Proposed Soil Indicators for Olive Mill Waste (OMW) Disposal Areas

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Abstract The disposal of olive mill wastes (OMW) is considered as a major environmental problem worldwide, but especially for Mediterranean countries. Disposal in evaporation ponds or directly on soil is a common practice, which causes serious damages to soil and to the environment. The present study was performed in the framework of the LIFE project "Strategies to improve and protect soil quality from the disposal of Olive Mill Wastes in the Mediterranean region-PROSODOL" and one of its main objectives was the identification of appropriate soil parameters that could be used as soil indicators to assess soil quality at OMW disposal areas. For this, a well-designed soil sampling strategy was developed and implemented in Crete, South Greece at five OMW disposal areas. Many soil parameters were monitored bimonthly for a year. After statistical evaluation, eight soil parameters were selected as being appropriate soil indicators for OMW disposal areas, i.e., electrical conductivity, pH, organic matter, polyphenols, total N, exchangeable K, available P, and available Fe. Although many researchers have extensively studied the effect of OMW on soil quality, yet the identification of soil indicators to assess and monitor soil quality is an innovative issue and has never been studied before.

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1 Introduction

Soils that accept wastes disposal, apart from progressive degradation, may cause serious problems to the surrounding environment (humans, animals, plants, water systems, etc.), and thus, soil quality should be necessarily monitored. Therefore, quality indicators, representative of the specific waste type, should be established and monitored periodically. Since waste composition is dependent on their origin, specific indicators for each waste type should be established. Considering agricultural wastes, such as specification, however, is difficult, since almost all agricultural wastes are characterized by increased concentrations of the same elements, namely, phosphorous, nitrogen, potassium, sulfur, etc.; contain large amounts of organic matter; and have very high values of chemical oxygen demand (COD), biochemical oxygen demand (BOD), and electrical conductivity.

1.1 Olive Mill Wastes

Disposal of solid and liquid wastes from olive oil mills is a major environmental issue in several olive-growing countries in the world. Inappropriate disposal of olive husk and olive mill wastewater creates environmental problems such as odor and ammonia released into the atmosphere and leaching of inorganic and organic substances to the soil as well as leaching of these pollutants into the groundwater.

The works of Sierra et al. ([2001\)](#page-10-0) and Kavvadias et al. ([2010,](#page-9-0) [2011](#page-9-0)) have shown that olive mill wastewater infiltration in the soil has caused carbonate dissolution and redistribution and modifications in pH values, electrical conductivity, nutrient contents, phenolic compounds, and biological activity of the soil horizons. The olive mill wastes have been, and continue to be, disposed onto farmlands, thus causing the inhibition of numerous microorganisms, a reduction in seed germination, and the alteration of several soil characteristics such as porosity and humus concentration. The introduction of olive solid and liquid waste into soil tends to increase the average diameter of the soil aggregates, bulk density, and slows down hydraulic conductivity (Colucci et al. [2002\)](#page-9-0). Polyphenols in olive husk are well known to affect nitrification in the soil and have deleterious effect on soil microbial activity. The high C/N ratio and low pH in the olive husk are also known to immobilize nitrogen in the soil (Benitez et al. [2004\)](#page-9-0).

The olive husk and wastewater produced from oil extraction processes contain macromolecules such as polysaccharides, lipids, proteins, and a number of monocyclic and polymeric aromatic molecules generally known as phenolic compounds. The levels of phenols in wastewater and olive husk can vary from 1 to 8 g/l (Di Gioia et al. [2002;](#page-9-0) Nair and Markham [2008](#page-10-0)) and from 2.9 to 3.7 mg/g (Nair et al. [2007\)](#page-10-0), respectively. The wastewater is also characterized by dark color due to chromophoric lignin-related materials with different degrees of polymerization and a sharp characteristic odor (Sayadi et al. [1996](#page-10-0)). Olive husk is also characterized by phytotoxicity, hydrophobicity, salinity, low pH, and polyphenols (Perucci et al. [2006](#page-10-0)). The presence of phenols as well as short and long chain fatty acids is considered to be responsible for the phytotoxicity (Isidori et al. [2005\)](#page-9-0) and antimicrobial (Fierentino et al. [2003;](#page-9-0) Isidori et al. [2005](#page-9-0)) nature of these wastes.

1.2 The Utility of Indicators: a General Approach

In principle, an indicator could be either a qualitative (nominal) variable, a rank (ordinal) variable, or a quantitative variable (Gallopin [1997\)](#page-9-0).

An indicator may be easy to measure and summarize in shorthand the effects of complex processes that are more difficult to measure or observe (Landres [1992;](#page-9-0) Harris et al. [1996\)](#page-9-0). Its purpose is to show how well or bad a system is working. If there is a problem, an indicator is useful in determining what direction to take to address the issue. Indicators can also be useful as proxies or substitutes for measuring conditions that are so complex that there is no direct measurement.

In summary, desirable indicators are variables that summarize or otherwise simply relevant information, make visible or perceptible phenomena of interest, and quantify, measure, and communicate relevant information. In addition, some indicators may be used to evaluate a condition or phenomenon. Indeed, it is maintained that one of the essential functions of indicators is to quantify. Moreover, indicators can also relate to qualitative phenomena.

Effective indicators, in addition to being quantifiable, are characterized by four basic features (Adriaanse [1993](#page-9-0)):

- Relevance: An indicator must be relevant, that is, it must fit the purpose for measuring. It shows something about the system that is needed to be known, for its assessment.
- Understandability: An indicator must be understandable, even by people who are not experts.
- Reliability: An indicator must be reliable. The user must trust the information that the indicator is providing. Reliability is not the same as precision. An indicator does not necessarily need to be precise; it just needs to give a reliable picture of the system it is measuring.
- & Accessibility of data: Indicators must provide timely information. In order for an indicator to be useful in preventing or solving a problem, it must give the information while there is still time to correct the problem. The information is available or can be gathered while there is still time to act.

1.3 Indicators for Soil Degradation Assessment

Soil degradation is difficult to comprehend in its totality while soil productive capacity cannot be assessed simply by any single measure. Therefore, to make assessments of soil degradation viable, indicators of its processes and effects have to be used. In the context of soil degradation due to waste disposal, indicators are variables that may show that degradation has taken place; they are not necessarily the variables controlling the

actual degradation itself. These indicators may be drawn from any aspect of how quality of soil degrades. Since there is much interlinkage between the various types and manifestations of land degradation, indicators are considered as a powerful tool for overall assessments (Stocking and Murnaghan [2001\)](#page-10-0).

Indicators need to be sensitive to changes in both management and climate. Soil characteristics that change within only a few weeks or months in response to the changing seasons, shifting weather patterns, and plant growth cycles are not appropriate soil quality/degradation indicators. Characteristics that begin to show change only after five or more years are not helpful indicators, often showing progressive soil degradation only after much of the productive topsoil is lost. The best soil quality/degradation indicators are those characteristics that show significant change between 1 and 3 years, with 5 years being an upper limit of usefulness.

Thus, the duration of an indicator's significance varies with the permanence of the data used to build the indicator. Some data change over the long term (e.g., topography and river networks) and are relatively permanent; others may change over the medium term (e.g., flora or erosion type); and some show short-term changes over years or seasons (e.g., soil moisture or livestock management).

The objective of this study is to establish a set of indicators that will be main soil parameters, specific for soils that accept olive mill wastes (OMW), which will assist in the continuous monitoring of soil quality and health. The periodic and scheduled sampling and identification of these parameters would allow controlled waste disposal and the undertaking of necessary measure and means in order to maintain soil quality and thus environment improvement and protection.

2 Materials and Methods

2.1 Area Under Study

For the achievement of the objective of the present study, many soil physicochemical parameters were monitored for a year by analyzing soil samples collected bimonthly from five OMW disposal areas (active and inactive) in Crete, South Greece.

The area under study belongs to the municipality of Nikiforos Fokas, prefecture of Rethymnon, Crete,

Greece; North latitude is 35°17′ while the East longitude is 24°21′. The region has subtropical/Mediterranean climate and is characterized by mild winters and dry– hot summers. The annual temperature varies between 7.9 °C in February (mean minimum temperature) and 28.4 °C in July (mean maximum temperature). Average precipitation is 692 mm; most of it falls between October and April while no precipitation is seen during summer. Limestones cover almost 40 % of the total area $(8,300 \text{ km}^2)$ of the island of Crete; dolomites, marbles, and alluvial deposits are also seen. Soils in the area under study are clayey or silty clayey, slightly to moderately alkaline and rich in carbonates, not well developed (mainly Entisols), highly eroded, and protected by terraces (Kavvadias et al. [2010](#page-9-0)). The OMWs are mainly disposed in evaporation ponds or directly on soil.

2.2 Sampling Strategy

A carefully designed—based on the land characteristics of the area—monitoring system was implemented in the pilot area, to monitor the quality of soil at OMW disposal areas.

Six soil sampling campaigns took place:

- & First sampling between 5 and 7 May 2009
- Second sampling between 6 and 9 July 2009
- & Third sampling between 28 September and 2 October 2009
- Fourth sampling between 14 and 18 December
- Fifth sampling between 1 and 5 March 2010
- Sixth sampling between 17 and 21 May 2010

Five sites, representing different disposal cases, were studied. The soil data over the six sampling campaigns from the monitoring sites are presented:

- Control soils: Representative soil of the area located in a distance from the waste disposal ponds.
- Pond soils of all active sites: All evaporation ponds were constructed by using native soil and simple engineering, while no impermeable membranes or other protective media were used. The ponds are in operation for more than 11 years.
- & Active sites with surface disposal of OMW (ACTDS): Three active sites were monitored which are in operation for more than 11 years:
	- ACTDS-1
- ACTDS-2
- ACTDS-3

ACTDS-1 is a typical disposal site located in a field with almost 10 % slope; pond dimensions are 50×10×10 m. ACTDS-2 represents a different case; the pond $(21\times8\times1.70$ m) was constructed by using native soil material excavated from the top of an adjacent hill. ACTDS-3 is a large field (1 ha) with almost 5 % slope and contains two evaporation ponds with dimensions 32×4.20×1.70 and 30×44×1.75 m, respectively. Direct disposal of OMW on soil takes place at ACTDS-3 every 2–3 days between May and September each year.

- Pond soils in inactive sites (INACTS): Two inactive sites were monitored:
	- INACTS-4
	- INACTS-5

which have been used for the disposal of OMW for 20 years but for the last 8 years are inactive. The dimensions of the inactive ponds at INACTS-4 site are 24×17×1.75 m and at INACTS-5 site are 28×6×1.30 m.

River site located at the downstream of the active site with surface disposal of OMW. Soil samples along the riverbanks were collected and analyzed in order to assess the potential of water pollution and nutrient loss pollution, from soils of the upper hill slopes, through runoff or downward leaching.

In order to select the sampling sites, an initial characterization of the physical characteristics of the soil including its type and the climatic conditions of the sites were evaluated with the help of simple tests (e.g., soil color, grain size, compaction–penetration test). The number of soil samples was decided by considering the general status and the characteristics of each disposal area and its history. The number of samples was the appropriate in order to obtain representative results. With this in mind, many samples were collected from the active disposal areas, while for the inactive areas, since the ponds were dry, it was decided to collect one sample from inside the pond and one control sample from a clean area near the pond each sampling time. The monitoring system foresaw the collection of soil samples at various depths from (a) disposal ponds, (b) close and around

the disposal ponds and mostly from the down slope side of the ponds, and (c) downstream of the disposal ponds where extensive leaching due to surface and subsurface water movement is likely to occur.

Soil samples were collected from:

- The pond walls and from the wider area of the disposal ponds from selected downslope distances, up to 105 m (usually every 3–4 m): In depth, samples were collected at 25 cm intervals up to 175 cm.
- Inside the ponds from the three active sites: In depth, samples were collected at 25 cm intervals up to 175 cm.
- Inside the inactive ponds: In depth, samples were collected at 25 cm intervals up to 175 cm.
- & Clean areas to be used as control samples: The samples were collected from all areas under study, namely, representative soil of the area located in a distance from the waste disposal ponds where soil contamination is practically nonexistent.

During the six sampling campaigns, 505 soil samples were collected from the disposal areas, while almost 16,200 analyses were conducted. Soil analysis was carried out using standard methodologies (Page et al. [1982\)](#page-10-0) as described in detail by Kavvadias et al. [\(2010](#page-9-0)). Soil samples were categorized relative to soil classification method and analyzed for: electrical conductivity (EC), pH, water saturation, total salts, texture, CaCO₃, organic matter, nitrogen, available P, cation exchange capacity, exchangeable K, exchangeable Ca, exchangeable Mg, exchangeable Na, watersoluble Na, available Fe, available Mn, available Cu, available Zn, total polyphenols, available B, watersoluble anions, i.e., CI^- , NH_4^+ , $SO_4^2^-$, $PO_4^3^-$, and NO_3^- , and microbial activity.

Olive mill wastes were produced from three-phase mills using the continuous centrifuge extraction process and have the following characteristics: pH 4.9– 5.4; EC 7.6–8.1 dS/m; total organic carbon 34–37 g/l; BOD 35–42 g/l; COD 55–74 g/l; total Kjeldahl N 750–790 mg/l; NH4 ⁺ 121–164 mg/l; total phenols 8,500–9,200 mg/l; Mg 152–160 mg/l; P 430– 480 mg/l; K 4,200–4,700 mg/l; Ca 430–500 mg/l; and Na 106–118 mg/l. The chemical parameters of OMW were determined in duplicates using established methodologies (Clesceri et al. [1998\)](#page-9-0). The initial digestion of the waste samples was performed by using the EPA 3052 method with $HNO₃$ digestion (EPA [1996\)](#page-9-0) in a microwave oven. The total phenol content was determined after extraction with methanol and subsequent quantification using the Folin–Ciocalteu method (Box [1983;](#page-9-0) Gutiérrez Gonzales-Quijano et al. [1977](#page-9-0); Allouche et al. [2004;](#page-9-0) Di Serio et al. [2008\)](#page-9-0).

3 Results and Discussion

3.1 Set of Soil Indicators for OMW Disposal

The continuous monitoring of the pilot area revealed that not all of the measured parameters are affected by the disposal of OMW. In particular, some of the measured parameters remained almost unchanged or the changes recorded were not significant relative to the control soil samples used for comparison (e.g., exchangeable Ca); others were subject to changes, but their values depended also on the different seasons and thus are inappropriate to be used as indicators (e.g., Cl⁻, NH₄⁺, SO₄²⁻, PO₄³⁻, NO₃⁻, microbial activity). Other parameters were significantly changed due to waste disposal, but this change lasted for a short time after ceasing of waste disposal, although the area was still very much degraded (e.g., N, B). Finally, there were parameters that exhibit significant changes which strongly depended on OMW disposal (e.g., organic matter, exchangeable K, available Fe). From the evaluation of the obtained results, it was clear that, since soil degradation at OMW disposal areas remains significant also for inactive-abandoned disposal areas, the indicators to be established should cover these two potential cases, namely, active disposal areas and inactive disposal areas.

Thus, it was decided, in order to select the most suitable soil parameters, to consider the following:

- 1. The four features mentioned before, i.e., relevance, understandability, reliability, and accessibility of data (Table [1\)](#page-5-0)
- 2. The percentage of soil samples collected from all disposal areas (active and inactive) that exhibit high values of the measured parameters compared to the control samples (Table [2\)](#page-6-0)

Regarding the four features, all measured soil parameters were characterized regarding relevance, understandability, reliability, and accessibility. The first column of Table [1](#page-5-0) describes the extent of OMW disposal effect on each soil parameter. For this, the results of the six soil sampling campaigns were used after statistical evaluation as well as data obtained from the literature (Sierra et al. [2001](#page-10-0)). An extensive and detailed description of the effect of OMW on soil parameters as well as details for the statistical processing of the obtained data is given by Kavvadias et al. [\(2010](#page-9-0), [2011\)](#page-9-0). As regards the feature "relevance," all parameters are considered relevant to OMW disposal since all of them were affected at some extent (i.e., significant or not). All of the parameters are also characterized by a high degree of understandability, except microbial activity, which, in comparison to the other parameters, is less understandable by people who are not experts and therefore are not able to evaluate it. In the same sense, all parameters are characterized by a high degree of reliability. However, as regards accessibility, not all parameters satisfy this requirement, as it was recorded during the soil monitoring stage of the present study and previous studies, as well (Kavvadias et al. [2010](#page-9-0), [2011](#page-9-0)). In specific, saturation percentage, dissolved salts, water-soluble $NO₃⁻, PO₄³⁻, Cl⁻, SO₄²⁻, and microbial activity were$ found to be subject to changes but their values were seasonal, while soil texture was not affected during the soil monitoring period. So, considering the evaluation of Table [1](#page-5-0), 13 soil parameters were initially selected (parameters marked with asterisk). It has to be highlighted that soil pH was not significantly affected by the disposal of OMW. This is because the soils of the pilot area were rich in clay and carbonates and had high pH values (between 7.2 and 7.8). These properties protect soils from the OMW's acidity and thus no changes were recorded in pH values. However, the effect of OMW on soils with lower pH and poor in clay and carbonates is anticipated to be significant, and for this reason, pH is included in the 13 preselected soil properties.

Following this first stage, the 13 preselected soil parameters were further evaluated regarding their concentrations. Table [2](#page-6-0) summarizes the statistical evaluation of the measured soil parameters for (a) control samples; (b) soils collected from active disposal sites with no surface disposal; (c) soils that accept surface disposal; and (d) pond soils collected from inactive disposal areas. The values presented are only those that are above the "high/very high" and "excessive" limits, considering the respective thresholds of Table [3.](#page-7-0)

 Y yes, N no

From the 13 preselected parameters, 8 were finally selected as being most appropriate for soil indicators. Exchangeable Mg was excluded since its background value is considered high, i.e., 50 % of the control samples have very high values compared to the thresholds of Table [3.](#page-7-0) Similarly, available Cu was excluded also due to very high background levels.

Also, available Mn, Zn, and B were not included to the proposed soil indicators due to the small percentages of affected soil samples, which had high values compared to the respective thresholds.

Thus, the following soil parameters are proposed as indicators for monitoring soil quality in areas of OMW disposal:

- Electrical conductivity
- Organic matter
- Total nitrogen
- Total polyphenols
- & Available phosphorous
- & Exchangeable potassium
- Available iron
- & Soil pH (mainly for acidic soil types)

All these soil parameters are characterized by the four basic features mentioned before:

& Relevance: All indicators are related to the disposal of OMW and as it was observed during the soil sampling campaigns and the analyses performed

The marked parameters (with an asterisk) are proposed as soil quality indicators

for many soil samples (affected and control), and during different seasons, their values are dependent only on disposal activity.

- Understandability: All indicators are soil parameters that are used for many years to characterize soil systems and thus are very much understandable, even by people who are not experts.
- Reliability: The proposed indicators are reliable as proved by many soil analyses, by periodically sampling from the same sites and by data evaluation.
- & Accessibility of data: Indicators provide timely information and as it was proved by the monitoring of

the disposal areas. One disposal was enough to increase these parameters to values much higher than the control samples. It is also significant to be mentioned that, during soil sampling campaigns, there were sites that were recognized as disposal sites after having analyzed these parameters.

3.2 Soil Color

Apart from the above soil parameters, one more that should not be ignored is soil color. Soil color is constituted of the overall hue (based on primary color),

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chroma (the strength of the color), and the degree of grayness (from black to white) of the soil. When soil degradation takes place, both texture and color change, and this change is often one of the first obvious indicators of soil degradation.

Color changes were observed for soils that accept surface disposal of OMW, while such degraded systems do not seem to recover after many years of last disposal. Munsell soil color charts give a full description and code for soil colors. It is necessary to standardize the moisture level of the soil for the color determination. Moreover, for soil degradation assessment, it is necessary to compare colors between undegraded and degraded conditions.

3.3 Monitoring Soil Indicators

The monitoring of soil quality indicators within a defined ecological zone requires (Arshad and Martin [2002\)](#page-9-0):

- & Direction of change: positive or negative increase or decrease, etc.
- Magnitude of percent change over the baseline values
- Rate of change duration: months, years
- Extent of change percentage of the area being monitored, i.e., what percentage of the area has changed with respect to the selected indicator during a specified period

Monitoring of soil indicators needs to set up sampling strategies allowing assessment of changes in soil quality. In general, changes in soil quality can be assessed by measuring appropriate indicators and comparing them with desired values (critical limits or threshold level) at different time intervals, for a specific use in a selected area system. A critical limit or threshold level is the desirable range of values for a selected soil indicator that must be maintained for normal functioning of the soil ecosystem health. Within this critical range, the soil performs its specific functions in natural ecosystems (Arshad and Martin [2002\)](#page-9-0). Thus, when a set of indicators is proposed, this list should be accompanied by threshold level for each one of the indicators in order to assist evaluation of collected data and of the chemical analyses results. The thresholds could be identified based on EU directives, on national laws, and also on the international literature. The peculiarity of the indicators proposed

for the case of OMW disposal is that they mainly correspond to soil properties associated with fertility and not to pollutants in the classical sense, such as heavy metals, and therefore are not included in national laws or EU directives. Nevertheless, the international literature can provide general limits as these properties have been extensively studied for many years. Given the complexities of setting limits and the uniqueness of each targeted area/region, it may be more efficient to develop guidelines that can help in setting up limits under certain land and environment conditions.

Thus, although a general definition of indicators thresholds could be performed after searching in international literature and national or EU legislative frameworks, it should be highlighted that the definition of indicators thresholds would be more effective and representative of each target area if they would be determined after evaluation of data collected from the areas of interest and by taking into account local characteristics and values of the indicators of representative control samples.

Especially for polyphenols, for which the assessment of their concentration in soil is considered difficult and with high degree of uncertainty due to the lack of generally accepted threshold, it is recommended to use local and site-specific thresholds as guidelines/normal values (Swartjes [1999;](#page-10-0) Sierra et al. [2001](#page-10-0); Mekki et al. [2007](#page-10-0); Di Serio et al. [2008](#page-9-0); Kavvadias et al. [2010](#page-9-0)).

A land information inventory of the area, based on geographic information systems (GIS), should be then designed and developed to store all collected data for further evaluation by local authorities, scientists, a.o.

4 Conclusions

The establishment of a set of soil indicators, specific for OMW disposal areas, would facilitate and promote effective monitoring of such highly degraded areas. The soil monitoring strategy that was developed during this study was implemented in five OMW disposal areas in Crete, South Greece. The results obtained revealed that some of the soil parameters satisfy all the criteria of effective and efficient soil indicators, and thus after statistical evaluation, eight soil parameters were proposed as appropriate indicators for assessing and continuous monitoring of soil quality at OMW disposal areas. The indicators are electrical conductivity, pH, organic matter, polyphenols, total N, exchangeable K, available P, and available Fe, while it is believed that their adoption (and by introducing a GIS-based land information inventory) could provide to national/local authorities an effective soil monitoring tool.

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