Olive Mill Waste Recent Advances for Sustainable Management



Edited by Charis M. Galanakis



Olive Mill Waste

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Editor

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Preface

The cultivation of olives and the production of olive oil have deep roots in the history of Mediterranean region. This tradition represents a very important asset for many countries, not only in terms of culture and health, but also in terms of wealth. However, olive oil production is accompanied with the generation of huge amounts of by-products and waste that leave a congested environmental footprint. These materials are undesirable for the olive oil industry in terms of sustainability and environmental impact, but perhaps more important in view of high disposal costs. Therefore, they have been considered as a matter of minimization, prevention, and treatment for as many decades as olive oil industrial production exists. Indeed, the proposed treatment methods and the respective literature and references are endless. Despite this fact, olive oil industry remains unsustainable, with few opposite examples that confirm this rule of thumb.

Why is this happening? Is it a matter of inadequate treatment technologies or is it about cost? Olive oil is a sector challenged by many directions. Consumers demand extra virgin olive oils of ultra-high quality, product's final price varies a lot from time to time, and local authorities demand from production units to reduce their environmental impact. Under these conditions, even cheap solutions that promise the total treatment of olive mill waste may collapse financially olive oil industries. Consequently, most treatment solutions have been rejected in practice due to industries' denial that claim to close down production and society's tolerance that delays the enforcement of environmental legislations implementation. Can olive oil industries overpass environmental legislations forever? Does this consideration fall in the frame of the modern bioeconomy? Can they adapt any alternative strategies? The urgent need for sustainability within olive oil industry has turned the interest of researchers and professionals to investigate the management of olive mill waste with another perspective. This resource contains valuable components, such as water, organic compounds, and a wide range of nutrients that could be recycled. The prospect of recycling ingredients from olive mill waste is a story that started few decades ago. For instance, solvent extraction had been applied to recover oil from olive kernel, which is one of the byproducts derived from olive oil production. Olive kernel is considered an established commodity similar to olive fruit, whereas scientists focus on the recovery of polyphenols, the reutilization of irrigation water, as well as the production of compost to be used as soil amendment. Subsequently, there is a need for a new guide covering the latest developments in this particular direction.

Following this trend, the current book covers the most recent advances of olive mill waste management in the name of sustainability. It aspires to fill in the gap of transferring knowledge between academia and industry by describing in details the viable industrial applications and scenarios. It highlights success stories and solutions that are already applied in some olive oil industries, whereas it explores the advantages, disadvantages, and real potentiality of relevant processes and products in the market. The ultimate goal is to inspire scientific community and producers that aspire to develop real commercialized applications.

The book consists of 12 chapters. Chapter 1 discusses olive oil production, its environmental effects, and the current sustainability challenges of the sector. Chapter 2 introduces the current advisable practices for the sustainable development of olive oil industry, focusing on olive mill waste, and two soil remediation methods, applied within the framework of European project. Chapter 3 deals with the industrial valorization of residues from olive oil industry within the integrated concept of biorefinery. Rest chapters focus on more specific applications. For instance, Chapter 4 presents the possibilities and

alternative strategies to recover energy from olive oil processing residues. In Chapter 5, the benefits and risks of using olive mill waste as a soil amendment are discussed and recommendations on their proper application are provided, too. Chapter 6 describes industrial case studies for the detoxification of olive mill wastewater using Fenton oxidation process followed by biological processes for energy and compost production. Chapter 7 presents an integrated and commercialized approach for the treatment of olive mill wastewater and solid residue using only biological treatments (i.e., trickling filters, constructed wetlands, and composting). Chapter 8 deals with the cocomposting of olive mill waste as well as the design and operation of two case (pilot-scale and full-scale) studies. Chapter 9 reviews the use of the different olive mill by-products in phytoremediation strategies of contaminated soils. Chapter 10 denotes the different available technologies for the recovery of bioactive compounds from olive oil process. The applications of membrane processes for this purpose are thoroughly discussed, whereas detailed information for the current patented and commercialized methodologies are provided. Finally, Chapters 11 and 12 describe available and potential applications of compounds recovered from olive mill waste in food products and cosmetics, respectively.

Conclusively, the ultimate goal of the book is to provide a handbook for all the professionals and producers activated in the field, trying to optimize olive oil industry performance and reduce its environmental impact. It concerns chemical engineers and technologists working in the olive oil and food industry as well as researchers, specialists, and new product developers working in the edge of food and environmental sectors. It could be used as a textbook and/or ancillary reading in graduates and postgraduate level, and multidiscipline courses dealing with agricultural science, food and environmental technology, sustainability, and chemical engineering.

I would like to take this opportunity to thank all the authors and contributors of the book for their collaboration and high quality work in bringing together different topics and sustainable approaches in a comprehensive text. I consider myself fortunate to have had the opportunity to collaborate fruitfully with so many knowledgeable colleagues from Cyprus, Greece, Italy, Kazakhstan, Morocco, Portugal, Spain, and Tunisia. Their acceptance of book's concept and their dedication to editorial guidelines is highly appreciated. I would also like to thank the acquisition editor Nancy Maragioglio for our collaboration in this project and all the team of Elsevier, particularly Billie Jean Fernandez for her assistance during editing and Caroline Johnson during production. I would also like to acknowledge the support of Food Waste Recovery Group of ISEKI Food Association, which is the most relevant group worldwide in the particular field.

Last but not least, a message for the readers. In a collaborative project of this size, it is impossible for it not to contain errors. If you find errors or have any objections to its content, please do not hesitate to contact me.

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OLIVE OIL PRODUCTION SECTOR: ENVIRONMENTAL EFFECTS AND SUSTAINABILITY CHALLENGES

1

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1.1 INTRODUCTION

Olive oil is produced from olive trees, each olive tree yielding between 15 and 40 kg of olives per year. Worldwide olive oil production for the year 2002 was about 2.5 million tons produced from approximately 750 million productive olive trees, the majority of which are in the Mediterranean region. In particular, there are about 25,000 olive mills worldwide. Mediterranean countries alone produce 97% of the total olive oil production, while European Union countries produce 80–84%. The biggest olive oil-producing countries are Spain, Italy, Greece, and Turkey (~ 0.9 , 0.6, 0.4, and 0.2 million tons in 2002, respectively), followed by Tunisia, Portugal, Morocco, and Algeria. Outside the Mediterranean basin, olives are cultivated in the Middle East, the USA, Argentina, and Australia (Paraskeva and Diamadopoulos, 2006).

Olive oil production tends to increase over the last decades as a valuable source of antioxidants and essential fatty acids in the human diet and constitutes one of the most important dietary trends worldwide. The olive oil extraction systems could be classified in two main categories: traditional pressing process (Kapellakis et al., 2008), used for many centuries with minor modifications, and centrifugal processes, including two centrifugation systems, called three- and two-phase systems. The extraction of olive oil generates huge quantities of wastes that may have a great impact on land and water environments because of their high phytotoxicity. The most pollutant and phytotoxic wastes are known as olive mill waste (OMW). During the olive oil production, almost all the phenolic content of the olive fruit (~98%) remains in the olive mill by-products (Rodis et al., 2002). Besides being a serious environmental problem, OMW represents today a precious resource of useful compounds for recovery and valorization purposes (Hamza and Sayadi, 2015; Skaltsounis et al., 2015; Chiaiese et al., 2011).

1.2 OLIVE FRUIT PROCESSING AND OLIVE OIL PRODUCTION TECHNOLOGIES

Olive oil extraction involves different processes, such as leaf removal, olive washing, grinding, beating, and separation of the oil. The amount and physicochemical properties of the produced wastes and effluents depend on the method used for the extraction. Olive oil is extracted directly from the fresh fruit of olive tree (*Olea europaea* L.) using only mechanical methods, in order to maintain its natural organoleptic characteristics according to the European Commission Regulation No. 1513/2001 (EC, 2001). Olive fruits must be processed as quickly as possible after harvesting to minimize oxidation and preserve low acidity. All the operations of the olive mill can influence on olive oil quality (Gimeno et al., 2002; Kiritsakis et al., 1998). The mechanical processes used to extract olive oil from olive fruit include olives crushing, malaxation of resulting paste, and separation of the oily phase by pressure or centrifugation. In the latest one, three different systems are commonly used: the traditional discontinuous press process, the three-phase, and the two-phase decanter centrifuge methods (Fig. 1.1).



(A) Traditional process. (B) Modern process.

1.2.1 TRADITIONAL DISCONTINUOUS PRESS SYSTEM

Traditional extraction press is still used in some mills. After grinding olives in stone mills, the paste is spread on fiber diaphragms (which are stacked on top of each other) and then placed into the press (Fig. 1.2A). Hydraulic pressure is applied on the disks, thus compacting the solid phase of the olive paste and percolating the liquid phases (oil and vegetation water). To facilitate separation of the liquid phases, water is run down the sides of the disks to increase percolation speed. Traditionally the disks were made of hemp or coconut fiber, but in modern times they are made of synthetic fibers, allowing easier cleaning and maintenance (Kapellakis et al., 2008).

This process generates a solid fraction called olive husk (or kernel), an emulsion containing the olive oil and a water phase. The olive oil is finally separated from the remaining wastewater by decantation or vertical centrifugation. The traditional process is reputed to produce a high quality olive oil. However, many precautions should be taken in consideration, for example, a proper cleaning of the disks and a rapid treatment of the paste to avoid fermentation. The latest generates unwanted flavors that lead to the production of defected olive oil. Nowadays, due to the need to process large amounts of olives and to obtain higher yields of olive oil, the evolution of the oil extraction process has led to the replacement of traditional pressure mills with modern continuous centrifugal extraction.

1.2.2 CONTINUOUS CENTRIFUGAL EXTRACTIONS

After washing, crushing, and malaxation steps, the mechanical oil extraction is mainly carried out using a continuous process based on centrifugation with a decanter. The decanter centrifuge is designed with





(A) Press system. (B) Decanter (two-phases). (C) Decanter (three-phases). Decanters photos courtesy of Flottweg Separation Technology, Germany flottweg.com.

screw conveyer and rotating bowl, allowing to process large amounts of olives in short time (Catalano et al., 2003). Actually two types of centrifugal decanters are currently used: two-phase centrifugation and three-phase centrifugation (Fig. 1.2B and C).

The continuous three-phase centrifugation process was introduced during the 1970s, in order to increase processing capacity and extraction yield and reduce labor (Demicheli and Bontoux, 1997). During the three-phase process, an addition of hot water is required to wash the oil. The process yields three phases: oil phase, solid residue: olive cake (olive pulp and stones) and the olive mill wastewater (OMWW). Solid residue is separated from the other two phases in the decanter. The liquid phases are subsequently submitted to a vertical centrifugation in order to separate the olive oil from OMWW.

A disadvantage of this process is the large amounts of produced wastewaters that are due to the high consumption of water, 1.25–1.75 times more water than press extraction (Vlyssides et al., 2004). The failure to develop a suitable end-of-pipe wastewater treatment technology challenged technology manufacturers to develop the two-phase process. The latest uses only washing water and delivers oil (liquid phase) and a very wet substrate (semisolid phase called wet olive pomace or two-phase OMW) using a more effective centrifugation technology (Vlyssides et al., 2004). This process has reduced environmental impact due to the lower water demands and waste amounts produced, but it requires extra energy for drying prior olive kernel oil extraction. As the olive mills are becoming bigger, the chances of automation are also increasing, reflecting an improvement in the olive oil yield and quality. Therefore, a detailed knowledge of the whole extraction process is crucial in order to design the appropriate control strategies (Bordons and Nunez-Reyes, 2008).

1.2.3 FEEDING, LEAF REMOVAL, AND WASHING

The first step in the olive oil extraction process is olive fruit cleaning and removal of stems, leaves, twigs, and other debris left with the olives. Washing is also aiming to remove pesticides and dirt. Light contaminants are removed by a heavy air flow and heavy objects sinks in the water bath. Olive washing in closed loop systems is a critical control point at the olive mill due to microbiological cross-contamination and fruit physical damage. Furthermore, when the olives were short-term stored before oil extraction, sensory attributes of virgin olive oil (VOO) diminished due to changes in phenolic and lipoxygenase derived volatile compounds (Vichi et al., 2015).

1.2.4 CRUSHING

Crushing step aims to break down the cellular membranes of olive fruits and thus release small drops of oil from the vacuoles (Rodis et al., 2002). This operation produces a mixture of two distinct liquid phases (raw oil and water) and an extremely heterogeneous solid phase (pit, skin, and pulp fragments). Crushing can be considered a critical point affecting the quality of the produced olive oil, especially in terms of phenolic content and volatile compounds (Servili et al., 2015). The constitutive parts of olive fruit are pulp, stone, and seed. The pulp is rich in phenolic compounds while the seed contains large amount of endogenous oxidoreductases and low amounts of phenols (Servili et al., 2004, 2007). Two enzymes, polyphenol oxidase and peroxidase, are highly concentrated in the olive kernel. Thus, crushing operation allows a direct contact of the peroxidase and polyphenoloxidase (POD) enzymes with phenolic compounds and induces their oxidation, which results in lower phenolic concentration in the oil. At this phase, the main hydrophilic phenols of VOO (e.g., secoiridoid aglycons) are generated



FIGURE 1.3 Types of Crushers (A) Stone Miller. (B) Disc Crusher. (C) Hammer Miller.

Photos courtesy of Alfa Laval, Italy alfalaval.com.

at the hydrolysis of oleuropein, demethyloleuropein, and ligstroside, and as catalyzed by endogenous β -glucosidases (Servili, 2014). The control of the enzymatic activity during the crushing step is a good strategy to maintain a high phenolic concentration in the resulting olive oil. For this purpose, mild seed crushing methods that reduce seed tissue degradation are used to limit the release of POD in the paste and decrease the rate of oxidation of hydrophilic phenolic compounds (Servili et al., 2007, 2015). Mechanical extraction from destoned olives has also been proposed to avoid enzymatic reaction catalyzed by POD, resulting in higher oxidative stability and nutritional value of the obtained olive oil (Del Caro et al., 2006). Moreover, oils extracted from destoned olives have a better sensory profile than the oils obtained from the traditional milling of entire fruits, although olive destoning lowers the oil yield (Ben Mansour et al., 2015). In another study aiming the evaluation of functional phytochemicals in destoned VOO, the oils showed higher contents of phenolic compounds, tocopherols, and aromas, whereas a potentially higher stability and shelf-life was reported (Ranalli et al., 2009).

On the other hand, olive seed oil is highly marketable in cosmetics and pharmaceutical industries while the destoned olive pomace containing a high phenolic concentration and high-value monounsaturated fatty acids, can be used in animal feeding (Servili et al., 2015). Crushing is generally carried out using a traditional stone mill or using hammer or disk crushers (Fig. 1.3). Hammer crushers produced oils with greater amounts of phenolic compounds as compared to stone crushers (Leone et al., 2015). Concerning the olive oil quality, Leone et al. (2016) reported that a flash thermal treatment of olive paste after crushing improves the phenolic and volatile profile of the oil significantly as compared to the traditional process. Since destoning process has led to better olive oil quality, destoner machines have been developed. The destoner consists of a cylindrical perforated grill (Amirante et al., 2006). The current trend in the olive oil market is the production of high quality monovarietal oils or mixing with the most widespread olives. The cocrushing of Picual variety with a Galician (Spain) local variety (80:20) was shown to improve the sensory and health properties of Picual extra VOO (Reboredo-Rodriguez et al., 2015).

1.2.5 MALAXATION

Malaxation consists of a slow and continuous kneading of the olive paste in order to facilitate the cohesion of small oil droplets obtained during crushing step, leading to separation of the oil from the water phase. This step is essential to achieve high yields of extraction. Several researchers have investigated the effect of malaxation parameters on the quality of produced oil. Malaxation time and temperatures found to affect greatly the olive oil quality especially the aroma and phenolic profiles (Jiménez et al., 2014; Reboredo-Rodríguez et al., 2014). Aliakbarian et al. (2008) demonstrated that increasing the malaxation time from 90 to 150 min decreased the nutritional quality of olive oil, mainly due to the increased oxidation of phenolics. In order to compromise quality and yield of olive oil, a low malaxation temperature and a process time between 30 and 45 min are recommended (Clodoveo et al., 2014). The old type of malaxation machines are designed with a stainless steel grill closure that was reported to cause a loss of the oil's phenolic and volatile compounds, due to the wholes of the upper grill (Amirante et al., 2006). In fact, the higher presence of O₂ during malaxation activate the POD and PPO enzymes responsible for the oxidation of phenolic compounds and loss of flavors (Tamborrino et al., 2014). A hermetic sealing improves the heat transfer leading to a reduction of the malaxation time and lower loss of volatiles and perfect control of the atmosphere contacting the paste (Leone et al., 2014). Besides, an innovative system has been developed to monitor the oxygen concentration during malaxation (Catania et al., 2013, 2016). At this case, the composition of the obtained olive oil in volatile compounds was enhanced by blowing pure oxygen in the headspace of the machine to modify the atmosphere. Recently, Catania et al. (2016) have developed a system to control the atmosphere in the headspace of the malaxation machine and improve the fatty acid composition of olive oil. The same group has developed a software that allows the acquisition and recording of the oxygen concentration in the headspace of the malaxation machine. Using N₂ during malaxation extended induction time, raised phenolic and tocopherol contents, leading to a strong antioxidant potential of oils (Yorulmaz et al., 2011).

The malaxation efficiency can also be improved by the addition of coadjuvants to promote the breakage of oil/water emulsions and consequently increase the recovery yield of the oil (Espinola et al., 2015; Guermazi et al., 2015; Sadkaoui et al., 2015). The list of studied coadjuvant consists of enzymes, talc, vegetable fiber, calcium carbonate, and salt. Researchers reported an increase of oil yield by the addition of natural microcrystalline talc during olive processing. In addition, an increase of oil stability due to the increase of phenolic content and tocopherols was denoted (Espinola et al., 2015; Caponio et al., 2015, 2016). The micronized natural talc is authorized by the European Commission and can be used during malaxation step (Caponio et al., 2016). The utilization of solid carbon dioxide (CO_2) for the extraction of extra-VOO has been proposed, too (Zinnai et al., 2015). For instance, the addition of cryogen to the olives during premilling phase greatly increased the extraction yield (ranging from 1% to 21%) improving olive oil quality and increasing its shelf-life (Zinnai et al., 2015).

Another approach is the addition of pectolytic, hemicellulolytic, and cellulolytic enzymes. The addition of these enzymes enables the breakdown of cell wall structure of olives and reduced the complexation of hydrophilic phenols with polysaccharides (Aliakbarian et al., 2008). More free phenolic compounds are consequently released into the olive oil depending on the used enzyme formulation (De Faveri et al., 2008). The modification of the pH of malaxation had also an influence on the produced olive oil. Aliakbarian et al. (2009) assessed the feasibility of increasing the free phenols in the olive oil paste by simultaneous addition of citric acid during the malaxation step and control of the kneading time. An increase of phenolic compounds in the olive oil was correlated to a better hydrolyze of pectic polysaccharides, cellulosic, and hemicellulosic fractions in olive pulp which allowed better release of phenolics from the paste to the oil.

1.2.6 HORIZONTAL CENTRIFUGATION

The centrifugation process is based on the density differences of olive paste components (olive oil, water, and insoluble solids). Decanters are constituted of a cylindrical conical bowl drum where a screw



(A) Vertical centrifuge system and (B) Hydrocyclone prototype (Altieri et al., 2015) used for oil clarification.

feeder is rotating at differential speed (Fig. 1.4B) (Skaltsounis et al., 2015). Separation is conducted due to the centrifugal force developed in the drum. Olive paste could be treated either by two-phase centrifugal decanter or a three-phase centrifugal decanter. In the two-phase decanter, the product is separated into a liquid phase (oil) and a solid phase (kernel fragments, pulp, and vegetable water). In the three-phase centrifugal decanter the product is separated into a light liquid phase (oil), into a heavy liquid phase (water), and into a solid phase (kernel fragments and pulp). In both two- and three-phases decanter centrifuges, the oil phase is discharged by gravity. In three-phase decanter, the water phase is discharged using a centripetal or gravity pump. The solid phase is discharged in the drum conical terminal of the decanter after being pushed by the screw feeder. The two-phase process requires no dilution during the malaxation step. However, in the three-phase process, large amounts of water are added to the paste. After malaxation, the olive oil is either completely free or in the form of small droplets inside microgels, or emulsified in the aqueous phase (Clodoveo, 2012). Adding water during the centrifugation improved the release of the oil fraction locked in the microgels (Clodoveo, 2012).

Since the olive oil extraction by decanter centrifuge is greatly dependent on the paste rheological properties (water content, variety, and temperature) (Altieri et al., 2013), it is crucial to control the process in order to optimize the extraction yield while preserving a high quality of oil. Recent developments include the design of an automatic system for the decanter able to adjust the machine operating parameters according to the olive paste characteristics and guarantee a constant feed to the decanter centrifuge even in the presence of physical changes in the olive paste (Altieri et al., 2013, 2014).

1.2.7 OIL CLARIFICATION

Oil clarification is the final cleaning step of olive oil. It aims to separate the residual water and impurities existing in the extracted oil from the decanter. This operation is necessary to avoid fermentation, hydrolysis, and oxidation reactions causing alteration of the sensory properties of olive oil (Baiano et al., 2014). The final purification step is generally made by a filtration or vertical centrifugation (Fig. 1.4A). Filtration is the most common technique used to clear the oil. A new processing arrangement was proposed and tested by Guerrini et al. (2015). It consists of the insertion of a steel prefilter into the system, which retains part of the suspension. Consequently, the plate filter-press retains only residual solids and water. The plate filter-press with the added prefilter was able to process about 1.8 times the amount of oil normally processed in a batch (Guerrini et al., 2015).

Vertical centrifugation contributes to a significant increase of dissolved oxygen concentration in oil and accelerates its oxidation. In order to reduce the oxygenation rate of olive oil during vertical centrifugation, Masella et al. (2012) suggested blanketing the vertical centrifuge with inert gas. Natural sedimentation is a good alternative to the vertical centrifuge but it is considered unsuitable for modern processes due to the extended time required to perform the operation (Gila et al., 2016). Altieri et al. (2014) compared the olive oil quality issued after improved process of natural sedimentation (made of a twin cylindrical columns) to the oil issued from the standard vertical centrifugation. The measurement of quality indices of oil, that is, peroxide, phenol, chlorophyll, carotenoid, turbidity, and K_{232} confirmed that sedimentation process reduces the oxygenation reaction and allows longer shelf-life of the oil. Recent developments suggest the use of bottom settling tanks for the purification step. These conic tanks have a working capacity between 400 and 10.000 L and can be used for both batch and continuous processes (Altieri et al., 2014). Altieri et al. (2015) introduced an innovative sedimentation plant for the separation of high quality VOO at industrial scale which is based on soft hydrocyclone action. The hydrocyclone system was introduced in order to define design requirements for a new olive oil clarification machine, aiming to improve oil quality, safety, and processing capacity (Altieri et al., 2015). Since the high variability of the oil issued from different extraction systems, the hydrocyclone should be carefully settled in order to optimize its use. The main parameters affecting this purification step are the density difference between liquid and solid particles, the particle size and the liquid viscosity, among others. These physical properties are dependent on the fatty acid composition of the oil and are strongly affected by temperature (Gila et al., 2015). Gila et al. (2016) proved that temperature is also an important parameter to monitor during static settling of olive oil. Therefore, the temperature of 30°C showed higher values of settling efficiency compared to lower temperatures (15 and 20°C) using both experimental and computational fluid dynamics procedures.

1.3 WATER CONSUMPTION FOR OLIVE OIL PRODUCTION

For the olive fruits production, the agricultural stage is responsible for an enormous consumption of fresh water including the water used in irrigation, fertilization, and pest control. A study was conducted by Avraamides and Fatta (2008) in Cyprus evaluating the consumption of water in various stages of olive oil production. The authors reported that a total of 3914 L of fresh water are consumed for the production of 1 L of olive oil. However, during olive processing, only 3.51 L of water are consumed for every liter of olive oil produced. This stage produces 4.34 kg of OMWW and 2.07 kg of solid waste (olive kernel) for every liter of olive oil produced (Avraamides and Fatta, 2008).

The water dilution of the olive paste affects the partition of hydrophilic phenols between oil and water and enhances their release in the water phase. The reduction of water dilution during the three phases process leads to an increase of the phenolic concentration in the olive oil, too (Amirante et al., 2002). Therefore, new generation of three-phase decanter centrifuges (water saving decanter) were designed for lower water consumption during the centrifugation process and consequently less generation of