Settling of virgin olive oil from horizontal screw solid bowl in static conditions

Abraham M. Gila^{1,2} · Mohamed A. Bejaoui¹ · Gabriel Beltrán¹ · Antonio Jiménez¹

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Abstract This work was aimed to study the clarification efficiency of natural decantation in settling tank on virgin olive oil obtained from a two-ways continuous process. For this purpose, the impurities content of the virgin olive oil were monitored during settling process in settling tank at two different depths. Efficiency of purging system was determined for two days. The experiments were performed at industrial scale during three crop years. During the first minutes of settling was observed an ascent of the smaller organic particles of the oil. Then, most of the virgin olive oil impurities were settled at 300 min, independently of the initial content of virgin olive oil. Finally, oil decantation showed slower rate. Higher clarification values were obtained for those decanter oils with higher impurities content, achieving clarification percentages between of 62.69 and 95.91% at 48 h of settling. The highest settling efficiency was observed for those decanter oils with initial higher impurities content. The purging system used in the settling tanks was not able to remove the most of settled impurities since a considerable amount of the impurities remained in the tank after 48 h, between 13.6 and 71.41% for the studied oils. In the tank purges was observed important oil losses. Therefore, decantation was not an efficient system for oil clarification since its settling capacity varied depending on the initial impurities content

and due to the settled impurities can not be removed fully by purging system.

Keywords Virgin olive oil · Olive oil quality · Olive oil settling · Olive oil decantation · Settling tank · Olive oil clarification

Abbreviations

VOO Virgin olive oil
SOM Solid organic matter
VCS Vertical centrifugal separator
CBST Conical bottom settling tank

Introduction

Virgin olive oil (VOO) is obtained from healthy olive fruits (*O. Europaea*) using only mechanic methods (IOC 2013). This fruit juice is the principal source of fat above all in the Mediterranean diet and is highly appreciated by consumers for its healthy (Covas et al. 2006; Estruch et al. 2006) and sensory properties (Angerosa et al. 2004; Kalua et al. 2007; Jiménez et al. 2012).

Currently, the so-called two-ways continuous extraction system is the most implanted in the Spanish olive oil mills. After olive crushing, the resulting olive paste is kneaded under controlled conditions (temperature, time...). Then, the oily phase is separated from the olive paste by horizontal screw solid bowl, also so-called ‘decanter’ (Altieri 2010; Altieri et al. 2013). The oily must obtained still contains suspended water and solid organic matter (SOM) that have to be removed in the clarification step (Uceda et al. 2006, 2008), since their presence during oil storage can induce anaerobic fermentations that can affect negatively the oil quality, chemical

✉ Abraham M. Gila
agila@citoliva.es

¹ IFAPA Centro “Venta del Llano”, Junta de Andalucía, P.O. Box 50, 23620 Mengíbar, Jaén, Spain

² Present Address: Fundación CITOLIVA, Centro Tecnológico del Olivar y del Aceite, Parque Científico-Técnico GEOLIT, C/Sierra Morena, manzana 11, Edif. CTSA módulo 1, 23620 Mengíbar, Jaén, Spain

composition and sensory characteristics (Tsimidou et al. 2005; Di Giovacchino 2013).

In the olive oil mills equipped with two-ways ‘decanter’, the resulting oily phase is normally formed by 95–98% of oil and 2–3% of SOM and vegetable water (Uceda et al. 2006), although these proportions can oscillate depending on olive oil fruit characteristics and processing conditions. Some decanter parameters can affect the oil quality and composition as lukewarm water addition, mass flow rate, technological coadjuvant addition during malaxation, diaphragm regulation of the liquids discharge and differential speed between conveyor screw and bowl (Caponio et al. 2014, 2015; Leone et al. 2015; Tamborrino et al. 2015). The clarification of oil from ‘decanter’ is currently carried out by vertical centrifuge separators (VCS) with a speed of 6500–7000 rpm, being a very quick operation that requires low labour. This operation needs a lukewarm water addition to the VCS inlet, around 1:1 (oil/water) (Jiménez et al. 1995; Di Giovacchino et al. 2002). Water addition increases the waste water generated by the extraction process, as well as costs, and also decreases the levels of phenolic compounds in the oil because of their high solubility in the aqueous phase (Jiménez et al. 1995; Di Giovacchino et al. 2002; Masella et al. 2009).

Alternative clarification systems have appeared in the last years, such as the conical bottom settling tanks (CBST). These tanks, have a cone angle between 45° and 60°, a capacity between 400 and 10,000 L, are provided with a purge system (manual or automatic), can be used for both static and dynamic conditions, and the clarification time ranges between 24 and 48 h before sending the clarified oil to the storage tank. The main factors affecting to this separation process are: the density difference between liquid phases and solid particles, solid particle size and concentration, the liquid viscosity, among others (Davis 2010). Some of these parameters, such as oil density and viscosity, have already been studied for several vegetable oils, including olive oil (Bonnet et al. 2011; Esteban et al. 2012), and is known that these physical properties depend on the VOO fatty acid composition and are strongly affected by temperature (Gila et al. 2015).

Although the use of settling tank is being implemented, there is not information on settling process of VOO from ‘decanter’ since natural settling was applied for clarification of VOO from pressure system (currently in disuse) that showed great differences respect to those from two-ways decanter (Uceda et al. 2006; Di Giovacchino 2013).

The aim of this work was to study the clarification of VOO from ‘decanter’ by natural settling tank, through of the evolution of the impurities during the settling process and the impurities removal efficiency of this system.

Materials and methods

The study was carried out for three crop years (2010/2011, 2011/2012 and 2013/2014). Around 400 L (366 kg) of virgin olive oil were extracted, for each crop year, from ‘Picual’ olive fruits harvested from the experimental olive orchard of the research center IFAPA “Venta del Llano” in Mengíbar (Jaén).

Olive oil extraction system

The experiments were realized in the experimental oil mill of IFAPA “Venta Del Llano”, which consists of a rotating-hammer crusher with 6 mm sieve diameter of the grid, a thermobeater formed by three containers (Pieralisi, Spain) 600 kg each and a horizontal screw solid bowl (Pieralisi SC-90), working at two ways mode.

To carry out the oil clarification was used a conical bottom settling tank (Secovisa, Spain), with a capacity of 400 L and equipped with a purge system.

Design of the assay

Figure 1 shows the design of the assay. The VOO obtained from the ‘decanter’ was passed through a 1 mm vibrational sieve and then was led to fill a settling tank. The time needed to fill a settling tank was 205 min and the oil temperature during settling process was controlled at 22 ± 2 °C. Samples of VOO (by triplicate), were taken after sieving (Initial VOO), during the tank filling in order to characterize the initial decanter oil. In the second crop year (2011/2012), some parameters of the ‘decanter’ were changed to obtain a VOO with higher impurities content than the other two crop years studied.

For settling analysis, once the tank was filled (being this time 0 min), oil samples were taken (by triplicate) from the tank, every 30 min until 6 h at two different tank depths 18 and 88 cm (corresponding to the outlet and inlet point of the tank, Fig. 1). Then, oil samples were taken at 24 and 48 h of settling. The changes in VOO moisture and SOM contents during oil settling were monitored.

The efficiency in impurities removing of natural decantation was determined by weighting and analysing the tank purges at different settling times: 21, 23, 25, 27, 28 and 48 h for 2010/2011 crop year, 21, 24 and 48 h for 2011/2012 crop year and 5, 24 and 48 h for 2013/2014. Settling tank purging was carried out by the purging system (Fig. 1), equipped with a butterfly valve with automatic pneumatic opening. The purging operation was performed until clear oil appeared in purges reception recipient. The total purges recuperated were collected, weighted, and

Fig. 1 Diagram of static settling assay of VOO from 'Decanter' and sampling points

samples were taken (by triplicate) to determine their moisture and solid organic matter content.

Determination of moisture and solid organic matter

To determinate the moisture, approximately 10 mL of olive oil or purge were weighted in a ceramic capsule with filter paper. The sample was dried in an oven at 105 °C until weight stabilization. The loss of weight gave moisture (%) and volatile matter (UNE 1973). After dried, solids were retained in the paper filter and the oil was extracted with petroleum ether in a Soxhlet system. Finally paper filter was dried and weighted to determine solid amount (%) (UNE 1962). Impurities content were the sum of moisture and solid organic matter.

Settling efficiency

The settling efficiency (Table 3) at different times was calculated by Eq. 1:

$$s_t = \left[\frac{(c_i - c_t)}{c_i} \right] \cdot 100 \quad (1)$$

where s_t is the settling efficiency at 't' time (%), c_i is the impurities content of the initial VOO from 'decanter' (%) and c_t is the impurities content at 't' time for 18 and 88 cm depth (%).

Removal efficiency

To calculate the percentage of impurities (Fig. 2b) removed at different settling times was used the Eq. 2:

$$r_t = \frac{p_i}{p_t} \cdot 100 \quad (2)$$

where r_t is the removal efficiency at 't' time (%), p_t is the total impurities amount in the tank once filled (kg) and p_i is the removed impurities amount at 't' time (kg). p_t was calculated by the total VOO weight in a full tank (366 kg) and the impurities content of the initial VOO from

'decanter' (%), while p_i was determined by total weight of each purge at 't' time (kg) and its impurities content (%).

Results and discussion

Settling process

The moisture and SOM content of VOOs from two-ways 'decanter' for the three crop years are shown in Table 1. These initial oils used for settling showed large differences in their composition between the crop years. The oil moisture ranged from 1.56 to 13.82%, whereas the SOM content varied between 0.32 and 1.09%. This variability between crop years allowed the study of this clarification system at standard and unfavorable processing conditions as in the second harvesting date.

For the 3 crop years oil settling was carried out for 48 h. The oil moisture and SOM values are presented in Table 2. As can be observed, at the beginning of the settling process (time 0), when the tank was completely filled, the impurities values (moisture + SOM) in the tank top (18 cm) were similar for the 3 years analysed, between 1.45 and 1.68%, for 2010/2011 and 2013/2014, respectively, independent of the impurities content of the initial decanter oil. At 88 cm depth the oil impurities showed large differences between years, 1.76 and 1.83% for the first and third crop year, whereas 60.47% for the second. This variability observed, at 88 cm of depth, can be explained by the different characteristics of the VOO used for each crop year.

Figure 2a shows the evolution of oil impurities content, at two different sampling depths (18 and 88 cm) in the settling tank for the three crop years. During the first minutes of the experiment, a slight increase of the percentage of SOM at the tank top was observed. In fact, a slim coating formed by SOM over the oil surface was observed. The just extracted VOO used to fill the tank includes emulsified air that releases as microbubbles, dragging the smaller size organic particles up to the top after the tank complete filling. In a laboratory study, Gila

Fig. 2 **a** Evolution of impurities content during the static settling of VOO from ‘decanter’ and **b** impurities removal efficiency of the purges system during the settling process for three crop years

et al. (2016) described an increase of the SOM content during the first minutes of VOO settling as described above for CBST. Then, moisture and SOM level decreased (Fig. 2a), showing for all crop years a settling curve composed by two phases well differentiated.

The first phase of hindered settling, which duration of this phase was different for each depth crop year. At 18 cm depth,

the time range of the hindered phase was 30–300 min, 0–300 min and 0–300 min for the 2010–2011, 2011–2012 and 2013–2014 crop years, respectively. However, at 88 cm the time for hindered phase was different: 0–300 min, 150–300 min and 0–300 min, for 2010–2011, 2011–2012 and 2013–2014 crop year, respectively. This hindered settling phase corresponded to a linear model (Eq. 3);

Table 1 Moisture and SOM of the initial VOO from ‘decanter’ for three crop years

Crop year	2010/2011	2011/2012	2013/2014
M (%)	2.20 ± 0.12 ^a	13.82 ± 1.26	1.56 ± 0.51
SOM (%)	0.32 ± 0.03	1.09 ± 0.35	0.37 ± 0.20

M moisture, SOM solid organic matter

^a Mean values ± SD (n = 3)

$$c = b + m \cdot t \quad (3)$$

where c is the impurities expressed in %, t is the time [min], b is the initial impurities content of decanter oil and m represent the settling velocity of impurities. The values of m and b have been calculated for each depth and crop year (Table 3).

Higher m values were observed at both sampling depths for 2011–2012 crop year. Then, as observed in Table 2, those oils with higher impurities content gave a higher settling velocity observed by moisture and SOM reduction, thus a higher efficiency of settling was observed. Attention should be paid to the clarification rate at 88 cm for this second crop year, where the values of the moistures and SOM remained stable until 150 min because of the settled impurities from superior levels were crossing this point. After this time, a high clarification rate was noted.

As settling continues, a compressed layer of particles began to form and the settling velocity decreases until oil impurities content remained stable. This second step, when the rate was stabilized, is called compression settling (Kynch 1952). The stabilization was achieved for all the crop years at 300 min, although the oil impurities content at this time varied between crop years. The impurities content remained practically stable from this time (300 min) until 48 h of settling. Gila et al. (2016) described a similar behavior for oil moisture and SOM during VOO decantation in settling columns.

Table 3 shows the percentage of VOO clarification (expressed as settling efficiency) achieved during the settling process at 0, 300, 1440 and 2880 min at 18 and 88 cm. Most of the impurities were settled at 300 min. From this time (300 min) to 48 h, a low variation of the clarification rate was observed. Hence, the clarification degree depends strongly on the initial decanter oil impurities content, reaching higher clarification values for VOOs with higher impurities content. At 18 cm of depth, the percent of impurities removed at 48 h were 78.97, 95.91 and 62.69% of, for 2010/2011, 2011/2012 and 2013/2014, respectively. Concerning to the clarification at 88 cm depth, showed a similar behavior than 18 cm, with higher clarification percentage for the decanter oils with higher initial impurities content.

Removal efficiency

In order to evaluate the removal efficiency of settled impurities, tank purges were performed at different settling times. Table 4 shows the impurities and oil content of the tank purges and their weights. Considering, the impurities content of the initial VOOs from ‘decanter’ and the capacity of the settling tank (0.4 m³), or 366 kg with an oil density of 916 kg/m³ (Uceda et al. 2006), the total impurities weight expected for the full tank should be 9.25, 54.57 and 7.10 kg for the crop years 2010/2011, 2011/2012 and 2013/2014, respectively.

For the first year, purges were performed at 21, 23, 25, 27, 28 and 48 h of settling. As can be observed, in Fig. 2b are represented the percentages of impurities removed by purge system at different times. Around 48.87% of the total impurities content was removed at 48 h and 23.72 kg of oil was lost in these purges. For the second year, considering that in the first year most of the impurities were removed at 24 h, only three purges were performed: 21, 24 and 48 h. The 86.4% of the total impurities content was removed in the three tank purges, of which 84.39% was removed in the first one. The total oil loss in the purges was 29.96 kg. For the last year, since results of previous years showed that most of the impurities were settled at 300 min, the purges were carried out at 300, 1440 and 2880 min. For the pool of purges, 28.59% of the total impurities content were removed and 11.09 kg of oil was lost, although most of the impurities were taken out in the first one (20.70%). According to these data, after 48 h of settling around 51.14, 13.30 and 71.41 percent of the impurities still remained in the tank for 2010/2011, 2011/2012 and 2013/2014, respectively. Generally the initial decanter oil with high impurities content showed the higher impurities removal percentage, although higher amounts of impurities still remained in the tank.

During the tank emptying was observed as an oil stream let to the oil be evacuated whereas most of the impurities remained adhered on the tank cone walls. Moreover, when the tanks were emptied, once completed the assays, at the bottom were observed visually impurities adhered on the cone walls and thus, these impurities were not removed during the purges although had been settled.

Conclusion

The experimental results showed that independent of the initial impurities content of VOO, most of the impurities were settled around 300 min. Therefore, settling times between 24 and 48 h used by the oil mills would not be necessary to obtain high clarifications.

Table 2 Moisture and SOM content of the oil samples during settling analysis at two different depths (18 and 88 cm) for the three crop years

Time (min)	Crop year 2010/2011			Crop year 2011/2012			Crop year 2013/2014		
	18 cm			18 cm			18 cm		
	M (%)	SOM (%)		M (%)	SOM (%)		M (%)	SOM (%)	
0	1.33 ± 0.08 ^a	0.12 ± 0.00	1.58 ± 0.17	0.18 ± 0.02	0.05 ± 0.02	52.35 ± 2.47	8.12 ± 0.74	1.54 ± 0.03	0.14 ± 0.01
30	1.50 ± 0.01	0.19 ± 0.03	1.37 ± 0.05	0.17 ± 0.03	0.10 ± 0.04	60.73 ± 1.24	5.63 ± 0.07	1.42 ± 0.02	0.17 ± 0.04
60	1.19 ± 0.08	0.17 ± 0.01	1.12 ± 0.09	0.11 ± 0.02	0.03 ± 0.00	58.93 ± 1.64	6.86 ± 0.85	1.40 ± 0.01	0.15 ± 0.02
90	1.06 ± 0.04	0.04 ± 0.01	1.01 ± 0.02	0.03 ± 0.01	0.04 ± 0.01	52.95 ± 4.16	8.52 ± 2.53	1.33 ± 0.04	0.15 ± 0.01
120	1.03 ± 0.03	0.03 ± 0.01	0.93 ± 0.02	0.00 ± 0.00	0.05 ± 0.04	56.18 ± 6.34	8.36 ± 2.18	1.29 ± 0.05	0.14 ± 0.02
150	0.94 ± 0.02	0.00 ± 0.00	0.88 ± 0.02	0.00 ± 0.00	0.06 ± 0.01	58.39 ± 1.46	5.89 ± 2.26	1.26 ± 0.05	0.17 ± 0.01
180	0.64 ± 0.10	0.13 ± 0.09	0.78 ± 0.01	0.00 ± 0.00	0.03 ± 0.02	35.91 ± 6.27	3.90 ± 0.84	1.16 ± 0.03	0.13 ± 0.03
210	0.71 ± 0.01	0.07 ± 0.02	0.74 ± 0.02	0.06 ± 0.00	0.04 ± 0.01	21.62 ± 6.69	1.97 ± 0.94	1.07 ± 0.04	0.14 ± 0.00
240	0.74 ± 0.12	0.05 ± 0.00	0.68 ± 0.03	0.04 ± 0.02	0.13 ± 0.10	9.85 ± 4.25	0.71 ± 0.51	1.06 ± 0.02	0.13 ± 0.01
270	0.64 ± 0.02	0.04 ± 0.00	0.72 ± 0.03	0.07 ± 0.04	0.03 ± 0.01	2.78 ± 0.54	0.34 ± 0.29	0.99 ± 0.01	0.12 ± 0.00
300	0.62 ± 0.08	0.04 ± 0.01	0.53 ± 0.01	0.04 ± 0.01	0.04 ± 0.01	1.91 ± 1.22	0.43 ± 0.19	0.96 ± 0.07	0.12 ± 0.01
330	0.75 ± 0.04	0.07 ± 0.00	0.62 ± 0.02	0.06 ± 0.02	0.04 ± 0.00	1.72 ± 0.14	0.06 ± 0.02	–	–
360	0.78 ± 0.11	0.10 ± 0.01	0.69 ± 0.06	0.07 ± 0.00	0.04 ± 0.01	1.82 ± 0.04	0.14 ± 0.03	–	–
1440	0.65 ± 0.02	0.00 ± 0.00	0.74 ± 0.12	0.00 ± 0.00	0.03 ± 0.00	3.51 ± 1.20	0.37 ± 0.15	0.63 ± 0.02	0.12 ± 0.01
2880	0.53 ± 0.05	0.00 ± 0.00	–	–	0.01 ± 0.01	0.57 ± 0.01	0.02 ± 0.00	0.58 ± 0.03	0.14 ± 0.01

M moisture, *SOM* solid organic matter^a Mean values ± SD (n = 3)

Table 3 Coefficients for impurities values of hindered settling of the VOO from decanter at two different sampling depths (18 and 88 cm) and settling efficiency of impurities at 18 and 88 cm of depth at the 0 (S_0), 300 (S_{300}), 1440 (S_{1400}) and 2880 (S_{2880}) minutes for the three crop years

Crop year	2010/2011		2011/2012		2013/2014	
Depth (cm)	18	88	18	88	18	88
$m(^*)$	−0.0034	−0.0034	−0.1680	−0.4026	−0.0013	−0.0026
$b(^*)$	1.4339	1.5105	1.7320	65.981	1.6211	1.7995
R^2	0.8470	0.8396	0.8509	0.9345	0.7829	0.9837
s_0 (%)	42.46	3.16	88.80	—	12.95	5.18
s_{300} (%)	73.81	77.38	94.77	84.31	44.04	37.31
s_{1440} (%)	74.21	70.63	92.89	73.98	61.14	51.81
s_{2880} (%)	78.97	—	95.91	96.04	62.69	51.81

b initial impurities content of decanter oil, m settling velocity of impurities

Table 4 Impurities (moisture and solid organic matter) and oil content of the different purges from settling tank for the three crop years

Crop year	Samples	Sampling time (h)	Impurities (%)	Purge weight (kg)	Impurities content (kg)	Oil content (kg)
2010/2011	Initial VOO	—	2.52 ± 0.15^a	366^b	9.25^c	356.75^d
	Purges	21	21.91 ± 0.45	7.81	1.71	6.10
		23	21.58 ± 0.13	3.50	0.76	2.74
		25	17.39 ± 0.18	3.69	0.64	3.05
		27	15.83 ± 1.31	4.18	0.66	3.52
		28	8.41 ± 0.78	5.32	0.45	4.87
		48	8.05 ± 0.31	3.74	0.30	3.44
				28.24	4.52	23.72
2011/2012	Initial VOO	—	14.91 ± 1.54	366^b	54.57^c	311.43^d
	Purges	21	68.42 ± 2.30	67.3	46.05	21.25
		24	16.64 ± 2.30	3.08	0.51	2.57
		48	21.46 ± 4.89	2.73	0.59	2.14
				73.11	47.15	25.96
2013/2014	Initial VOO	—	1.93 ± 0.70	366^b	7.10^c	358.90^d
	Purges	5	17.98 ± 0.13	8.15	1.47	6.68
		24	12.91 ± 0.81	2.38	0.31	2.07
		48	9.47 ± 0.08	2.59	0.25	2.34
			Total purges	13.12	2.03	11.09

^a Mean values $\pm$ SD ($n = 3$)

^b Total VOO weight in a full tank for initial VOO

^c Total impurities in the VOO contained in the tank for initial VOO

^d Total oil contained in the tank for initial VOO

During the first minutes, an ascent of the smaller organic particles was observed probably dragged by the emulsified air in form of microbubbles from freshly extracted VOOs.

The purging system was not able to remove the most settled impurities even after 48 h. Although an important percent of impurities could be removed purging the tank after 300 min of settling. This inefficiency of the purging may be due to the Venturi effect created by a stream formed in the center of the conical bottom during the purges. The removal efficiency was higher for VOOs with higher impurities content. Furthermore, a significant oil

amount was lost during the purges for the three crop years, being the greatest loss when the initial VOO had higher impurities content.

Therefore, although the use of settling tanks for VOO clarification step may reduce the water consumption, the wastewater generated and energy consumption, are not effective since most of the settled impurities were not removed after 48 h, losing a significant oil amount in the purges. Furthermore, because of the direct contact of these impurities in the tank bottom, with VOO, its quality and sensory properties could be negatively affected. Further works focused in these aspects should be carried out.

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