Evaluation of mechanical and rheological aspects of the malaxed olive paste

Tamborrino A (1), Catalano P (2), Leone A. (3)
(1) University of Bari Aldo Moro. Dept. DISAAT
Via Amendola 165/A – 70126 Bari, ITALY.
Tel 0039 0805443122, Fax 0039 0805443080
Email corresponding Author: a.tamboorino@agr.uniba.it

(2) University of Molise. Dept. SAVA, Mechanics Section
Via De Sanctis. n.c. – 86100 Campobasso, ITALY.
Tel 0039 0874404682

(3) University of Foggia. Dept. PRIME
Via Napoli, 25 - 71100 Foggia, ITALY
Tel 0039 0881589120 - Fax 0039 0881 589120

Abstract
A malaxer prototype has been developed and it is able to control the oxygen both in the head space and in the paste. Besides, an innovative system to inject oxygen has been introduced on this machine. To investigate on the mechanical parameters a torque monitoring system was implemented on this prototype. In this research has been studied the correlations among oxygen dissolved in the malaxed olive oil paste and rheological properties. The torque and viscosity correlation is important to define an in line parameter directly detectable during kneading to understand when the paste is ready to get the next phase. Experimental tests were done. In the trials, the malaxer prototype was filled with olive paste. A different malaxing condition were chosen (Nitrogen; 15' injecting air; 30' injecting air; air). During the malaxation process the torque on the reel were measured using a rotating torque transducer RT2 (AEP). Rheology measurements were carried out using a Brookfield rotational rheometer (R.v model; Brookfield DV-II+Brookfield Engineering laboratories, Inc., Stoughton, MA, USA). This study examines the mechanical and rheological aspects of different olive oil malaxed paste. The results indicates that the oxygen inject during malaxation is a factor that influence the torque and the viscosity of the paste. The viscosity of the investigated olive oil paste shows dependency on the oxygen concentration. As regarding as the data of the torque a progressive decrease of the torque during malaxation process is observed too. The lower viscosity and torque value are observed when oxygen is inject in the olive paste.

Keywords: apparent viscosity, malaxer, oxygen

Introduction
The malaxation is a phase of the olive oil extraction process. After the crushing of the olive the olive paste obtained is kneaded and heated to achieve the right properties to optimize the extraction yields in the next phase, which is the process of solid-liquid separation by centrifugation. The malaxing phase can be considered as an important phase of the extraction process for its influence on the yield and the quality of the olive oil. There are essentially some main effects generated during the malaxation process, which make the process not only a mechanical process but also biochemical and physical-chemical process. The first effect of the mixing of the paste is to promote the breakage of the unbroken cells containing oil; the tissues and cells of the olive in fact are broken with contact to the pieces of stone formed during the crushing process, this allows the release of drops of oil and at the same time allows the occurrences of the coalescence phenomena. The minute oil droplets come together
forming jet larger droplets this renders an easier and more complete separation of the oil and thus influences the yield. The main effect of this phenomena is the decrease of the viscosity of the paste. But, during malaxation process the oil is in contact with the aqueous phase in which various lipases and oxidases (such as Polyphenoloxidase, Peroxidase and Lipoxygenase pathways) are active. The different enzymatic reactions cause both the formation of a great number of compounds responsible of the aromatic fraction of the oil and the oxidation of the phenolic compounds resulting in reduced phenolic concentration of the oil (Kalua et al., 2007). The enzymes are triggered by the crushing phase and are active during the malaxation step.

Mixing conditions and the malaxer machine model can both influence the activity of these enzymes, which affect the volatile and phenolic composition of virgin olive oil, and as a consequence affect its sensory qualities and beneficial properties for human health. Several research have been carried founding the best malaxing conditions to improve the olive oil quality. Low temperatures are recommended for the malaxation step (i.e., ≤30 °C), whereas the optimal malaxation time should be between 30 and 45 minutes; this satisfy yield and a good quality. This time, according to rheology of the olive pastes, seem to satisfy these requirements, because compounds responsible for attractive perceptions, such as esters, are still present at high level, and concentrations of those giving unpleasant sensations such as trans-2-hexen-1-ol and hexan-1-ol are rather low. In addition, the amount of secoiridoid compounds is great enough to assure a suitable shelf-life of the pro-duct and the content of branched aldehydes is in the range typical of olive oils of high quality (Angerosa et al., 2001). Thus, the right temperature and the time of malaxation is important in order not to inactivate the LOX pathway and in order to reduce activity of PPO and POD, combined with good oil extraction yields. Several studies have been carried out on the effect of O₂ on the olive oil quality (Servili et al., 2003a; Servili et al., 2003b; Amirante et al., 2006; Servili et al., 2008; Amirante et al., 2008a; Tamborrino et al., 2010). Malaxer machine that included the use of inert gas processing and oxygen concentration control have been used. Malaxation under N₂ flush seemed to inhibit PPO and POD activities, resulting in an increase in the phenolic concentration. Using a malaxer machine with hermetic cover cap it is possible to work at low oxygen level without the use of N₂ flush. As a result of a decrease in the oxygen content, PPO activity was slowed down and, consequently, a reduced degradation of phenolic compounds occurred. The LOX pathway was affected by decreasing the oxygen content; the total C₆ compound content was reduced, and, at the same time, the enzyme activity was increasingly addressed to the formation of aldehydes, especially trans-2-hexenal (Migliorini et al., 2006).

The research carried out until now have always considered the control of the oxygen in the headspace through the reduction of it using a malaxer machine able to do it. In a previous research the use of oxygen in the paste, during malaxation has been introduced and a preliminary study has been conducted on a malaxer prototype machine. A malaxer prototype was able to control the oxygen both in the head space and in the paste. Moreover, the malaxer prototype included an innovative system, never used before: a set of micro-oxygen injectors able to inject oxygen by two distributors in sintered steel directly in the paste, on real time. (Tamborrino et al., 2011). If the oxygen plays a important role for the quality of the resulting olive oil, undoubtedly it could influences the rheological behaviour of the malaxed paste. One of the fundamental parameter characterizing olive paste behaviour is viscosity. In fact, viscosity affects velocity gradients, and therefore the motion of suspended solid particles. (Amirante and Catalano, 1995).

Current, the olive extraction equipments do not have devices to assess this important parameter, so the evaluation of the viscosity is approximate and based on the experience of
the operator. The malaxer prototype has been also equipped with a torque monitoring system on the reel to evaluate the torque measured on the paste. The torque and viscosity correlation is important to define an in line parameter directly detectable during kneading to understand when the paste is ready to get the next phase. Changing the processing conditions an influence of olive paste rheological properties was implied and thus, the knowledge of the a relationship between the rheological behavior of the olive paste and the mixing conditions could be of fundamental importance to optimize decanter performances (Amirante et al., 2008b). In this research are showed the results of the experimental tests carried out to investigate the rheological behaviour of the olive paste in different processing conditions and a torque measurements for the different conditions were investigated too. A correlation between torque and apparent viscosity value (η) are showed.

Materials and methods

Plant material

Olive fruits (*Olea europaea*) cvs. Coratina at medium ripeness were harvested during the 2010-2011 season in the Foggia area (Apulia-Italy) and transported the same day to the mill. A hammer crusher was used for the olive oil crushing phase; a malaxer prototype was used for the olive oil malaxation phase; a solid/liquid horizontal centrifugal decanter (two-phase) was used for the centrifugation phase. A liquid/liquid vertical plate centrifuge completed the plant.

Malaxer prototype

A malaxer prototype machine was an industrial tank having a capacity of 350 litres, with a circular spiral shape. The tank had a length of 1250 mm. The hermetic sealing was enclosed with three separated doors. These doors enabled to directly access the interior of the tank if required. Stainless steel material was used for the prototype machine. A window enabled to continuously monitor the conditions of the paste during the scutching phase. The polycarbonate was used to made the window. The prototype was also equipped with a valve for inert gas processing and equipped with the paste and water temperature probes; they enabled to accurately monitor the trend of temperature both in the paste as well as in the jacket of the tank, where hot water circulated. All the data were show on the LCD screen. The malaxer prototype was completed with a PLC. The software of this PLC allowed to the signals to be received and to provide the conversion of these electrical measurements into physical measurements and their visualization on the LCD display. The system allowed monitoring of the acquired data and it also allowed the setting of the parameters. The parameters set were: the on/off engine, the paste temperature and water temperature, the reel speed.

An oxygen supply system Ossigena is used. It provide for the supply of oxygen during the malaxation process using different regulations. It is able to supply oxygen continuously, or in single dose, or in a way that provides a very high flow for a very short time.

To measure the torque in real time a rotating torque transducer RT2 (AEP) was fixed between the motoreducer and the reel. The output was 2mV/V or ±10Vdc, the nominal Speed: 4000 rpm and a contact transmission has been used.

The prototype machine has been also equipped with a series of sensors which continually measure the quantity of oxygen both in the air of the head space and in the liquid phase of the paste (Oxygen measurement sensors - Mettler Toledo)
**Experimental tests**

The olives were processed into paste with the hammer crusher machine. Thus, the malaxer prototype was filled with olive paste. Different operational processing conditions were carried out for O₂ concentration in the paste. Each trial was performed in duplicate by processing homogenous olive batches (400 kg), under the same malaxation temperature and malaxation time conditions; the experimental tests have been carried out setting 27 °C as paste mixing temperature and 30 minutes as mixing time. The oxygen dissolved in the paste was the parameters controlled during processing. During malaxation, oxygen dissolved in the paste was measured using a Mettler Toledo Oxygen Sensor. Results are expressed as ppm of O₂. Four different conditions were studied: (P30): injecting continuously air in the paste using a flow equal to 80 mg/sec for all the malaxation time; (P15): injecting continuously air in the paste using a flow equal to 80 mg/sec for the first 15 minutes of malaxation time; (PN): saturating with N₂ the malaxer before to fill the paste using a nitrogen cylinder connected with a manometer (max 2 - 3 % of oxygen); (PA) under air for the entire time malaxation, leaving the doors opened.

**Samples**

Paste during mixing were sampled after 0, 15, 30 min. The samples taken were conditioned to the same temperature (27°C). The viscosity measurement was done. Rheology measurements were carried on the 36 samples of 600 mL olive paste. Three replicate trials have been done for each samples.

**Torque measurements**

During the filling of the tank torque measurements were done as well as during the entire malaxation process. Besides, data gathering were done on the paste when the machine was on going. The measurements were showed on the LCD display and express as N m.

**Rheology measurements**

Rheology measurements were carried out using a Brookfield rotational rheometer (R.v model; Brookfield DV-II+Brookfield Engineering laboratories, Inc., Stoughton, MA, USA) equipped with interchangeable disc spindles, 1–6 (model RV/H/A/HB; Brookfield DVII + Brookfield Engineering laboratories). The spindle was driven by a synchronous motor through a calibrated spring; the deflection of the spring was indicated by a pointer on a dial. By utilizing a multiple speed transmission and interchangeable spindles, a variety of viscosity ranges were measured. The samples were directly loaded into the 1000 mL glass containers, where interchangeable spindles were inserted and an equilibrium time of a particular shear rate was ca. 40 s. A controlled temperature bath circulated water through the jacket surrounding the rotor and cup assembly to kept the temperature at the chosen thermal level chosen (27°C). The readings of the apparent viscosity were taken at rotational speeds from 0.5 to 100 rpm. To interpret the experimental results in terms of viscosity, the torque-speed data and scale readings were converted into shear stress–shear rate relationships using numerical conversion values (Mitschka, 1982). An empirical power-law model was used to calculate the apparent viscosity and the flow behaviour index from the shear rate (XUEWU et al., 1996) which is frequently used for engineering applications. It is given by the following equation (1):

\[
\eta_{\text{app}} = k \gamma^{(n-1)}
\]  

(1)
where $\eta_{app}$ is the apparent viscosity, $\gamma$ is the shear rate ($s^{-1}$), $n$ is the flow behaviour index (dimensionless) which is less than unity for pseudoplastic behaviour, $k$ is the consistency index (Pa s$^n$).

**Statistical analysis**
Statistical analysis was carried out using Microsoft Excel software. Significant differences between treatments were determined using one-way ANOVA.

**Results and discussion**
Regarding the data of the torque a progressive increase of the torque during the filling phase of the malaxer has been registered. After that, during the malaxation process a decrease of the data of the torque was observed. The air seems to have an influence on the torque; the lowest torque values were observed for the samples P30 and P15. The highest torque values were observed for the samples PA. As regarding as the viscosity, the experimental data, consisting of apparent viscosity values ($\eta$) and the related shear strain rate ($\gamma$), were collected. These data were processed by means of a linear regression in logarithmic scale in order to verify the consistency of the power law model for olive paste rheological behaviour, according to the equation (1) where $k$ is the consistency coefficient, and $n$ is the flow index (table 1). The Figure 1 shows the correlation between the torque values and the apparent viscosity. The correlation degree ($R^2$), for all the conditions studied, highlights that the variables are highly correlated except for sample PA. This could be due to the high difficulty in controlling air - paste mixing during malaxation: further investigation will be necessary in order to asses other parameters influencing torque and viscosity measurements.

**Conclusion**
This study examines the mechanical and rheological aspects of different olive oil malaxed paste. The results indicates that the oxygen injected during malaxation is a factor that influence the torque and the viscosity of the paste.

The viscosity of the investigated olive oil paste shows dependency on the oxygen concentration, as well as on the time of malaxation according with Di Renzo e Colelli, 1997. The viscosity decreases when the malaxation time increases. As regarding as the data of the torque a progressive decrease of the torque during malaxation process is observed too. The lower viscosity and torque value are observed when oxygen is injected in the olive paste. The main advantage of the introduction of the torque monitoring system is that the torque value is directly read on the display in real time and when the machine is ongoing and highly correlated with viscosity. This device could be associated at the routine equipment of the malaxer machine since that it is easy to handle for the operator. More data should be acquired to better correlate torque and viscosity in the different tested conditions. This is necessary in order to use torque measurements as an evaluation parameters for paste consistence during malaxation.

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Table 1. Experimental data and linear regression results of olive paste rheological analysis.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Sample description</th>
<th>Consistency Coefficient $(k)$</th>
<th>Flow index $(n)$</th>
<th>Correlation Degree $R^2$</th>
</tr>
</thead>
<tbody>
<tr>
<td>PN$_0^\prime$</td>
<td>Hammer crusher outlet</td>
<td>113965</td>
<td>-0.831</td>
<td>0.95</td>
</tr>
<tr>
<td>PN$_{15}^\prime$</td>
<td>Malaxed: extracted after 15 minutes</td>
<td>89299</td>
<td>-0.634</td>
<td>0.98</td>
</tr>
<tr>
<td>PN$_{30}^\prime$</td>
<td>Malaxed: extracted after 30 minutes</td>
<td>76552</td>
<td>-0.811</td>
<td>0.97</td>
</tr>
<tr>
<td>P15$_0^\prime$</td>
<td>Hammer crusher outlet</td>
<td>133864</td>
<td>-0.833</td>
<td>0.99</td>
</tr>
<tr>
<td>P15$_{15}^\prime$</td>
<td>Malaxed: extracted after 15 minutes</td>
<td>127423</td>
<td>-0.798</td>
<td>0.99</td>
</tr>
<tr>
<td>P15$_{30}^\prime$</td>
<td>Malaxed: extracted after 30 minutes</td>
<td>108294</td>
<td>-0.751</td>
<td>0.99</td>
</tr>
<tr>
<td>P30$_0^\prime$</td>
<td>Hammer crusher outlet</td>
<td>98206</td>
<td>-0.779</td>
<td>0.99</td>
</tr>
<tr>
<td>P30$_{15}^\prime$</td>
<td>Malaxed: extracted after 15 minutes</td>
<td>62207</td>
<td>-0.854</td>
<td>0.99</td>
</tr>
<tr>
<td>P30$_{30}^\prime$</td>
<td>Malaxed: extracted after 30 minutes</td>
<td>42681</td>
<td>-0.815</td>
<td>0.99</td>
</tr>
<tr>
<td>PA$_0^\prime$</td>
<td>Hammer crusher outlet</td>
<td>185402</td>
<td>-0.625</td>
<td>0.94</td>
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<tr>
<td>PA$_{15}^\prime$</td>
<td>Malaxed: extracted after 15 minutes</td>
<td>80362</td>
<td>-0.761</td>
<td>0.99</td>
</tr>
<tr>
<td>PA$_{30}^\prime$</td>
<td>Malaxed: extracted after 30 minutes</td>
<td>119073</td>
<td>0.799</td>
<td>0.98</td>
</tr>
</tbody>
</table>

Figure 1. Variation of torque with apparent viscosity during malaxation
References


volatiles and phenolic compounds in virgin olive oil. Journal of Agricultural and Food Chemistry, 56, 10048-10055.
