Recycling of olive oil Mill effluents stored in ponds through agricultural land disposal.

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Abstract
To promote and assess agricultural use of olive Oil-Mill effluents (OME) stored in evaporation ponds, the characteristics, fertilizer value and evolution of this wastewater were determined in 11 different ponds during two seasons. The effects of irrigation with OME in olive trees and soils were studied in four experimental fields. The OME pH was initially acid (4.5-6.5) but later became basic values (7.5-9). As the OMEs were concentrated, their electrical conductivity, soluble solids and metals in solution, except calcium, slightly increased. The nitrogen and total carbon concentrations remained relatively stable. Potassium was the most abundant element, its mean concentration being 1288 mg/L. No significant differences were found between yields and nutrient content of olive trees irrigated with OME and controls. The mean yield of olives was of 44.6 ± 0.98 kg/tree. At the end of the trial, the reserves of potassium and magnesium available in the soil did rise by 200 and 17%, respectively, but the rest of the parameters measured did not undergo any significant changes.

Introduction
Growing olive trees for oil has been experiencing a notable expansion. In Spain, the production of olives for oil has gone from 4.7 million tonnes in 2000 to 6.7 million tonnes in 2010. Parallelly, the extraction industry has been modernized and the adoption of the continuous 2-phase centrifugation system has been generalized. This process originates two classes of by-products: a semi-solid organic paste and a liquid coming from the washing water of oils and olives, which is obligatorily stored in evaporation ponds. These ponds, which are of a considerable capacity and surface, not only collect the abovementioned liquid effluents but also the rainwater coming from the facilities devoted to oil extraction. However, the ponds are the source of numerous environmental and economic problems. Also, there is a great deal of information supporting the agronomic use of effluents which are more concentrated than OME [1] [2] [3] [4] [5] [6].
The objective of this work has been to characterize and study the evolution of effluents stored in ponds in order to assess the benefits and limitations of their possible application on agricultural soils.

Material and Methods
Sample taking and analyses:
The ponds of 11 olive oil mills (almazaras) were sampled fortnightly for two years in the periods going from the end of the season (first days of March) to the end of August.
In the effluent samples the following parameters were determined systematically: pH, total nitrogen, and soluble phosphate, potassium, sodium, copper, calcium and magnesium, chemical oxygen demand, solid residues, total carbon, electrical conductivity, density, bicarbonates and chlorides. The polyphenols have occasionally been measured.
Field Experiments:
The effect of these effluents has been studied in four assay fields established in olive groves located in the province of Córdoba (Spain). The fields measured between 1 and 2 hectares. The elemental plot consisted of 10 trees surrounded by two rows of trees to isolate each individual plot. The main characteristics of the fields are shown in Table 1:
It was planned to apply 3 irrigations of 800 l per tree with a 8 m^3 slurry tanker during the months of July and August in each season. In the first year of the assay it was only possible to apply two irrigations except in the "S Seb" field, which received three. The treatments were replicated four times.

Table 1: Mean characteristics of soils (20 cm depth) in the four assay fields. Each value is that of the mean of four data corresponding to a composite sample taken from each elemental plot.

Local.	pH water	Org, Mat. %	Avail. K	Avail. P	Avail. Mg	Sand	Silt	Clay	$CO_3^=$	Particle classes
			mg/kg			%				
Cañ	8.14	1.07	299	17	185	35	34	31	25	Sandy-Loam
S Seb	7.82	1.73	346	43	117	47	30	23	2	Loam
FN	8.30	0.65	170	15	56	45	34	21	37	Loam
NC	8.42	1.64	114	15	50	22	47	31	79	Sandy-Loam

The mean characteristics of the effluents and the volumes employed for irrigation in each locality during the assay are detailed in Table 2:

Table 2. Mean characteristics of the effluents used for irrigation during the two seasons.

Local.	pH	Elect. Conduct. dS/m	Sol.K	Sol.P	Sol. Ca	Sol. Mg.	Solids %	DQO mg O_2/l	Volume applied m^3/ha	Density g/cc
			g/m^3							
Cañ	8.57	10.4	3785	33	407	119	1.85	8648	400	0.97
S Seb	8.21	2.6	603	10	196	31	0.20	1273	960	0.97
FN	8.27	6.9	2037	7.8	227	68	0.73	3376	625.	0.99
NC	8.14	2.8	515	4.1	177	32	0.25	1015	625	0.98

During the seasons of 2009/10 and 2010/2011 the olive and oil yields, the size of the olives and the nutrient level in the tree and soil were measured and the soil was sampled at 0-13cm and 14-26 cm.

Results

It was observed that there was a greater difference between olive-oil-mill ponds than between seasons. The pH was the least variable parameter; it underwent a gradual increase as the time that the effluent was stored in the ponds went by. The pHs of the OMEs at the beginning of the sampling presented acid values (4.5-6.5), which gradually evolved to basic ones (7.5-9). Their mean value was 7.19 and coefficient of variation (CV) 19%. Nitrogen and total carbon remained relatively stable, with a mean of 0.93 and 29.3% and a CV of 47 and 35%, respectively. The metals became concentrated as the effluents evaporated, with the exception of calcium, whose concentration diminished as the pH of the effluent increased (Figure 1). Potassium stood out as presenting a high mean concentration of 1288 mg/l. The only potentially toxic trace metal whose presence could be limiting was copper. It appeared in very low concentrations, with a mean of 0.07 and median of 0.02 mg/l. The solid residues also underwent a light concentration throughout the season, with the highest values being found at the end of August. Their mean concentration was of 8.7 g/l and their median of 4.8 g/l. The phosphates hardly altered with the time and presented a mean concentration of 26.8 mg/l.

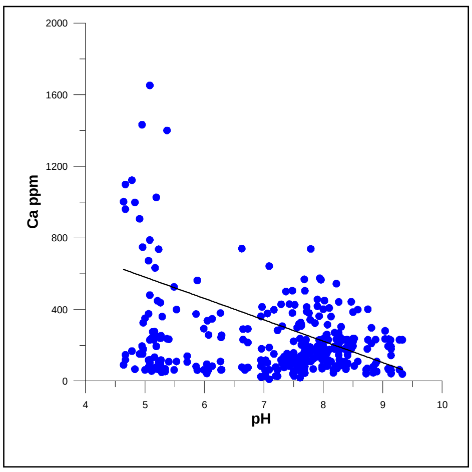

Figure 1.Relationship between the evolution of the pH of the OMEs and their content in soluble calcium

Electrical conductivity is an indirect measurement of the amount of salts dissolved in OMEs. As a general rule it increased, the same as the pH, at the same time as the storage time in the evaporation ponds increased. The values found at the beginning of the sampling reflect, in some cases, salinity inherited from previous seasons. The initial values ranged between 2 and 5 dS/m, rising in July to values of between 3 and 8. In August, when the OMEs were concentrated these values reached 11-25 dS/m. Thus, pond number 11, which began the season 2009/10 by being completely clean, is an example of the evolution of those parameters with no outside influences. In this pond, the initial value evolves from 2.6 to 3.5 dS/m. The mean of all the determinations was of 5.29 dS/m, with a median of 3.82 dS/m and a coefficient of variation of 71%.

No effects were detected from irrigation with effluents on: olive or olive oil yields or on 100 fruits size during the two seasons. The olive average yield was estimated at 44.60 ± 0.98 kg /tree, and a significant difference between localities was found.

These two years were very rainy ones and a slight effect of the agronomic year was observed, in such a way that the average harvest was somewhat smaller in 2010/11 than in the preceding one. The S Seb field did not follow this general trend and its harvest in that year was significantly larger.

The nutritional state of the olive grove and the possible modifications caused by irrigation with effluents in the trees were studied by means of an analysis of the leaves of the olive trees used in the assays. Only in the Cañ field was a decrease in the potassium levels in the first year of the assay observed, which can hardly be attributed to the effect of the irrigation, begun in that same year and month. The agronomic years were more of an influence on the concentrations than the irrigations.

The nutrient levels in the olive trees were not significantly modified by the OMEs. In studying together the data from the two samplings, it was verified that the potassium levels in the leaves of the olive trees found in S Seb and NC fields were significantly lower and closer to the deficiency limit (<0.8%) than those present in FN and Cañ olive trees. In the case of nitrogen, the NC field gave concentrations close to the deficiency range (1.4%). The highest levels were found in the S Seb olive trees. The phosphorus levels in all the locations were always above the levels considered to be sufficient. The S Seb field stands out with very much higher phosphorus concentrations than those in the rest of the assays.

To assess the effects of irrigation with effluents on the soil, the sampling made at the end of the assay was employed since the intermediate sampling barely showed any significant changes. A summary of the data obtained can be seen in Table 3.

The pH of the irrigated soils underwent a generalized and small rise over the control, of the order of 2% in the top surface of the soil (0-13 cm). This increment was detected both in the measurement of an aqueous extract and in a weak solution of Cl_2Ca (0.01 M). In the deepest horizon (14-26 cm) no significant changes were noted.

The abovementioned increase in pH was accompanied by a slight increment in extractable sodium in the 26 cm analyzed. The mean value of sodium in the first 13 cm of the four fields rose from 49 to 75 ppm and from 50 to 69 ppm in the horizon of 13-26 cm, these values all being considered low or very low ones. The percentage of exchangeable sodium in the soils irrigated ranged between 0.83 and 2.20 for the first 13 cm and 0.84 and 2.45 in 13-26 cm. These percentages were far removed from the value of 15% considered to be a limit for alkaline soils.

The greatest fertilizing value of these effluents lies in their richness in potassium. Irrigation significantly increased the potassium available in all these places. Its increase doubled the original values (Table 3). The mean value of the four control fields was of 241 ppm and rose to 583 ppm in irrigated fields. The increment experienced by the soil is a purely superficial one since, at a greater depth (13-26 cm) it does not reach any significance.

Table 3. Effect of irrigation on some parameters on top surface of soil (0-13 cm). R treated and T soil control.

Loc.	pH water		pH Cl_2Ca		Avail. K ppm		Avail.Na ppm		Int. Na . %/CEC		Avail. Mg ppm		C org. %	
	R	**T**	**R**	**T**	**R**	**T**	**R**	**T**	**R**	**T**	**R**	**T**	**R**	**T**
Cañ	8.53	8.4	7.76	7.58	1253	375	99	42	1.96	0.83	216	185	1.14	0.76
S Seb	8.07	7.9	7.28	7	522	287	47	28	0.83	0.49	145	117	1.27	1.11
FN	8.58	8.29	7.7	7.57	380	191	66	54	2.09	1.71	64	56	0.63	0.73
NC	8.45	8.47	7.6	7.55	176	112	90	72	2.20	1.76	57	50	1.08	1.02
Signif..	●		*		*		●		●		***		**NS**	

*** $p < 0.001$ %, * $p < 0.05$ % y ● $p < 0.10$ %.

The effluent is also a valuable source of magnesium. The content in available magnesium of irrigated soils significantly increased by 17%. The mean content of available magnesium in the 0-13 cm horizon went from 102 ppm in the control soils to 121 in the irrigated ones. Its levels in the deepest horizons were not modified.

It should be noted that, although all the trees were irrigated with the same dose of effluents, their mean composition was very different, as can be verified in Table 2. The highest and lowest increases in available potassium and magnesium were observed in the fields receiving the highest (Cañ) and lowest (NC) potassium and magnesium inputs. It was seen that the FN field did not respond to irrigation in the same way as the rest of the assay fields.

The remaining assay parameters studied in the soil (nitrogen and organic carbon, available phosphorus) have still not reflected any statistically significant modifications. However, the higher inputs made in Cañ also increased them to a greater extent than in the rest of the fields.

Conclusions

The effluents stored in ponds from mills with the 2-phase system are apt for being re-used as a fertilizing irrigation in agricultural lands in view of their lack of any pollutants at levels potentially dangerous to the soil, and for their richness in organic matter and potassium. The volume and conditions of their application should be regulated to prevent runoff and pollution of water courses. To study their effects on olive trees and their yield, assays of a longer duration would be necessary.

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