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SOLID OLIVE MILL WASTE AS AN AMENDMENT FOR CALCAREOUS SOIL AND ITS EFFECT ON PHOSPHORUS FORMS AND SOME SOIL CHEMICAL PROPERTIES

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ABSTRACT

Experiments were conducted to study the effect of addition solid olive mill waste (SOMW) to calcareous soil on phosphorus (P) forms and some chemical properties as well as dry matter and P uptake by wheat plants (*Triticum aestivum* L.) grown on soil from Wadi El-Assiuti area, Assiut Governorate, Egypt. Rates of SOMW are zero, 10, 20 and 30gkg^{-1} were mixed with 200g of soil samples and moistened to the field capacity, and then incubated at 30±2ºC for zero, 30, 60 and 90 days. The results indicated that the soil P forms of soluble-P, $NaHCO_3$ -P, NaOH -P, and HCl-P increased with increasing the rate of (SOMW) application, also increases up to 60 days of incubation, then decreased at 90 days. Residual-P form decreased with increasing the rate application, as well as the increase in the time of incubation. The highest of all P forms (except the residual-P one) were obtained at the highest application level. For the soil chemical properties, soil salinity of the studied soil significantly increased. On contrast, the soil pH and calcium carbonate ($CaCO_3$) decreased with increasing both of SOMW level and the time of incubation. Meanwhile, soil organic matter increased with increasing the level of SOMW, and decreased with increasing the incubation time. Dry mater yield and P uptake by wheat plants grown on the soil decreased with increasing the addition of the waste material.

Key words: Calcareous soils, solid olive mill wastes, incubation, P forms.

INTRODUCTION

The olive oil industry is one of the important sectors for agricultural economy especially in the Mediterranean countries. Thus there are a need for research to consider possible ways to clean and recycle the produced olive oil mill wastes (Benítez *et al*., 2000). Annually, about 11 million megagram (Mg) of olives are produced and about 1.7 million (Mg) of olive oil is produced representing 95% of the world production (IOOC, 2008). Olive mill waste is rich in organic matter, nitrogen, phosphorus, potassium and magnesium (Mekki *et al*, 2006). Several advantages were reported form the use of olive mill wastes such as for plant nutrients and as a soil amendment improve soil properties (Altieri and Esposito, 2008). Killi and Kavdır, (2013) reported that agricultural industrial wastes from olive mill may play an important role as organic fertilizers, enhancing crop production and improving soil property. Soils in semi-arid and arid areas have low organic matter content. In calcareous and alkaline soils the phosphorus deficiency is one of the major limiting factors of this fertility due to their low P availability caused by P fixation by calcium (López-Piñeiro and Gercia Navarro, 1997). Organic amendments increased recovery of applied P because of a decrease in precipitation of soluble P. Application of fresh olive mill waste for 5 years, was reported by López-Piñeiro *et al*. (2009) to increase all P fractions and decrease of the P desorption. In other studies, López-Piñeiro *et al.* (2011) reported that application of 27 and 54 mg ha^{-1} of olive mill waste for 7 years increased P forms in soil.

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The current study aims at investigating the effect of solid olive mill wastes on soil P forms and some chemical properties o a calcareous soil as well as the dry mater yield and P uptake by wheat plants grown on the soil.

MATERIALS AND METHODS

Soil Sampling

Calcareous soil was collected from the surface layer (0-20 cm) from region of Wadi El-Assiuti, Assiut Governorate, Egypt to study the effect of olive mill wastes on soil P forms and chemical properties as well as the growth and P uptake by wheat plants grown on that soil. The soil sample was air-dried, ground and sieved through a 2-mm sieve and kept for the present study. Physical and chemical properties of the studied soil sample are shown in Table 1.

Solid Olive Mill Waste

Solid olive mill wastes (SOMW) collected from the olive oil factory located in the Farafra Oasis, New Valley, Egypt, was washed with water, dried, sieved through 2 mm. Physical and chemical properties of SOMW are shown in Table 2.

The Incubation Experiment

An incubation experiment was conducted to study the effect of solid olive mill waste on some soil properties. Solid olive mill waste was added at rates of zero, 10, 20, and 30 gkg^{-1} and mixed with 200 g soil in plastic pots. The soil samples treated with solid olive mill waste levels were incubated at 30±2ºC for zero, 30, 60 and 90 days. The moisture content of the soil samples in the pots were daily adjusted to field capacity by distilled water. Soil samples were taken at the end of each incubation period for soil chemical analysis and soil P fractionations.

The Greenhouse Experiment

A greenhouse experiment was carried out to evaluate the effect of adding solid olive mill waste on the growth, dry mater yield and P uptake of wheat plants (*Triticum aestivum* L.). Plastic pots containing 3kg of soil sample in each pot were used in this experiment. Rates of solid olive mill waste were zero, 10, 20 and $30gkg^{-1}$ mixed with the soil in the pots before experiment beginning. After one month, three seeds of wheat were sown in each pot. Watering was done daily to keep the soil at 90% of the soil retention capacity. The design of both experiments was a randomized complete blocks with three replicates.

The plants in each pot were thinned to two plants after 15 days of sowing. The recommended amounts of N and K were added to each pot by fertilization. The harvested plants at 60 days age, were washed by distilled water, oven dried at $70^{O}C$ for 72 hours, weighed, ground and kept for chemical analysis.

Soil Analysis

Particles-size distribution, electric conductivity, soil pH, and organic matter content were determined using methods reported by Jackson (1973). Total calcium carbonates were determined by the calcimeter method (Nelson, 1982).

Solid Olive Mill Waste Analysis

The principal properties of the SOMW (Table 2) were determined by standard methods (Rodier, 1984). Total N, P and K were determined according to method described by (Page *et al*., 1982). Measurement of of Fe, Mn, Zn and Cu in extracts was done by atomic absorption spectrophotometer (Shuman, 1979).

Sequential Phosphorus Extraction

A sequential phosphorus fractionation method reported by Hedley *et al*. (1982) was used to study the chemical forms of P in the soil. Soluble-P was extracted by shaking 1.5 g soil sample with 30 ml distilled water into 50 ml centrifuge tubes. The soil sample remaining after the previous step was shaken with 30 ml of 0.5 M $NaHCO_3$ at 8.5 pH for 16 hr. After the extraction with $NaHCO_3$, the soil sample remaining was shaken with 30 ml of 0.1M NaOH for 16 hr. The soil sample from the previous step was shaken with 30 ml of 1M HCl for 16 hr. Organic and inorganic P forms were determined in both $NaHCO_3$ and NaOH extracts after the addition 10 ml of H_2SO_4 and 0.5 g of $K_2S_2O_8$ and then the digestion for 1 hr., at $127^{0}C$ (Zheng *et al*., 2002). The organic P was calculated as the difference between the total P from the digestion and inorganic P. Phosphorus concentration in extracts was determined by spectrophotometer method.

Table 1. Physical and chemical properties of the soil used in the study

Property	Value
Particle size distribution	
Sand (%)	88.60
Silt (%)	6.10
Clay (%)	5.30
Texture	Sand
Saturation percent	21.50
pH (1:2.5)	8.34
EC (1:2.5, dS/m)	0.76
Organic matter (g kg^{-1})	3.30
$CaCO_3$ (g kg^{-1})	165.00
Soluble cations and anions (mmolkg^{-1})	
Na^+	5.57
K^+	1.51
Ca^{2+}	8.56
Mg^{2+}	1.03
HCO_3^-	0.51
Cl^-	2.20
$SO_4^=$	13.96
P forms (mgkg^{-1})	
Water soluble-P	19.34
$NaHCO_3$-P_i	45.04
$NaHCO_3$-P_o	23.22
NaOH-P_i	53.05
NaOH-P_o	31.05
HCl-P	145.18
Residual-P	680.70
Available P (mgkg^{-1})	34.57
Total P (mgkg^{-1})	997.58

Table 2. Principal properties of solid olive mill waste

Property	Value
pH (1:2.5)	6.2
EC (1:2.5, dS m^{-1})	5.7
Organic matter (g kg^{-1})	520
N (g kg^{-1})	9.0
P (g kg^{-1})	4.5
K (g kg^{-1})	1.6
Ca (g kg^{-1})	3.8
Mg (g kg^{-1})	0.7
Na (g kg^{-1})	0.3
Fe (mgkg^{-1})	1118
Mn (mgkg^{-1})	54.22
Cu (mgkg^{-1})	11.31
Zn (mgkg^{-1})	22.43

Plant Analysis

Plant samples were digested using concentrated H_2SO_4 and $HCLO_4$ (Chapaman and Pratt, 1961) and analyzed for P concentration in the digests measurement by the spectrophotometer.

RESULTS AND DISCUSSION

Soil Chemical Properties

Soil chemical properties, including Electric conductivity (EC), pH, organic matter (OM), and calcium carbonate ($CaCO_3$) of the studied soil surface layer samples are shown in Table 3.

Soil Salinity

The results in Table 3 indicate that soil salinity as determined in terms of electric conductivity (EC) of soil extract was significantly affected by SOMW addition depending upon the addition level and the time of incubation. The EC increased with increasing the incubation time and the application level of SOMW up to 20g kg $^{-1}$ after which these was a decrease. The increase may be due to releasing soluble salts from SOMW to the soil and particularly as the time of incubation increased (Killi and Kavdır, 2013). The two highest values of EC were recorded at the levels of 20 and 30 g kg^{-1} after 90 days of incubation. The increase in the soil treated with solid olive mill waste indicates the high contents of soluble salts in the material as shown in Table 2. Mekki *et al*. (2006) reported marked contents of soluble salts in solid olive mill waste Achak *et al*. (2009) reported that application of solid olive mill waste to soils increased salts in the soil.

Soil pH

The examined application rates of solid olive mill waste and various incubation periods showed significant effects on the soil pH (Table 3). pH decreased by application of SOMW. This is in agreement with those obtained by Remzi *et al*. (2013) who found that the soil pH decreased with applying the olive oil solid at rates of 79 g kg^{-1} to a clay loam soil. The pH decreased with time. The decrease with increased rates may be due to some acids components in the waste, and the decreased with time may be due to formation of acidic substances during decomposition of such organic material (Achak *et al*., 2009).

Table 3. Effect of adding solid olive mill waste and incubation time on the electric conductivity, pH, organic matter and $CaCO_3$ in the calcareous soil

Character	Rate of SOMW (g kg^{-1})	Incubation time (day)			
		Zero time	30	60	90
EC (1:2.5) dS/m	0	0.79	0.84	0.90	0.92
	10	1.07	1.07	1.09	1.11
	20	1.38	1.43	1.46	1.48
	30	1.29	1.35	1.44	1.47
	$LSD_{0.05}$	0.04	0.06	0.05	0.05
pH (1:2.5)	0	8.28	8.14	8.08	8.01
	10	8.14	8.01	7.92	7.81
	20	7.91	7.74	7.69	7.60
	30	7.90	7.62	7.41	7.19
	$LSD_{0.05}$	0.05	0.07	0.06	0.06
OM (g kg^{-1})	0	2.9	3.6	3.3	3.1
	10	9.3	14.1	13.2	12.6
	20	11.1	14.8	14.1	13.2
	30	11.9	15.3	14.6	13.8
	$LSD_{0.05}$	0.4	0.3	0.3	0.2
$CaCO_3$ (g kg^{-1})	0	184.2	181.4	180.1	179.8
	10	183.1	180.0	178.9	178.2
	20	181.4	178.6	177.8	177.0
	30	181.0	177.9	176.8	176.2
	$LSD_{0.05}$	0.8	0.7	0. 6	0. 6

In most cases, the lowest value (7.19) of the soil pH was observed with the highest level waste addition after 90 days of incubation. These results are in a agreement with those obtained by Kutuk *et al*. (2002) who found that soil pH decreased due to soil incubation with liquid humic acid at different incubation periods up to 90 days. Mekki *et al*. (2009) reported slight variation in soil pH upon adding olive mill waste. Di Serio *et al*. (2008) found that application 160 m^3 ha^{-1} $year^{-1}$ of olive mill waste liquid slightly increased soil pH and attributed this evolution of NH_3 during decomposition of the added material.

Organic Matter

The organic matter content of the soil increased with increasing application of SOMW as well as it increased with time of incubation up to 30 days, then the decrease occurred with prolonged incubation time (Table 3). The highest value of organic matter content was recorded with highest level of application after 30 days of incubation. These results agree with those obtained by Nektarios *et al*. (2011) and Remzi *et al*. (2013) who found that application of olive mill waste increased organic matter content in soil.

Calcium Carbonate Content

In general, results indicate that the soil $CaCO_3$ decreased with increasing the rate of SOMW application as well as increasing the incubation period (Table 3). These results agree with those obtained by Doula *et al*. (2009) who found that application of olive mill wastes to clay or silt clay soils, decreased $CaCO_3$ content in soil. The increased organic acids and CO_2 evolution during the decomposition of the organic materials with increasing both the application level and the incubation time may cause dissolution of $CaCO_3$ in the soil (Hamed, 2008).

Soil Phosphorus Forms

Data in Table 4 show effects of SOMW application and the incubation period on soil phosphorus forms. Generally, values of all P forms except the residual form increased with increasing the application level of SOMW.

Soluble -P

The soluble P is considered the most available P pool for plant uptake. Soluble P increased with application of SOMW, and there was an increase with time up to 60 days and then, decrease occurred at 90 days (Table 4). These results agree with those of Kashem *et al*. (2003) who found that water soluble P increased with applying bio-solids, and cattle manure. The highest value of water soluble P was recorded at the highest application level of SOMW and 60 days of incubation. On the other hand, the lowest value was obtained at the lowest level of SOMW and zero time of incubation compared to the control treatment.

$NaHCO_3$-P

The $NaHCO_3$ extractable P is closely associated with P uptake by plants (Aulakh *et al*., 2003). Result of the current study cleare that both $NaHCO_3$-inorganic extractable P (P_i) as well as levels of the $NaHCO_3$-organic extractable P (P_o) increased with increase the application rate of SOMW (Table 4). López-Piñeiro *et al*. (2011) reported that increased contents of P forms in soil upon applying olive mill wastes. The highest values of both $NaHCO_3$ P_i and P_o were obtained at the highest rate of SOMW application at 60 days of incubation time. This result is in agreement with those obtained by Kavvadias *et al*. (2010) who found that application of olive mill waste to soil increased Olsen extractable P up to 28 times. Damodar *et al*. (2000) found that the amount of $NaHCO_3$ P_i and P_o increased to a highest level upon adding manure. Kashem *et al*. (2003) showed that $NaHCO_3$ extractable P_i and P_o increased with the addition of cattle manure as well as P fertilizers. Their results also showed that $NaHCO_3$-extractable P_i were higher than those of $NaHCO_3$-P_o ones. They attributed this to that the low organic matter in the soil.

NaOH-P

The NaOH-extractable P is strongly held P by chemisorption to Fe and Al components in soil (Williams *et al*., 1980). Soil phosphorus extracted by NaOH includes both inorganic and organic P forms. Both P_i and P_o forms extracted by NaOH increased with increasing the level of SOMW (Table 4). The values of P_i extracted with NaOH were higher than those of P_o. Both forms increased with increasing incubation time up to 60 days and then decreased. Contents of both P forms were highest in soil of the highest rate of SOMW addition after 60 days of incubation. Other studies showed a similar increase in the NaOH-P_i of the soil due to applying organic matter (Iyamuremye *et al*., 1996). Saleque *et al*. (2004) showed that NaOH-P_i was lowest in soils which did not receive organic matter.

HCl-P

The HCl-extractable P is the Ca- and Mg-associated inorganic P (Williams *et al*., 1971). Results regarding HCl extractable P are present in Table 4. Contents of HCl-P increased with increasing the applied level of SOMW (López-Piñeiro *et al*., 2011). Also, there was an increase with increasing the incubation time up to 60 days, then HCL-P was decreased after 90 days. The highest value of HCl-P was obtained at the highest level of SOMW and after 60 days of incubation. Inskeep and Silvertooth (1988) attributed the increase of available P in the calcareous soils, to organic acids produced during decomposition of organic materials in the soil. On the other hand, Zheng *et al*. (2003) reported that the HCl-P was predominant P pool in the soil and its relative content was not significantly affected by either liquid dry manure or mineral fertilizers.

Table 4. Effects of SOMW and the incubation time on soil phosphorus forms of the calcareous soil

SOMW Rates (g kg^{-1})	Water Soluble- P	$NaHCO_3$-P		NaOH-P		HCl-P	Residual-P
		P_i	P_o	P_i	P_o		
	Mgkg^{-1}						
			Zero time				
0	23.16	55.29	34.36	65.6	43.29	187.15	588.71
10	37.21	68	46.2	76.6	61.49	256.62	496.44
20	54.3	77.26	59.19	86.18	72.42	323.19	415.02
30	61.52	87.11	64.18	93.71	77.83	362.93	385.28
			30 days				
0	24.23	54.25	37.22	67.2	45.45	191.23	577.98
10	47.81	77.43	56.44	88.34	65.23	247.21	460.10
20	68.06	86.59	66.28	100.87	77.44	336.56	351.76
30	75.1	97.13	73.23	109.82	83.52	375.85	317.91
			60 days				
0	26.33	57.36	39.35	70.82	51.17	209.04	543.49
10	56.94	85.24	64.15	112.37	73.11	259.26	391.49
20	80.12	96.97	73.67	120.93	81.14	361.98	272.75
30	94.16	105.71	81.2	130.11	91.23	382.09	248.06
			90 days				
0	22.47	55.49	36.4	67.19	48.54	201.98	565.49
10	55.96	84.89	63.86	112.23	73.07	258.89	391.66
20	79.22	94.55	72.91	124.25	83.98	362.54	270.11
30	92.17	99.34	81.81	134.31	96.91	380.71	247.31

Residual-P

The residual P pool contains the phosphorus that is likely in the stable humus fraction (Stewart *et al*., 1980). Application effect of SOMW on the residual P of the calcareous soil is shown in Table 4.Generally, residual P decreased with increasing both SOMW level and the incubation time. The highest value of the residual P was recorded in the untrated at zero time of incubation while, the lowest one was shown in soil treated with the highest SOMW level after 90 days of incubation. The decrease in the residual P may be due to increases in the other P forms. Iyamuremye *et al*. (1996) reported that organic or inorganic amendments had little effects on residual P with slight but constant increases from some recalcitrant P forms present in the organic residues products decomposition. Kpomblekou and Tabatabai, (2003) stated that olive mill wastewater has potential to increase P in calcareous soils, but the action of its acids is considerably reduced by free carbonate in the soil.

Effects of Adding SOMW on Dry Matter Yield and P Uptake by Wheat Plants Grown on Calcareous Soil

Table 5 shows that adding of SOMW decreased dry matter yield and P uptake by wheat plants. Plants which received the highest rate produced the lowest dry matter yield and P uptake. This result coincides with those obtained by Remzi *et al*. (2013) and Killi and Kavdır (2013) who applied olive oil solid wastes at

Table 5. Dry matter yield and P uptake by wheat plants grown in the calcareous soil amended with SOMW

SOMW rats ($g\ kg^{-1}$)	Dry matter yield (g/pot)	P uptake (mg/pot)
0	1.48	11.99
10	1.31	8.25
20	1.18	5.04
30	1.09	3.49
LSD $_{0.05}$	0.06	0.52

rates up to 70 g kg^{-1} and obtained a decrease in wheat growth. They attributed the negatively affected to phytotoxicity and high C: N ratio of the waste amended. Previous studies by Vlyssides *et al*. (2004) showed that excessive application of olive solid waste compost (above 80 g kg^{-1}) resulted in a reduction in plant photosynthetic performance .Toxins and organic acids contained in unprocessed olive solid wastes had been demonstrated to result in significant microbial inhibition when applied directly to soils (Diacono *et al*., 2012). Such negative effect on microbial activity impairs soil health, decrease availability of nutrients for plant growth (Hamilton and Douglas, 2001; Wardle *et al*., 2004).

Conclusion

Organic wastes of various origins and nature are widely used as amendments to increase soil organic matter and crop productivity. Application of the investigated SOMW to a calcareous soil improved chemical properties of the soil. On tents of all P forms except residual P one, increased with applying SOMW indicating its importance in the redistribution of P among its various soil forms. Therefore, it may be recommended to apply to calcareous soils, particularity those that are newly reclaimed.

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استخدام مخلفات معاصر زيت الزيتون الصلبة كمصلحات للتربة الجيرية وتأثيرها على صور الفوسفور وبعض خصائص التربة الكيميائية

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أجريت تجربة تحضين لدراسة تأثير إضافة المخلفات الصلبة لمعاصر زيت الزيتون Solid Olive Mill Wastes (SOMW) على صور الفوسفور وبعض الخواص الكيميائية للتربة وكذلك المادة الجافة والكمية الممتصة من الفوسفور بواسطة نباتات القمح وذلك فى تربة جيرية جمعت من الطبقة السطحية (صفر - ٢٠ سم) من منطقة الوادي الأسيوطى بمحافظة أسيوط، مصر، تم حساب مستويات الإضافة على أساس محتوى SOMW من المادة العضوية، حيث تم خلط المستويات صفر، ١٠ ، ٢٠ ، ٣٠ جم/كجم مع ٢٠٠ جرام من عينة التربة تحت الدراسة وتم توصيلها إلى السعة الحقلية ثم تم تحضينها عند درجة حرارة ٣٠ ± ٢ ºC لمدة صفر، ٣٠، ٦٠ و٩٠ يوماً، أشارت النتائج إلى زيادة صور الفوسفور Soluble-P HCl-P, NaOH -P, NaHCO3- P مع زيادة مستويات SOMW حتى ٦٠ يوماً من فترة التحضين، ثم انخفضت قليلا خلال فترة ٩٠ يوماً، من ناحية أخرى، انخفض الفوسفور المتبقى Residual-P مع زيادة المستوى من SOMW ومع زيادة فترة التحضين عموماً، كانت أعلى قيم لصور الفوسفور (ماعدا صورة الفوسفور المتبقى) مع أعلى مستوى للإضافة منSOMW، بالنسبة لخواص التربة الكيميائية، أعطى التوصيل الكهربائي (EC) للتربة تحت الدراسة زيادة معنوية بينما أنخفضت حموضة التربة وكربونات الكالسيوم مع زيادة مستوى الإضافة من SOMW وزيادة فترة التحضين أيضاً، زادت المادة العضوية في التربة مع زيادة مستوى الإضافة من SOMW بينما انخفضت مع زيادة فترة التحضين، بالإضافة إلى ذلك، أدت زيادة مستوى الإضافة إلى انخفاض فى المادة الجافة والكمية الممتصة من الفوسفور بواسطة نبات القمح النامى في هذه التربة.

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