The spent cake from olive oil filtration as biomass feedstock

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Abstract: The most widespread method for virgin olive oil filtration at industrial scale is the so-called precoat body-feed filtration by means of filter aids, mainly consisting of perlite and cellulose. The residue of the process is a spent filtration cake, which has the potential for upgrading if it is understood in the context of the biorefining concept. Data about the production and composition of the spent cake were collected from an industrial case-study. The potential availability of the spent cake has been estimated both at local scale (the industrial case-study) and at larger scale based on the Spanish context, that is the world leading virgin olive oil producer. The mean percentage composition of the spent cake is 5% oil impurities, 7% cellulose, 28% perlite, 52% oil and 8% water. About 10 g of spent cake are produced per kilogram of virgin olive oil to be processed; while about 316 and 7482 Mg of spent cake could be potentially available for the local and large scale occasion, respectively. Owing to its composition entailing oil and waxes, cellulose and mineral fractions, the spent cake has the potential as feedstock for the emerging biotechnologies and biorefineries.

Keywords: spent cake, biorefinery, filtration, virgin olive oil, waxes

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1 Introduction

The current practice of virgin olive oil (VOO) industrial filtration basically involves the use of filtration aids (diatomaceous earth and/or perlite, cellulose), in the so called precoat-body-feed filtration (Sanchez, L. et al., 2010; Masella et al., 2011). The main residue of this process is the spent filtration cake, which is generally considered as a waste. Some years ago a work thesis (Masella, 2002) was carried-out with the main aim to investigate the relationship between some virgin olive oil physicochemical characteristics and the performances of the filtration process at industrial scale by the conventional filter press body-feed filtration. Some years later, part of the data was newly processed by means of multivariate statistic allowing the development of a linear model for the oil filterability prediction at an industrial scale. The results were subsequently published (Masella et al., 2011). However, some of the original data were not included in the paper, because it was out of the scope of that work. Specifically, such data were related to the amount of residual oil in the spent cake (perlite and cellulose) of the filtration process and of its waxes concentration. Today, this information gains a renewed interest if understood in the context of industrial residues upgrading by means of the recently developed approach of biorefining (Cherubini, 2010; Demirbas, 2009). In fact, the virgin olive oil spent filtration cake owing to its composition entailing perlite, cellulose and oil, representing a suitable candidate as a multipurpose feedstock for biorefineries exploitation. The present paper describes the composition of this likely valuable residue from an industrial case study. The potential availability for a biorefinery supply has also been drawn both at local (the case study) and larger scale (the Spanish context, i.e. the world leading virgin olive oil producer).

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2 Material and methods

2.1 Filtration plant

The study was conducted at industrial scale on a horizontal filter-press (114 m2 filter area, 2.240 m^3 filter volume) for precoat body-feed filtration (Galigani Filtri Srl, Italy). This plant was located as an Italian olive oil packaging company where a detailed the company working procedure, as well as of the filtration apparatus and filtration processing can be found (Masella et al., 2011). Briefly, the ordinary factory work involves filtration of blends of different virgin olive oil mainly from Italy, Greece, and Spain. The filtration process consists firstly in the formation of a precoat layer by using cellulose aid with about 160 kg, at average particle size 30 μm. Then, the bodyfeed filtration take place by continuously dispensing perlite aid (average particle size 1 μm) at a variable rate of the inlet oil (mean dose of about 0.30% by weight). Indicatively, the total amount of perlite dispensed was about 600 kg per cycle; cycle duration was about 26 hours; the total processed oil was about 200000 kg. Typically, filtration was initially managed at constant feed rate (about 11000 kg h-1) and increasing differential pressure until a pressure threshold of about 1bar to 1.5 bar. Then, the feed rate was progressively reduced to counterbalance the pressure increase. Filtration was generally stopped at about 3.5 bar.

2.2 Experimental procedure

Thirteen filtration cycles were investigated. For each cycle certain processing parameters were recorded, for example, operation time (h), total amount of processed oil (kg of inlet oil), filtration temperature (C°), total amount of perlite and cellulose aids dispensed (kg). The inlet unfiltered oils were sampled (about 1 kg) and analyzed for determination of total impurities content and waxes concentration. At the end of filtration, the spent filtration cake was sampled and analyzed to determine the water and oil content, as well as the wax esters concentration.

2.3 Chemical analysis

Total waxes concentration was determined according to European Official Method of Analysis (European Commission, 2003), corresponding to the sum of the following separated fractions: C36, C38, C40, C42, C44, and C46 esters. The same procedure was followed for the analysis of the oils recovered from the spent filtration cake. The oil content of the spent cake was determined by the conventional Soxhlet extraction procedure with n-hexane as a solvent (ISO, 1988). The results were expressed as percentage of oil weight obtained after extraction per weight of wet sample. Total impurities content was determined on the unfiltered oils according to the ISO standard method (ISO, 2000). All the reagents were of analytical grade from Sigma-Aldrich srl (Italy).

3 Results

The most relevant operative data of the filtration cycles studied in the present work are summarized in Table 1. The filtration aids consist of a mixture of cellulose and perlite. The amount of cellulose is fixed across the filtration cycles as it is used to forms a constant thickness precoat layer in the filtration chambers of about 8 mm, before the actual filtration takes place by the body-feed. This refers to the continuous dosing of perlite to the influent unfiltered oil. According to Masella et al. (2011) the full amount of perlite per cycle depends on the total filtered oil allowed by the filtration performances. On the average this corresponds to about 720 kg of perlite per cycle (Table 1). The spent cake amount at the end of filtration was not directly measured, but indirectly estimated on the base of the inlet oil impurity content, the total used aids (cellulose plus perlite) and the oil and water content measured on the spent cake after filtration. On the average these latter correspond to about 50% and 8% by weight of the spent cake as sampled after filtration, respectively.

Table 1 Main data recorded during the filtration cycles

Filtration data	minimum	maximum	average
Cellulose (kg) [a]	160	160	160
Perlite (kg) [a]	560	681	622
Total aids added (kg) [b]	720	841	782
Amount of filtered oil ($x10^3$kg) [a]	116	342	212
Oil in spent cake (kg kg^{-1}) [a]	0.31	0.64	0.52
Water in spent cake (kg kg^{-1}) [a]	0.04	0.11	0.08
Impurity in unfiltered oil (mg kg^{-1}) [a]	0.42	0.65	0.52
Total impurity in the filter (kg) [b]	49	223	110
Amount of spent cake (kg) [b]	1170	4327	2231
Total waxes in unfiltered oil (mg kg^{-1}) [a]	224.5	293.5	244.3
Total waxes in recovered oil (mg kg^{-1}) [a]	426.0	1636.0	764.1

Note: [a] measured values; [b] calculated values

The unfiltered oil impurities content was of about 0.05% by weight on the average, corresponding to a mean impurities load in the filter of about 110 kg. Thus, the estimated total spent cake amounts to a mean value of about 2230 kg per cycle. The resulting mean percentage composition of the spent cake is 5% oil impurities (organic material deriving from olive fruits according to Koidis et al. (2008), 7% cellulose, 28% perlite, 52% oil and 8% water. Further, the oil recovered from the spent cake showed a significantly higher total waxes concentration than the unfiltered counterpart, with a mean content of about 760 mg kg^{-1}, i.e. an increment of about 520 mg kg^{-1}. It is useful to present the data of the spent cake production scaled on the unit of processed virgin olive oil, giving a sort of residue intensity index, i.e. the amount of available co-products from the filtration process per unit of virgin olive oil to be processed. This depiction is shown in Table 2 with reference to the average data of Table 1.

Table 2 Production rate of filtration co-products per kilogram of processed VOO

Co-products	amount [a]
Spent cake (g kg^{-1})	10.52
Cellulose (g kg^{-1})	0.75
Perlite (g kg^{-1})	2.93
Recoverable oil (g kg^{-1})	5.47
Recoverable waxes (mg kg^{-1})	4.18
Carbon based material (g kg^{-1})	0.52

Note: [a] computed from average values of Table 1

4 Discussion

The spent cake of virgin olive oil industrial filtration showed interesting traits for a potential exploitation. This is mainly due to its valuable composition, comprising beside the perlite (i.e. the main filtration aid) an important cellulose fraction, residual oil and waxes content, and a minor fraction of carbon based material derived from the olive drupes. All these components can be suitably processed in a biorefinery path to give liquid biofuels and/or value-added bio-products (Demirbas, 2009). Dealing with bio-resources such as biomass or food industrial residues, an important issue is the real availability of such feedstocks. For the virgin olive oil spent cake two possibilities may be built differing in scale, to give an idea of the potential of its exploitation. The first possibility was based on the industrial case study (namely local scenario), where the data of the present work were collected. At the time of the experiment in year 2001 the company processed a yearly amount of olive oil of about $30x10^3$ Mg (Carapelli Firenze S.p.A., personal communication). Direct and reliable information about the current year 2013) oil processed by the company are unavailable to the author, but based on some web reports it seems that the company is now approaching an yearly amount of about 130 x 10^3 Mg processed olive oil (Scarci, 2012). To the purpose of reliability, the local scenario was built assuming the processed oil amount of the year 2001. Of course, this is a conservative choice. As shown in Table 3, the yearly spent cake availability amounts to more than 300 Mg with a potential recoverable oil of about 160 Mg which may be a suitable feedstock for biodiesel production, for instance.

Table 3 Estimated potential availability of the spent filtration cake and that of the relative components for the two assumed studies

Co-products	local [a]	large scale [b]
Spent cake (Mg)	315.7	7482.2
Cellulose (Mg)	22.6	536.6
Perlite (Mg)	88.0	2086.6
Recoverable oil (Mg)	164.2	3890.7
Recoverable waxes (kg)	125.4	2972.8
Carbon based material (Mg)	15.6	369.7

Note: [a] corresponding to the industrial case-study; [b] corresponding to the Spanish study in 2007/2008 period

Moving to the large scale scenario, Spain represents a reliable case, being the world's virgin olive oil top producer (IOOC, 2011). A recent study referring to the Spanish olive crop season 2007/2008, reports that out of the 123.6 x 10^4 Mg of produced olive oil, about 711 x 10^3 Mg of oil were processed and packed in packing plants with a working procedure (raw material collection, filtering, blending, packing and packaging) similar to that of presented case study (Laìn, 2010). Thus, 711 x 10^3 Mg of virgin olive oil to be filtered can be reasonably used to build the large scale scenario, resulting in potential recoverable oil from the spent cake of about 3890 Mg. This is an important figure accounting, for instance, to about 9% of the potential availability of waste cooking oils in Spain as already estimated (Iglesias et al., 2012). In both the considered scenarios, the waxes recovery could have an important role as valuable products in the contest of green chemistry. In fact, vegetable waxes are important bio-compounds with potential in several sectors such as food, pharmaceutical and cosmetic, and their recovery from biomass materials (crops straw), has been already proposed as a part of integrated bio-refineries (Athukorala and Mazza, 2010; Deswarte et al., 2006). Different recovery technologies have been proposed spanning from solvent extraction to supercritical carbon dioxide. Also the most simple membrane filtration seems a viable option (Snape et al., 1996). A further interesting component of the spent cake is cellulose. This fraction could be suitably utilised by the conventional pathway to produce bioethanol through the typical steps involved in the route of hydrolysis-fermentation Conde-Mejìa et al., 2012). The residual mineral fraction consisting of perlite could be suitably processed to give dyes adsorbents (Dogan et al., 2000; Dogan et al., 2003). Alternatively, this residue could be properly used as animal fodder coadjutant in the diet of poultry for instance or as a bulking agent for the composting of livestock wastes (Talebali et al., 2006; Naddaf, et al., 2011). A more advanced and interesting option for the comprehensive exploitation of the cellulose and perlite fraction of the spent cake could be the preparation of carbon-mineral adsorbents by pyrolysis (Zieba, S. et al., 2012; Lédé, 2012). In alternative, pyrolysis can be suitably applied to give conventional pyrolytic products such as charcoal, bio-oils, tars and gases (Lédé, 2012). All these options are summarized in Figure 1, building a potential branch of an ideal biorefinery, where the virgin olive oil spent filtration cake could be an important part of the feedstocks supply.

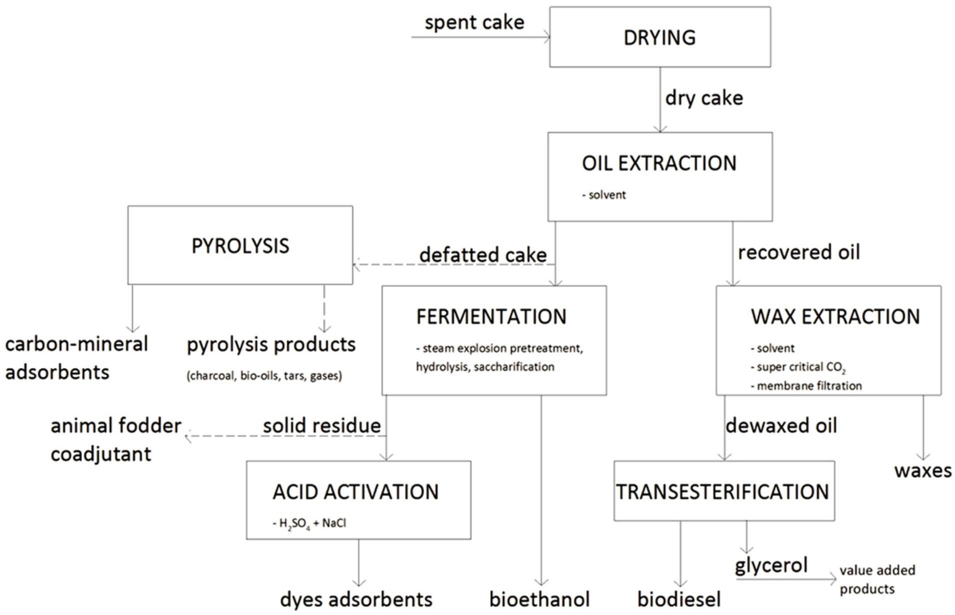

Figure 1 Flowchart of processes that could be implemented in an ideal biorefinery for a full exploitation of the virgin olive oil spent filtration cake (dashed connectors indicate alternative pathways).

5 Conclusions

Starting with data collected some years ago from an industrial virgin olive oil filtration plant, the large scale availability of the spent filtration cake and that of the relative components has been estimated on the base of updated data on olive oil production. Owing to its composition entailing oil and waxes, cellulose and mineral fractions, the spent cake appears a valuable feedstock to supplement the supply of the emerging biotechnologies and biorefineries.

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