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# Radiation Processing of the Olive-Press Wastewater for Detoxifying the Water for Agricultural Reuse

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**Abstract:** The effect of gamma irradiation on the degradation of phenol and polyphones in olive-press wastewater was investigated. The radiation effect was evaluated for the main parameters COD and BOD5 of samples. Spectrophotometer (UV-Vis), chromatography (HPLC), and GC-mass were used to monitor the changes in the radiation solutions.
The results noticed that there was not any fort effect of radiation on the polyphenols at low doses, while some organic acids and aliphatic compounds were found by the analysis of the irradiated samples.
**Keywords:** radiation technology, treatment of olive-press wastewater, degradation of pollutants, and reused water.
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## 1 Introduction

Environmental pollution is now recognized as a significant concern worldwide. While industrial sources are most common other sources can also be significant. Many of the contaminants biodegrade very slowly and may have adverse effects on humans and ecosystems [1-2].
Olive-press wastewater (OPW) generated by the olive oil extraction process is the main waste product of this industry. Approximately, 1.8x106 tons of olive oil is produced annually worldwide where the majority (98%) of it is produced in the Mediterranean basin [3]. The olive-press wastewater production in Mediterranean countries is estimated to be over 3x107 m3 per year [4]. Treatment of OPW is becoming a serious environmental problem, due to its high organic COD concentration, and because of its resistance to biodegradation due to its high content of biomass-inhibiting growth, mainly phenolic compounds [5].
The improper disposal of OPW to the environment or to domestic wastewater treatment plants is prohibited due to its toxicity to microorganisms, and also because of its potential threat to surface and groundwater [6]. However, due to the current lack of appropriate alternative technologies to properly treat OPW, mostly, OPW in the Mediterranean area is discharged directly into sewer systems and water streams or concentrated in cesspools even though such disposal methods are prohibited in many Mediterranean countries [7].
Olive oil technology uses two methods for the production of oil from olive fruit: the discontinuous press process or the continuous solid-liquid centrifuge system. The "waste" consists of solid and liquid waste produced by whichever processing method is used [8].
To extract the precious oil from the mesocarp, or flesh cells, of the olive fruit, the extraction process naturally focuses on the separation of the oil and supplementary liquids from the solid material [9]. So, the process of olive oil production started firstly by washing the harvested olives with water and removing the leaves, which are the preliminary steps in this ancient process. Then crushing the harvested olives with either stone mills or metal crushers produces a paste with easily extracted oil droplets within the resulting crushed substance. Finally the extraction of the "liquid gold" is accomplished by pressing, percolation, or centrifugation [10].
The pressing process is the oldest and most common method of oil extraction by applying pressure to stacked mats, smeared with paste, that alternate with metal disks. The oil is then expressed through a central spike. On the other hand, the percolation process incorporates the use of a metal plate dipped into the mixed paste which in theory becomes wetted with oil, and not with oil mixed with water, when withdrawn. Otherwise, the centrifugation method uses high-speed centrifuges that extract the oil from the beaten paste through a fine screen. The advantages include the speed of the process, efficient and compact equipment/machinery, and low labor requirements.
The waste produced from olive-press processes consists of solid (paste) and liquid waste. This waste is used in different ways [11].
The diagram of the oil olives pressing products is presented below.

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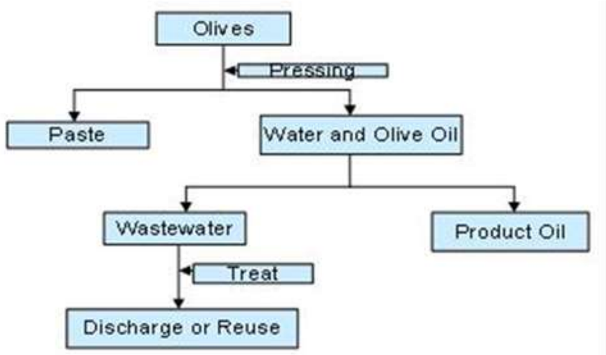


olive oil production process

Solid waste (paste) is usually used as fuel, fertilizer, herbicide, animal feed, road construction material, olive bricks, and worm breeding material.
The most exciting use is fuel due to the extremely high cost of energy sources around the world. In Syria, Jordan, and Turkey the primary use of pressing waste is fuel to heat households and power kilns if completely dried, the solid waste is pressed into logs for burning yielding extremely intense burning and an aromatic scent. Commercially sold charcoal consists in part of dried solid olive waste [12].
When it is used as a component of fertilizer and mulch, the olive waste should be mixed with soil and bark and should not be concentrated over the olive tree roots because the roots may burn. Usually, the olive waste is distributed around an orchard or farm and it serves as a natural herbicide, discouraging grass and weed growth. Some American farmers have reported the emergence of red clover where previously none existed. Red clover is a dynamic accumulator of nitrogen and phosphorous, and the presence of red clover (Trifolium pretense) is also an indicator of potassium [13].
This olive residue material is also a component of feed for animals such as cattle and poultry; however, goats and sheep eat it "straight" separating the edible portion from the woody parts. On the other hand, Americans use tons of olive waste mixed with bitumen as a component of road construction material [14].
The olive-press wastewater is discharged directly into municipal sewage systems or to irrigate the agricultural areas in Mediterranean regions [15]. Biochemical (biological) and chemical oxygen demand (BOD, COD) of this waste may be as high as 100 and 400 g/l respectively. The organic fraction includes some sugars, tannins, polyphenols, polyalcohols, pectins, and lipids. This wastewater is highly toxic because of its high content of phenolic compounds [16]. The phenolic content of this waste causes phytotoxic and antimicrobial effects. Many phenolic and aromatic compounds have been detected in this waste. The dark color of this waste is caused by polyphenols. This color depends on the age and type of olive processed and also the type of technology used [17].
The presence of this wastewater in rivers decreases the dissolved oxygen content but increases the organic matter and K, Fe, Zn, and Mn contents. Olive oil mill wastewater pollution also decreases the fish population [18]. (Cabrera et al., 1984). Many studies have been carried out to degrade and use this wastewater [19-20].
Conventional wastewater treatment methods are relatively ineffective for removing these kinds of pollutants. New methods for the treatment of this wastewater must be developed, in particular, by using microorganisms [21], and radiation [22-23]. and chemical treatments [24].
The goal of this study is to examine the radiation degradation of the phenol and polyphone toxic compound exited in olive wastewater and to compare this technique with others such as biodegradation or chemical coagulation to reuse this wastewater.

## 2 Experimental Protocol:

Olive wastewater samples for this study were obtained from different olive press mills in the Damascus and Draa areas. The parameters COD, BOD, TSS, pH, and alkalinity of the collected samples were determined according to the standard Methods [25]. The preparation procedure of olive-press wastewater samples was done by the method of Antolovich [26]. Olive-press wastewater sample preparation was done by:
1- Chemical coagulation
As the olive-press wastewater contains a reasonable amount of sugar and protein, it must separate compounds before any treatments, and then centrifuge the suspension to take the olive wastewater samples.

To separate the suspended materials from the water it was used:
1- carrez solution:
$Zn(CH_3COO)_2.2H_2O + CH_3COOH$
Then $K_4Fe(CN_6).3H_2O$
2- Dilution and filtration
- 1/5 sample/distill water
- 1/10 sample/distill water

Then polyphenols and other phenolic compounds were extracted by n-hexane and dichloromethane.
Qualitative and quantitative analysis of the polyphenols and by-products was performed using UV/Vis Spectrophotometry (Shimadzu UV-Vis, A-120) and GC-mass (Shimadzu-QP5050 A) with OPTIMA 5-Accent column with He gas as mobile phase and the Electron impact as detector. These techniques were used to determine the total content of organic compounds and the effect of gamma radiation on samples [27]. The gamma irradiation of olive wastewater samples was conducted using a $^{60}Co$-gamma unit, Russian type 140 kCi, at a dose

rate of 30 kGy/h. The dose and dose rates were determined using a modified Fricke dosimeter [28]. Samples were irradiated at 20 $^{o}$C with doses ranging from 0 to 20 kGy. The irradiation of samples was conducted using a 20 mL sample placed in a sealed glass tube.

Table 1 shows the typical characteristic parameters of olive wastewater samples. All chemicals of analytical grade were purchased from Sigma.

**Table 1:** The analysis of fresh olive press wastewater:

| | |
|---|---|
| pH 6.0 | |
| Water Content 90 % | Polyalcohols 1.5 % |
| Organic and Volatile | Protein3.5 % |
| Material 15% | Polyphenols 17 % |
| Mineral Solids 2% | Suspended Solids 40 g/l |
| Residual Oil 1% | $BOD_5$ 70 g/l |
| Total Sugars 6 % | COD 300 g/l |

## 3 Results and Discussion

The radiation effect was noticed for COD and BOD5 of samples. The results showed that these parameters decreased from 300 g/l and 70 g/l to 30 g/l and 10 g/l respectively.

To obtain better knowledge of the effect of □-irradiation on poly-phenolic compounds, samples of chemical coagulation and filtration were irradiated and passed by UV-vis.

Figure 1 shows the UV spectrum. It can be noticed that the saturation plateau between 220-250 nm (wavelength) disappears after the radiation.

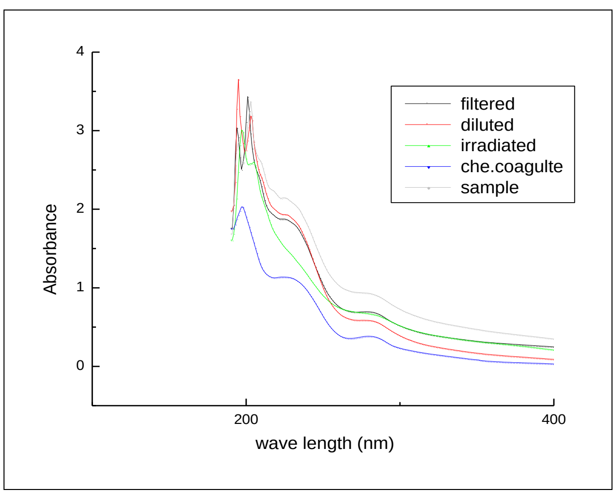


Fig.1. UV Spectrum of olive-press wastewater

Figure 2 represents the GC-mass chromatogram of the olive wastewater sample extracted by n-hexane before irradiation. This chromatogram shows the 22 picks that exist in the sample, but the important picks that appeared between 20 -30 min.

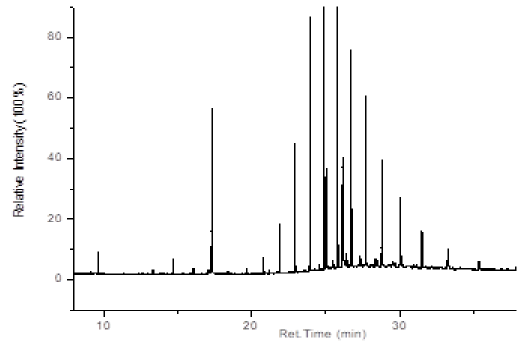


Fig. 2. GC-mass spectrum of olive waste water sample extracted by n-Hexane.

The GC-mass peaks exist in the olive waste water sample extracted by n-Hexane presents in table 2.

**Table 2 :** presents the names of the 22 picks that exist in the sample extracted by n-Hexane.

| Peak | Ret.Time | A/H | Conc. | Name |
|---|---|---|---|---|
| 1 | 9.617 | 1.72 | 0.82 | NEOPHYTADIENE |
| 2 | 14.675 | 1.59 | 0.53 | Phenol, 2,6-bis(1,1-dimethyl ethyl)-4-methyl- |
| 3 | 15.35 | 2.17 | 0.05 | Phenol, 4-(1,1,3,3-tetramethyl butyl) |
| 4 | 16.067 | 2.21 | 0.3 | Phenol, 4-(1,1,3,3-tetramethyl butyl)- |
| 5 | 17.292 | 1.94 | 7.33 | CYCLOHEXANE, 1,5-DIISOPROPYL-2,3-DIMETHYL- $$ |
| 6 | 19.692 | 1.69 | 0.18 | Heptadecane, 2,6,10,15-tetramethyl |
| 7 | 20.825 | 2.01 | 0.72 | Tetracosane (CAS) n-Tetracosane |
| 8 | 21.9 | 1.97 | 2.21 | Tetracosane (CAS) n-Tetracosane |
| 9 | 22.942 | 1.99 | 5.9 | Heneicosane $$ n-Heneicosane |
| 10 | 23.933 | 1.81 | 10.57 | Nonadecane |
| 11 | 24.883 | 1.9 | 12.1 | Octacosane (CAS) n-Octacosane |
| 12 | 25.058 | 1.94 | 4.46 | Phenol, 2,2'-methylenebis[6-(1,1-dimethylethyl)-4-methyl |
| 13 | 25.458 | 1.85 | 0.33 | Tetratetracontane (CAS) n-Tetratetracontane |
| 14 | 25.8 | 1.77 | 11.78 | Octacosane (CAS) n-Octacosane $$ |
| 15 | 26.133 | 2.38 | 5.84 | 1,2-Benzenedicarboxylic acid, diisooctyl ester |
| 16 | 26.708 | 2.22 | 11 | Pentacosane (CAS) n-Pentacosane |
| 17 | 27.7 | 2.15 | 8.31 | Pentacosane $$ n-Pentacosane |
| 18 | 28.8 | 2.75 | 6.6 | Nonacosane |

| | | | | |
|---|---|---|---|---|
| 19 | 30.058 | 3.15 | 4.96 | Tetratetracontane |
| 20 | 31.525 | 3.78 | 3.16 | Tetratetracontane |
| 21 | 33.275 | 3.9 | 1.67 | Tetratriacontane , n-Tetratriacontane |
| 22 | 35.4 | 6.61 | 1.18 | Tritetracontane |

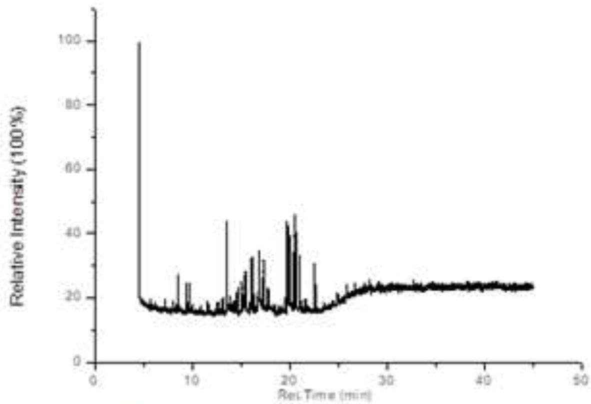

Fig. 3 GC-mass spectrum of olive waste water sample extracted by Dichloromethane (DCM).

Otherwise, figure 3 shows the GC-mass peaks that exist in the olive wastewater sample extracted by Dichloromethane (DCM). It can be noted that the 22 picks exist in the sample also, but the important picks appeared between 20 -30 min. Table 3 presents the names of the 22 picks that exist in the sample extracted by Dichloromethane.

It can be shown from Figure 4 the effect of gamma radiation on the main peaks of polyphenols in the samples extracted by h-hexane. In Figure 5 the main peaks of the sample extracted by dichloromethane can be seen.

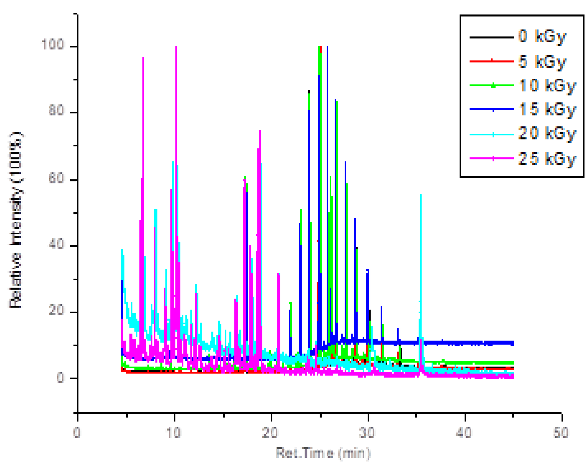

Fig. 4. NH comparison with 0, 5, 10 , 15, 20 and 25 kGy

It can show the effect of irradiation by the presence of by-products detected by measuring the organic acids and aliphatic compounds in the irradiated samples using HPLC.

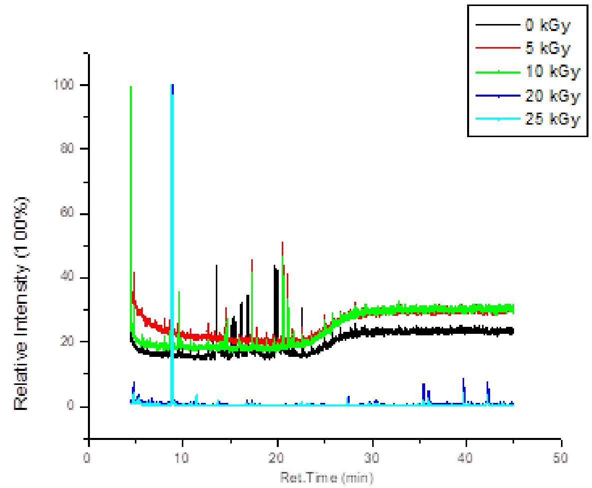

Fig. 5. DCM comarison with 0, 5 , 10, 20 and 25 kGy

A kinetic degradation of phenols and other pollutants compounds adapted from that of [29-30-31-32] was applied to explain the dates. The mechanism of the degradation of phenols was done according to [1-2-33]. The final products were determined by comparing HPLC elution times and UV spectra of irradiated solutions with the standard spectra.

A possible explanation of the decomposition reaction of polyphenols depends on the hydrated electrons that exist in the irradiated solution. They attack the polyphenols and through dissociative electron attachment, the hydroxyl ions are eliminated. The hydroxyl radicals will attack the aromatic ring and at low result in the formation of ydroxypolyphenols or aliening water molecule forming phenols. Then through a rather complex series of reactions and at higher phenols ring cleavage occurs and numerous aliphatic acids are formed [34].

The intermediate products, which may represent the stability stage compounds, are further decomposed as reported earlier [1-2-33]. Other substituted products, which are resistant to radiation, are still there and were determined as shown in the mechanism.

**Table 3.** presents the names of the 22 picks that exist in the sample extracted by Dichloromethane.

| Peak | Ret.Time | A/H | Conc. | Name |
|---|---|---|---|---|
| 1 | 8.575 | 3.3 | 4.46 | Phenylethyl Alcohol |
| 2 | 9.408 | 3.58 | 3.78 | Phenol, 3-ethyl- (CAS) m-Ethylphenol |
| 3 | 9.658 | 1.82 | 1.88 | NEOPHYTADIENE |
| 4 | 13.5 | 3.37 | 10.94 | 6-Octadecenal (spectrum disagrees) (CAS) |
| 5 | 14.592 | 1.59 | 0.82 | ACETYL-PRO-LEU-GLY-HYPRO-HYDROXYL |
| 6 | 14.642 | 1.88 | 1.42 | Hexahydro-1-oxa-cyclopropa[d]inden-2-one |
| 7 | 15.05 | 3.07 | 3.39 | Phenol, 2-methoxy-4-(methoxymethyl)- |
| 8 | 15.308 | 2.17 | 2.39 | 7-Oxabicyclo[4.1.0]heptane, 3-oxiranyl- |
| 9 | 15.483 | 2.26 | 2.71 | 2,6-Dimethoxybenzoquinone |
| 10 | 16.05 | 2.16 | 3.65 | Bicyclo[3.1.1]heptan-3-one, 2,6,6-trimethyl-, (1.alpha.,2.beta.,5.alpha.)- |
| 11 | 16.275 | 2.37 | 4.43 | 7-Oxabicyclo[4.1.0]heptane, 3-oxiranyl- |
| 12 | 16.858 | 2.99 | 5.78 | 2-Decyn-1-ol |
| 13 | 17.317 | 1.47 | 1.94 | Cyclohexane, 1-(cyclohexylmethyl)-2-methyl-, cis- |
| 14 | 17.783 | 5.3 | 3.85 | 3-Nonyn-2-ol (CAS) |
| 15 | 19.775 | 3.98 | 12.38 | Cyclopentaneacetaldehyde, 2-formyl-3-methyl-.alpha.-methylene- |
| 16 | 19.958 | 3.51 | 10.19 | .alpha.-Campholonic acid |
| 17 | 20.492 | 2.49 | 6.72 | Morpholine, 4-(1-cyclohexen-1-yl)- |
| 18 | 20.583 | 2.06 | 6.56 | 4-t-Butyl-2-(1-methyl-2-nitroethyl)cyclohexanone |
| 19 | 20.683 | 2.03 | 5.25 | 4-t-Butyl-2-(1-methyl-2-nitroethyl)cyclohexanone |
| 20 | 21.108 | 1.97 | 3.77 | 3-t-Butyl-oct-6-en-1-ol |
| 21 | 21.633 | 2.02 | 0.88 | 2,4,4-Trimethyl-1-pentyl methylphosphonofluoridate |
| 22 | 22.625 | 1.95 | 2.81 | Ethyl Oleate |

## 4 Conclusions

Through this study, It can be noticed that radioactive destruction represents one of the solutions to destroy harmful pollutants present in the water resulting from the olive industry, which can be recycled to be used to irrigate agricultural lands again.

The mechanism and speed of the destruction of these multicyclic organic compounds by irradiation is still the subject of multiple studies in many laboratories, especially when the destruction is carried out in the presence of auxiliary chemical ions contained in wastewater (negative and/or positive).

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