Terrestrial and Aquatic Environmental Toxicology ©2010 Global Science Books

Olive Oil Recovery Methods

Serap Saglik Aslan*

Department of Analytical Chemistry, Faculty of Pharmacy, Istanbul University, 34116 Beyazit, Istanbul, Turkey

Correspondence: * **serapsaglik@yahoo.com**, **ssaglik @istanbul.edu.tr**

ABSTRACT

Olive mill wastewater is formed during olive oil production and is also an important environmental problem in the Mediterranean area. The amount of oil and grease, which is the one of the parameters used for the characterization of this wastewater, cannot be determined easily using a standard method. This review focuses on the progress in olive oil recovery methods used for dealing with the environmental problem of olive mill wastewater. Studies have been conducted with the aim either to increase the efficiency of oil recovery using established methods or to develop novel methods. A number of methods and various reactives have been evaluated for these purposes.

Keywords: olive mill wastewater, olive oil industry, oil recovery

INTRODUCTION

Olive oil is a typical Mediterranean product, in terms of production and consumption. There are about 805 million olive trees in the world. 98% of these trees grow in the Mediterranean area which provides for 97% of the total olive production and 91% of world consumption (Gurbuz *et al.* 2004). As production volumes increase, the focus is increasingly directed at the by products of oil extraxtion; with growing competition, the recovery of the valuable olive oil found in the residues, and the reduction of disposal costs becomes ever more attractive from an economic perspective (Hruschka 2006).

Olive seeds are usually crushed and disposed of after processing the olive oil. The aqueous liquid wastes produced by the oil mills are dramed as sewage. The amount of liquid waste produced depends upon the milling process, ranging from about 50 kg of water per 100 kg of olives in a traditional batch mill to about 110 kg of water per 100 kg of olives in a continious process. The solid waste typically contains 6-8% oil. It may be treated first with solvents to extract the residual oil and the exhausted solid waste may then be compressed into briquettes and used as inefficient polluting heating fuel (Amat *et al.* 1999; Vitolo *et al.* 1999; El Hamouz *et al.* 2007).

Olive mill wastewater (OMW) generated during olive oil production causes a serious environmental problem in Mediterranean countries. Many attempts have been made to reduce this pollution effect of OMW. The lipids in OMW along with other organic compounds exert a serious pollution effect. Therefore recovering the oily phase from OMW is two-fold essential: on the one hand pollution is reduced and on the other hand an economically important by-product is regained (Saglik *et al.* 2002).

In the following sections the information available in the literature both on the laboratory studies and industrial scale processes for recovery of olive oil from olive mill wastewater are reviewed.

RECOVERY METHODS

In the standard method, a filter consisting of a muslin cloth disk overlaid with filter paper is prepared. 100 mL diatomaceous silica suspension (12%) are passed through the filter on a Buchner funnel and washed with 500 mL distilled water applying a vacuum. Then a 50 mL sample is acidified to pH 2 with HCl (1+1) and filtered through the prepared system. The residue on the filter paper is dried, fitted into a cartridge and extracted in a Soxhlet apparatus using petroleum ether. The solvent is distilled off and the residue is left to stand in a desiccator containing P_2O_5 and then weighed (Standard Methods 1989).

Domínguez *et al*. (1994) used enzymatic treatment to enhance oil recovery from olive, avocado or coconut pastes with excellent results both on a laboratory and industrial scale (olive) obtaining the oil in shorter times and increasing the capacity of the equipment. This treatment is tried for the extraction of oil and protein from oilseeds on a laboratory scale. Considering that two thirds of the total fat and oil production is supplied by oilseeds, this is a promising field for biotechnological applications.

Birch *et al*. (1998) took mixed liquor samples (50 mL; 1.5% TS w/w) via a sample port at selected times from a stirred 18 L anaerobic batch digester with the method of Broughton *et al.* (1998) following the addition of emulsified tallow and are processed immediately. Lipid material is extracted from the samples with the method of Bligh and Dyer (1959) as follows. Subsamples are dispensed into a separating funnel, and chloroform/methanol (1:2 v/v, 15 mL) is added. Aqueous samples of stirred digester contents on aqueous suspensions of the pure lipid materials are mixed with buffer and Sudan III and vortexed. The mixture is incubated for 10 min at 60°C, cooled on ice and centrifuged. The supernatant is decanted, the washed pellet is redissolved in hexane heated to 60°C to extract the incorporated dye species and recentrifuged. Nonspecific dye uptake by the mixed liquor in the absence of added lipid waste causes a colorimetric response equivalent to 3.4 g/L or 14% (w/w) of the sludge solids content. When the digester contents are spiked with 10.0 g/L emulsified tallow, the dye binding lipid assay gives 11.0 ± 0.3 g/L lipid after the respective background interference (3.4 g/L) is subtracted.

Vitolo *et al.* (1999) examined effluents (vegetation waters and olive husks) from both a traditional extraction process and a continuous extraction process. From preliminary examinations it is noted that the husks derived from the two different processes do not differ significantly in their heating values or ash content and composition. Since the vegetation waters derived from the traditional process have a substantially higher level of pollutants, it is decided

Received: 18 May, 2009. Accepted: 27 June, 2009.

Techniques Paper

to study the effluents from this process. The vegetation waters from the continuous process are studied for comparison. Two samples of vegetation water and one sample of olive husk from oil mills in the Tuscany area are then examined. Vegetation water sample A and the olive husk sample are produced in a traditional batch mill, while vegetation water sample B is collected from a continuous mill. These samples are representative of the waste products obtained in a typical week of production for the two mills. The olive husk sample from the traditional batch mill is ground before use, giving 80% of the weight of the sample a particle size of under 600 pm, and this portion is used in the tests. The vegetation water are concentrated by evaporation in a rotating evaporator under either vacuum or atmospheric pressure. Each test is conducted with a 500 mL sample; the vapours are condensed and collected periodically as the evaporation proceeded.

Saglik *et al.* (2002) investigated the Standard Method (*Method A*, 1989) and six methods. In the first method (*Method B*), 0.4 g calcium oxide and 0.4 mL distilled water are mixed. After 15 min, 50 ml olive mill wastewater are added gradually and the mixture is mechanically stirred, first slowly for 10 min, then quickly for 15 min. Thereupon the mixture is acidified to pH 2 with HCl (1+1) and worked up according to *Method A*. In the second method (*Method C*), the first method is applied without using diatomaceous-silica. In the third method (*Method D*), the second method is applied without using filter paper. In the fourth method (*Method E*), is applied without acidification. In the fifth method (*Method F*), 0.4 g calcium oxide and 0.4 mL distilled water are mixed. After 15 min, 50 mL olive mill wastewater are added in portions. Then air is passed through the mixture for 10 min to 14 h using a simple aquarium pump and the pH values are determined all along. In the sixth method (*Method G*), the fifth method is applied separately by passing air through the mixtures for 30 min, 1, 2 and 3 h, respectively. Then the mixtures are filtered through a muslin cloth disk on a *Buchner* funnel. The residues are dried on muslin, fitted into a cartridge and worked up according to the *Method A*. As a result, *Method G* by passing the air for 1 h is proposed for the recovery of the lipids from OMW. The range of percental recoveries is between 70.6 and 96.4% and the average value is 86.2%.

Hruschka (2006) investigated the valuable oil recovery process. Although in contrast to the 3-phase centrifugal process the 2-phase process does not require the addition of diluting water during the actual extraction, subsequent processing does require water as in any other process. In fact, around 200 litres are needed for each tonne of olives processed. Apart from water, this mixed phase also contains olive oil and extremely fine particles, primarily sediment. If it is treated in parallel with the actual oil extraction, the oil it contains can also be recovered and its quality is practically identical to that of the originally extracted oil. In fact, up to a half a kilo of oil can be recovered from every 100 kg of olives processed. This is made possible by the inclusion of an additional 3-phase decanter, which separates both the extremely fine particles as well as the residual olive oil from the primarily sediment stream in a single process step. However, in order to recover the oil in economical quantities, and to obtain residual solids that are as dry as possible, the employed 3-phase decanter must exhibit an extremely high level of separation efficiency. The RCD 3-phase decanter, which has proven its separation efficiency in many areas of application, was therefore selected for use in the primarily sediment process.

El-Hamouz *et al*. (2007) extracted the oil from fresh olive waste using the Soxhlet method. Solid olive pips (180 g) are placed in a thimble. Solvent (80 mL, lead-free gasoline, ether or acetone) is refluxed over the solid waste for 10 min. The liquid solution, including recovered oil, is then taken and distilled to isolate the recovered oil. In each distillation, the evaporated solvent is recovered in a cooled container, with percentage loss no more than 2%. To achieve highly concentrated oil solutions, used batches of solid pips are replaced with fresh batches. The fresh batches are then extracted using the same residual solvent. The procedure is repeated to up six times, giving concentrated oil, before the final separation. More than 5% of solid waste could be recovered as oil. Maximum oil recovery percentage values are 4.4, 3.7 and 5.4% using ether, acetone and unleaded gasoline, respectively.

Sharma *et al*. (2007) investigated the enzymatic pretreatments to enhance oil recovery in olive oil extraction process. The laboratory scale mechanical method consisting of crushing of olive fruits in fruit mill, malaxation in oil press for 60 min followed by pressing in hydraulic press at 15 ton/m^2 for 30 min and finally centrifugation of oil-water mixture (5000 rpm for 20 min) to separate oil is optimized.

CONCLUSION

The principal focus of this review was on olive oil recovery from OMW. Numerous studies have been maden on this subject appearing in the recent years. The established methods were investigated by several research groups with the aim to increase the efficiency and environmental acceptance of olive oil recovery. According to the literature, the efficiency of olive oil recovery using different methods is similar. Thus, it will be not easy to identify a method of choice. However, it is necessary that other important factors are, also taken into consideration. In a wide range of recovery methods show the efficiency of olive oil recovery which is better than that using older methods. That processes may be tested in pilot areas and then validated with regards to environmental and economical advantages in the olive oil industry.

REFERENCES

Amat AM, Arques A, Miranda M (1999) *p*-Coumaric acid photodegradation with solar light, using a 2,4,6-triphenylpyrylium salt as photosensitizer: a comparison with other oxidationmethods. *Applied Catalysis B: Environmental* **23**, 205-214

Birch JB, Beyer R, Fraser NS, Thiele JH (1998) Methods of analysis of esterified and free long-chain fatty acids in high-lipid food processing wastes. *Journal of Agricultural and Food Chemistry* **46**, 5332-5337

Bligh EG, Dyer WJ (1959) A rapid method of total lipid extraction and purification. *Canadian Journal of Biochemistry and Physiology* **37**, 911-917

Broughton MJ, Thiele JH, Birch EJ, Cohen A (1998) Anaerobic batch digestion of sheep tallow. *Water Research* **32**, 1423-1428

Domínguez H, Nunez MJ, Lema JM (1994) Enzymatic pretreatment to enhance oil extraction from fruits and oilseeds: a review. *Food Chemistry* **49**, 271-286

El-Hamouz A, Hilal HS, Nassar N, Mardawi Z (2007) Solid olive waste in environmental cleanup: Oil recovery and carbon production for water purification. *Journal of Environmental Management* **84**, 83-92

Gurbuz S, Kiran-Ciliz N, Yenigun O (2004) Cleaner production implementation through process modifications for selected SMEs in Turkish olive oil production. *Journal of Cleaner Production* **12**, 613-621

Hruschka S (2006) Valuable oil recovery: process improves extraction and lowers costs. *Filtration-Separation* **1/2**, 38-39

Saglik S, Ersoy L, Imre S (2002) Oil recovery from lime-treatment wastewater of olive mills. *European Journal of Lipid Science and Technology* **104**, 212-215

Sharma R, Sharma PC (2007) Optimization of enzymatic pretreatments for maximazing olive oil recovery. *Journal of Scientific and Industrial Research* **66**, 52-55

Standard Methods, For the Examination of Water and Wastewater (17th Edn) (1989), Oil and Grease (5520), American Public Health Association, Washington DC (USA), pp 5-45, 5-46

Vitolo S, Petarca L, Bresci B (1999) Treatment of olive oil industry wastes. *Bioresource Technology* **67**, 129-137