d.m. in the control up to 153.22 mg/kg⁻¹ d.m. in the treatment with the most amount of sludge applied. In

the control treatment, no variation was observed with depth, whereas in the treatments fertilised with waste, there is a decrease in the amount of assimilable phosphorus. On the surface level of the treated columns, there seems to be an increase over time until the third sampling, with a decrease in the fourth.

The bioavailability of P in calcareous soils is usually limited by the precipitation of secondary calcium phosphates with low solubility. There is not a clear trend of the variation of soluble P with depth, and over time, a certain decrease to its extractability was observed in horizons 20–40 cm and 40–60 cm with the treatment (Table 5). In the surface horizon of the treatment with the highest dose (T₁₂₀), there was a considerable increase in the three last samplings compared to the other treatments. This fact indicates the presence of soluble P compounds, either due to the presence of labile Fe or Al phosphates, or more soluble calcium compounds such as monocalcium phosphate.

In no case were the obtained values very high. In the leachates, a certain tendency was observed for the phosphorus to increase with the dose of sludge applied, although the differences among treatments are only significant in samplings 1 and 3. The

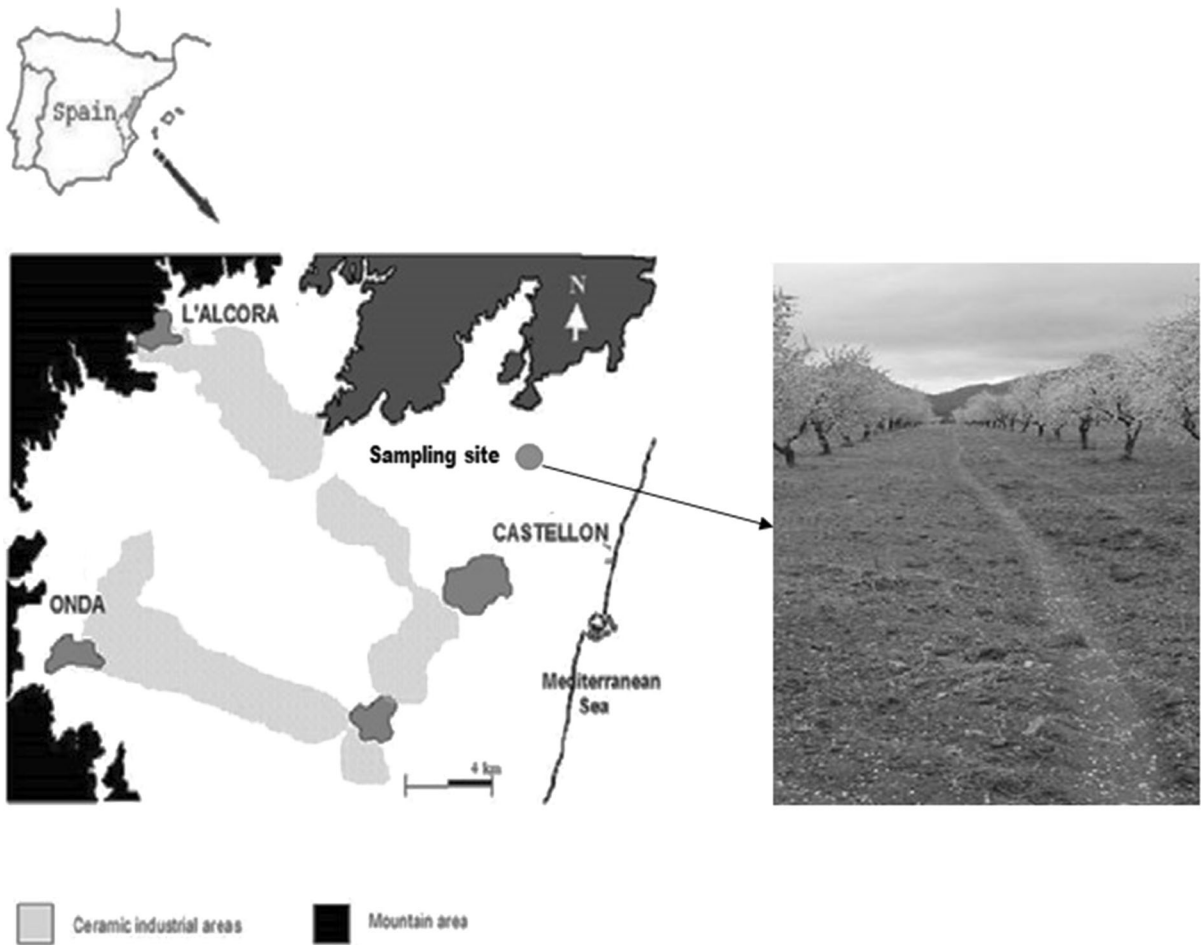


Fig. 1 Geographic location of the experimental land where the soil was sampled. Photograph that shows the soil for the growing of almond trees and seasonal vegetables

concentration of phosphorus in leachates tends to decrease over time in soils amended with sludge (Table 6). The carbonated nature of this soil has prevented the loss of phosphorus.

The mineralogical composition of the soil surface (1–20 cm) for each treatment is shown in Table 7. Regarding the availability of P driven by the addition of sludge to the soil, the values obtained are relevant because in calcareous soils, the precipitation of P can take place as calcium phosphate (from monocalcium phosphates to hydroxyapatite), decreasing its possible absorption by plants. The intent was to learn which was the majoritarian inorganic formed crystalline phase that immobilised the movement of phosphorus through the percolation columns. The main inorganic forms of phosphates in the soil are well known: dehydrated dicalcium phosphate ($\text{CaHPO}_4 \cdot 2\text{H}_2\text{O}$),

dicalcium phosphate (CaHPO_4), octacalcium phosphate ($\text{Ca}_8\text{H}(\text{PO}_4)_3 \cdot 2.5\text{H}_2\text{O}$), β -tricalcium phosphate ($\text{Ca}_3(\text{PO}_4)_2$) and hydroxyapatite ($\text{Ca}_5(\text{PO}_4)_3\text{OH}$). The results obtained with X-ray diffraction are not conclusive, although they point to the formation of octacalcium phosphate. The spectroscopic identification of octacalcium phosphate is unclear, because the diffractograms of the studied samples have similar lines to those of apatites (Fig. 2). Apatite is an almost insoluble calcium and phosphorus compound. In the soils of arid regions, where the calcium content remains high and the pH is alkaline, the apatite lingers, representing the main phosphor mineral. Posner (1955) considers octacalcium phosphates to be defective apatites in which, for each mole of PO_4 , one mole of Ca has been replaced by two moles of H. This transformation takes place by way of a diffusion

Table 3 pH for each depth and treatment

Sampling		Treatment								ANOVA
Days	Depth	T ₀		T ₄₀		T ₈₀		T ₁₂₀		F
		Mean	C.I	Mean	C.I	Mean	C.I	Mean	C.I	
60	20	8.70	0.10	8.30	0.14	8.12	0.05	7.81	0.11	***
	40	8.68	0.20	8.49	0.05	8.29	0.06	8.30	0.15	***
	60	8.67	0.12	8.45	0.11	8.40	0.02	8.43	0.04	***
	80	8.60	0.09	8.40	0.07	8.36	0.03	8.46	0.13	**
F		ns		**		***		***		
120	20	8.81	0.05	8.42	0.04	8.22	0.11	8.12	0.14	***
	40	8.76	0.21	8.55	0.01	8.50	0.10	8.40	0.07	***
	60	8.68	0.20	8.64	0.12	8.56	0.07	8.49	0.06	**
	80	8.62	0.10	8.60	0.03	8.51	0.13	8.41	0.09	**
F		9.13**		19.46***		30.72***		41.37***		
180	20	8.87	0.01	8.54	0.06	8.40	0.15	8.10	0.09	***
	40	8.90	0.07	8.70	0.10	8.52	0.03	8.45	0.11	***
	60	8.85	0.25	8.60	0.10	8.55	0.05	8.49	0.05	***
	80	8.84	0.18	8.61	0.01	8.52	0.06	8.51	0.03	***
F		ns		*		*		***		

Table 4 Assimilable P content (Burriel-Hernando method) (mg/kg m.s.) for each horizon and treatment

Sampling		Treatment								ANOVA
Days	Depth	T ₀		T ₄₀		T ₈₀		T ₁₂₀		F
		Mean	C.I	Mean	C.I	Mean	C.I	Mean	C.I	
60	20	16.08	5.03	55.51	13.29	93.00	50.18	153.22	47.09	***
	40	20.87	6.81	24.72	5.83	26.71	10.89	25.72	10.05	ns
	60	15.42	3.48	15.49	3.98	14.09	8.74	15.42	0.74	ns
	80	15.42	1.39	17.92	8.01	1.91	0.81	11.90	4.07	*
F		*		***		***		***		
120	20	14.29	7.26	76.56	5.12	128.02	15.66	198.48	24.35	***
	40	16.45	2.15	19.31	12.34	25.31	6.79	21.51	15.88	ns
	60	17.93	4.37	15.69	4.02	15.47	7.26	13.82	6.28	ns
	80	14.22	8.67	15.37	1.36	12.99	5.34	14.48	3.77	ns
F		ns		***		***		***		
180	20	4.55	1.66	51.80	15.37	76.59	22.34	166.65	45.94	***
	40	4.20	2.94	14.82	6.19	31.99	22.29	37.70	24.40	**
	60	5.61	2.43	4.09	3.00	4.41	0.74	4.89	2.33	ns
	80	4.55	5.24	3.67	2.98	6.07	2.12	4.16	2.21	ns
F		ns		***		***		***		

in the solid phase and not by crystallisations. Hence, the difficulty in preparing pure octacalcium phosphates and the reason why phosphates that are composed of octacalcium phosphates and/or hydroxypapatites are so common.

Conclusions

The addition of biosolid to this calcareous soil has modified the pH of all the horizons, and very significantly of the surface horizon where the waste was applied. From an agrochemical point of view, the

Table 5 Soluble P content ($\mu\text{g}/\text{kg}$ m.s.) for each depth and treatment

Sampling		Treatment								ANOVA
Days	Depth	T ₀		T ₄₀		T ₈₀		T ₁₂₀		F
		Mean	C.I	Mean	C.I	Mean	C.I	Mean	C.I	
60	20	280	93	471	922	142	81	261	365	ns
	40	401	355	305	310	107	191	62	86	*
	60	421	526	186	60	222	231	2001	201	ns
	80	430	251	168	39	151	159	152	64	**
F		ns		ns		ns		ns		
120	20	635	202	562	319	428	132	1455	271	***
	40	701	391	685	576	626	701	396	502	ns
	60	371	503	699	428	511	519	601	402	ns
	80	665	426	761	522	522	801	300	143	ns
F		ns		ns		ns		***		
180	20	722	509	199	276	369	679	1306	553	**
	40	566	305	708	329	338	569	230	488	*
	60	378	211	415	738	422	506	462	1078	ns
	80	569	411	759	302	460	538	420	367	ns
F		ns		*		ns		**		

Table 6 P content ($\mu\text{g}/\text{L}$) in leachates

Treatment	Sampling							
	60 days		120 days		180 days		240 days	
	Mean	C.I	Mean	C.I	Mean	C.I	Mean	C.I
T ₀	72	13	75	31	65	12	71	48
T ₄₀	85	21	90	22	81	16	76	33
T ₈₀	66	31	96	46	73	13	78	13
T ₁₂₀	95	40	88	21	79	18	80	15
F	*		ns		*		ns	

Table 7 Mineralogical composition of the soil surface (1–20 cm) for each treatment

	High	Moderate	Little	Traces		
T ₀	Cal	Qtz	Il-M	F, Do	Ch	Ka
T ₄₀	Cal	Qtz	Il-M	F, Do	Ch	Ka
T ₈₀	Cal	Qtz	Il-M	F, H	Ch	Ka, Ap
T ₁₂₀	Cal	Qtz	Do, Il-M, H	F, H	Ch	Ka, Ap

Qtz quartz; Cal calcite; F feldspar; Ka kaolinite; Ch chlorite; Do dolomite;

Il-M illite-muscovite; Goe goethite; H hematite; Ap apatite

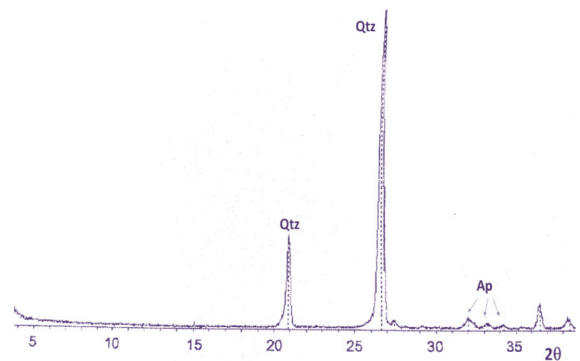


Fig. 2 XRD diffraction pattern of soil sample treated with HOAc-NaOAc and H₂O₂ showing peaks for apatite (Ap) and quartz (Qtz)

appliance of sewage sludge favoured the bioavailability of phosphorus. The appliance of sludge in the tested amounts does not entail the risk of groundwater contamination with phosphorus, as the mobility of phosphorus in this type of soil is very limited. Without doubt, the carbonated nature of this soil has greatly prevented the movement and loss of phosphorus from the soil (precipitation of calcium phosphates). The formation of Ca-P mineral phases depends on the concentration of Ca^{2+} ions, which are very abundant in the studied soil, as well as on the pH. The P from sewage sludge added to the first 20 cm of soil was rapidly insolubilised. These results suggest that calcareous soils are able to form insoluble P phases due to their high reservoir of exchangeable cations.

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