

## Combined Continuous Machine to Condition Olive Paste: Rheological Characterization of Olive Paste

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Food matrix structures are becoming more and more investigated from a rheological characterization point of view because this influences their behavior in the food process phases. In this study, a brief characterization of olive paste is attempted according to their rheological profile. Moreover, the influence of new technologies to prepare the olive paste on the rheological characteristics of olive paste is presented. The small olive oil droplet size, solid volume fraction, and the strong interactions of droplet interfaces are significant factors that change the rheological profile of the olive paste during the olive oil extraction process. The process involved, in fact, favors a decrease of viscosity of the initial matrix used which occurs mainly with the passage of the oil paste in the malaxer reaching an adequate viscosity. However, the viscosity of processed olive paste depends on the raw material, according to various factors such as the pedo-climatic conditions, agronomic techniques, the ripening index of the drupes, cultivars used, processing technologies that have a strong influence on the solid/liquid ratio. Therefore, the objective of the present study is to investigate the rheological properties of the olive paste when a combined heat-exchange and microwave machine are used.

### 1. Introduction

New technologies have been tested to prepare the olive paste. The introduction of ultrasounds, microwaves, pulsed electric fields, heat exchangers are among the new technologies studied for applications in the olive oil extraction industry (Leone et al., 2018; Caponio et al., 2019; Romaniello et al., 2019; Servili et al., 2019; Tamborrino et al., 2019b; Tamborrino et al., 2020b). An industrial-scale plant has recently been developed to study a new combined machine including an innovative heat exchanger and a microwave module, to conditioning in continuous mode, at industrial scale, the olive paste for extra-virgin olive oil extraction. The goal of these transformations that take place during a unit operation in food industry is to optimize, on one hand, economic and energetic aspects and, on the other hand, consistency, taste, aroma and nutritional value of the final product (Bianchi et al., 2013; Leone et al., 2015; Perone et al., 2017; Tamborrino et al., 2019a; Catalano et al., 2020; Tamborrino et al., 2020a). About process optimization in the olive oil mills, in terms of maximum extraction yield with excellent quality, it is essential to understand how the components of the olive paste are transformed by the technological treatments (type, size and spatial distribution of particles, interactions between particles) and how the flow behavior of the olive paste can be related to the treatment conditions (Masella et al., 2008). Olive paste is a coarse solid-liquid mixture consisting of two distinct liquid phases (oil and water) and an extremely heterogeneous solid phase. Processing conditions strongly influence its physical and chemical characteristics (relative amounts of different phases and their interactions, degree of emulsion, and solid fragment dimensions) which in turn influence its rheological properties (Tamborrino et al., 2014; Petrakis, 2006). In food processing, rheology plays a key role because it assesses the quality of raw materials, predict material behavior during processing, and meeting storage and stability requirements (Ibarz and Barbosa-Cánovas, 2003; Toledo et al., 2018). To have a wider understanding of the rheology of olive paste, it is first necessary to analyze the chemical composition of the raw material and therefore of the drupe (fruit of the olive tree). Pedo-climatic conditions, agronomic techniques, degree of ripeness of the drupe,

cultivars used, processing technologies adopted for the processing of olives, state of preservation of the drupe, are the most important variables in defining the quality of the raw material. In general, it is found that each olive paste phase shows its rheological behavior, but if they are considered as a whole they can assume very complex characteristics. Olive oil, in fact, shows Newtonian behavior (Ayadi et al., 2009), while the vegetation water and solid particles are non-newtonian fluids. The olive paste assumes non-Newtonian behavior (Di Renzo & Colelli, 1997; Tamborrino et al., 2014 Romaniello et al., 2017). More specifically, it is a shear-thinning fluid, or pseudoplastic, characterized by a reduction in viscosity as the shear rate increases (Boncinelli et al., 2009; Boncinelli et al., 2013). Catalano et al. (2001) and Formato et al. (2005) reported that the power-law mathematical model can accurately describe the rheological behavior of olive paste concluding that it behaves as a time-independent shear-thinning (pseudoplastic) fluid characterized by a shear limit. Thus, viscosity is an important parameter for characterizing the behavior of olive paste when it is under the influence of various technological process parameters. It varies from the inlet to the outlet of the oil extraction line and generally can be assessed through viscosimetric measurements (in-line or off-line), or it can be based on the experience of the operator; however, it is not an objective parameter of assessment. The objective of this paper is to investigate on rheological characteristics of the olive paste when conditioned with a new technology consisting of a heat exchanger and microwave continuous system (HE-MW). So far as can be determined, reports about the rheological behavior of olive paste when changing the processing conditions are scarce, they always need in-depth analysis, especially when new processes are introduced. Such studies are of practical importance as they help to understand the behavior of the olive paste when it is under the influence of different technological process parameters. It could also serve as a significant role in the analysis of flow conditions to propose a mathematical model that can correctly describe how the viscosity of the olive paste varies during the various stages of its processing.

## 2. Materials and Methods

### 2.1 Industrial olive mill and experimental equipment with HE and MW continuous system

Experimental tests were carried out in an industrial olive mill located in Corato (Puglia region – Italy). Figure 1 shows the flow chart of the main operations involved in the extraction process of the mill. The extraction process began with a defoliator (mod. Morgana, Mercuri, Rosarno (RC), Italy) and a washing machine (mod. LVST, Vitone Eco S.r.l., Bitonto (BA), Italy) to remove leaves and clean the drupes. A hammer crusher (mod. FR 25, Vitone Eco S.r.l., Bitonto (BA), Italy) was used to mill the cleaned olives, and the obtained paste was then moved toward the combined continuous machine (HE-MW). The paste is then malaxed (malaxer, mod. GR 700, Vitone Eco S.r.l., Bitonto (BA), Italy), before undergone a solid-liquid separation (2-phase decanter centrifuge, mod. V2, Vitone Eco S.r.l., Bitonto (BA), Italy) and a subsequent liquid-liquid separation (vertical centrifuge, mod. V2700 Mr.Oil, Vitone Eco S.r.l., Bitonto (BA), Italy).

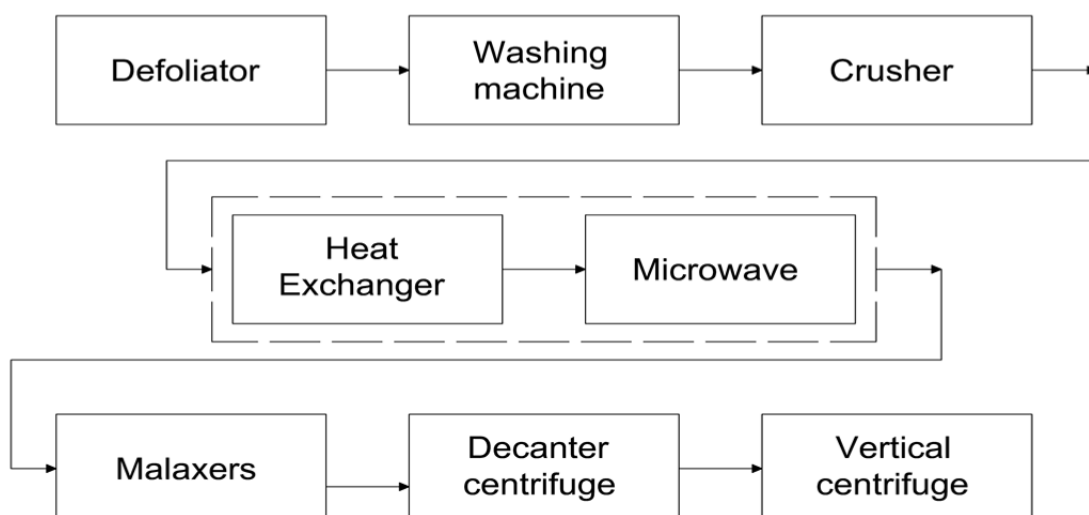


Figure 1: Flow chart of the extraction process mill.

The combined continuous machine consists of two sections assembled in series. The first section is constituted by a tube-in-tube heat exchanger, in which the olive paste flows in the inner tube, and hot water in

the external jacket, in a counter-current direction. The inner tube is equipped with a rotating helix moved by an electric motor driven by a mechanical speed reducer. The inner helix has the main aim of improving the heat transfer, reduce the operating pressure, and gently kneading the olive paste. Connected to the outlet of the heat exchanger, it was placed the microwave section. It was made of a cavity resonator, in the middle of which is located a polypropylene tube for the microwave treatment of the paste flowing in it. The control of this new conditioning technology happens through a PLC that allows adjusting the output temperature of the paste in the function of its inlet temperature and mass flow rate.

## 2.2 Experimental design

Experimental trials were carried out processing the olive paste of three different varieties in an industrial olive oil mill by using the HE-MW continuous system. Data were acquired concerning viscosity measurements carried out on olive paste before and after the HE-MW treatment. The collected data consist of apparent viscosity values, the related shear rates, and the composition of the olive paste. These data were interpolated using a power-law model, whose parameters were determined by means of linear regression in a bi-logarithmic scale. The test carried out by using olive fruit of the varieties Cima di Bitonto, Frantoio and Peranzana (*Olea europaea* L.) having a maturity index of 2.1 (Cima di Bitonto), 2.3 (Frantoio) and 2.5 (Peranzana). The maturity index was detected using the method reported in Uceda et al. (1975).

Five consecutive oil extraction tests were carried out for each variety, using homogeneous batches of 700 kg of olives.

The experimental design is summarized as follows:

- Before treatment, where the olive paste was obtained and sampled after the crushing stage;
- After treatment, where the olive paste was obtained and sampled after the HE-MW conditioning, conducted by flowing in a continuous way the olive paste through the heat exchange and MW system and setting the output temperature of 27 °C.

For each test, three samples of olive paste were taken, before and after treatment, for the rheological measurements.

## 2.3 Rheological measurements

Viscosity determinations were carried out using a rotational rheometer (model DV2-HBT Brookfield Engineering Laboratories, Inc., Stoughton, MA, USA) equipped with a disc spindle (model RV/HA/HB-4; Brookfield DVII + Brookfield Engineering Laboratories). Viscosity measurements were carried using 500 mL of olive paste, loaded into a 1000 mL glass container conditioned at 27 °C in a thermostatic bath. The apparent viscosity was taken at rotational speeds from 0.5 to 200 rpm (Tamborrino et al., 2014). Numerical conversion values were used to convert the torque-speed and scale readings into shear stress–shear rate relationships. An empirical power-law model was used to calculate the apparent viscosity and flow behavior index from the shear rate (Eq. (1)).

$$\eta_{app} = k\gamma^{n-1} \quad (1)$$

where  $\eta_{app}$  is the apparent viscosity,  $\gamma$  is the shear rate ( $s^{-1}$ ),  $n$  is the flow behavior index (dimensionless),  $k$  is the consistency index ( $mPa s^n$ ). The results are expressed as mean of three replications.

## 2.4 Statistical analysis

The machine learning and statistic toolbox of MATLAB<sup>®</sup> was used to process the experimental data. The significance among means of group of data was detected by the one-tailed t-test hypothesis test ( $p < 0.05$ ).

## 3. Results and discussion

### 3.1 Influence of the HE-MW continuous system on the rheological characteristics

Figure 2, 3, and 4 report the apparent viscosity in a bi-logarithmic scale of the paste as a function of the strain rate, for olive pastes respectively of Cima di Bitonto, Frantoio e Peranzana varieties. A lower apparent viscosity is observed in all the cases of HE-MW treatment. The points were fitted by means of the power-law model as reported in 2.3. Through this model, the most important parameters for pseudoplastic fluids were obtained: the consistency index ( $K$ ), and the flow behavior index ( $n$ ). These two parameters can be deduced by the power function in Figure 3. In particular, the base represents the consistency index (in  $Pa s^n$ ) and the exponent is  $n^{-1}$  (dimensionless), from which is easily obtainable the flow behavior index. It is worth noting that the power-law model returned an optimal approximation of the rheological behavior of the paste, confirmed by the high values of the coefficient of determination ( $R^2$ ), which is always above the 99 %, exception made for

the Peranzana cultivar before HE-MW treatment. In all cases the, the apparent viscosity for each strain rate value shows a significant difference between before and after HE-MW treatment.

The curves depicted a typical pseudoplastic behavior, particularly the shear-thinning one. For all those fluids with molecular dispersions or with asymmetrical particles, which present a disordered state, as the shear stress increases, the viscosity decrease because the particles are oriented towards the direction of propagation of the applied force and therefore always align themselves more by reaching a state of molecular order. Heterogeneous systems, containing a dispersed phase (Van Hecke et al., 2012) like olive paste, are characterized by a typical shear-thinning behavior. When the hydrodynamic forces during fluid shear become sufficiently high, the interparticle bonds, which usually occur at rest, break down and a decrease in viscosity results. Less resistance to flow during shear is then offered. The rheological behavior is well represented by the evaluation of the viscosity, as velocity gradients and consequently the motion of suspended solid particles and heat transfer are affected (Amirante & Catalano, 1995).

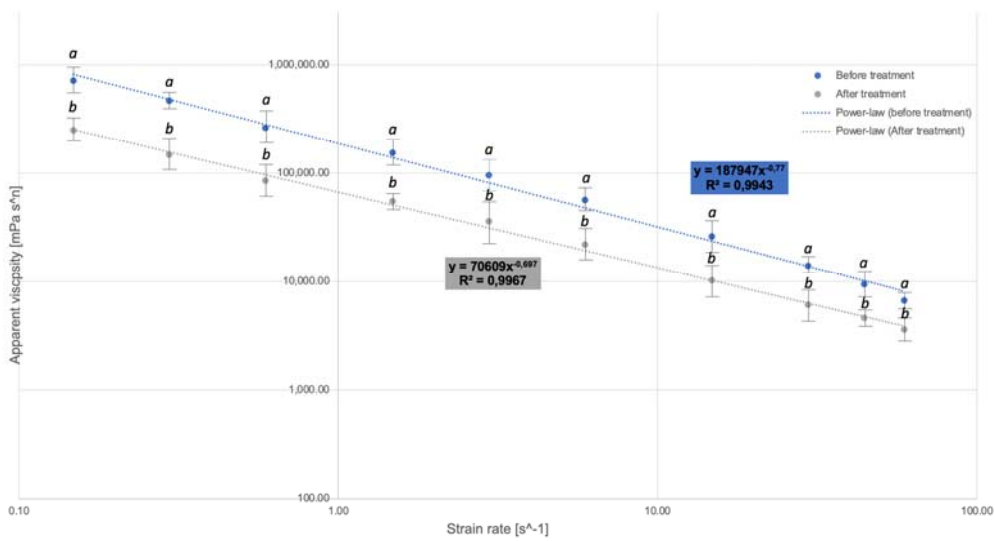


Figure 2: Apparent viscosity vs strain rate in log-log scale before and after the combined treatment for olive pastes of Cima di Bitonto variety

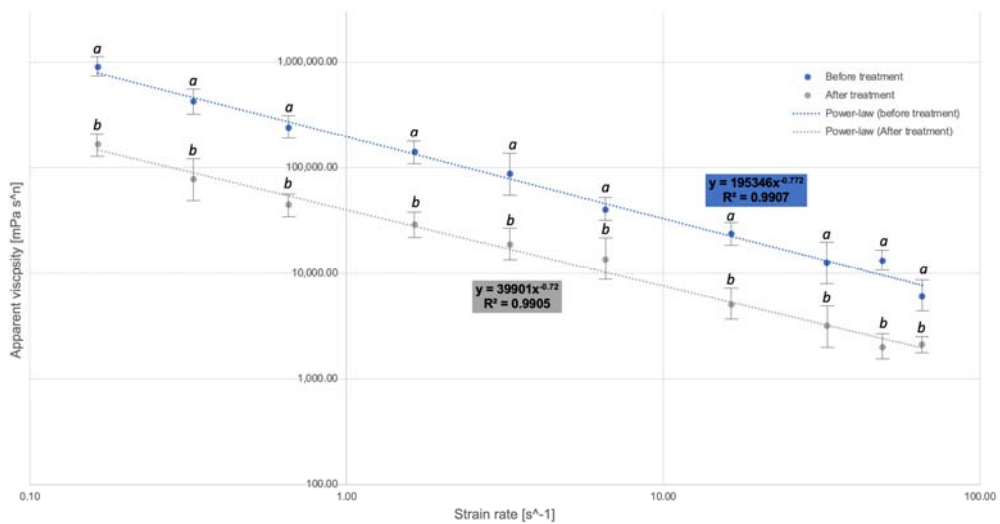


Figure 3: Apparent viscosity vs strain rate in log-log scale before and after the combined treatment for olive pastes of Frantoio variety

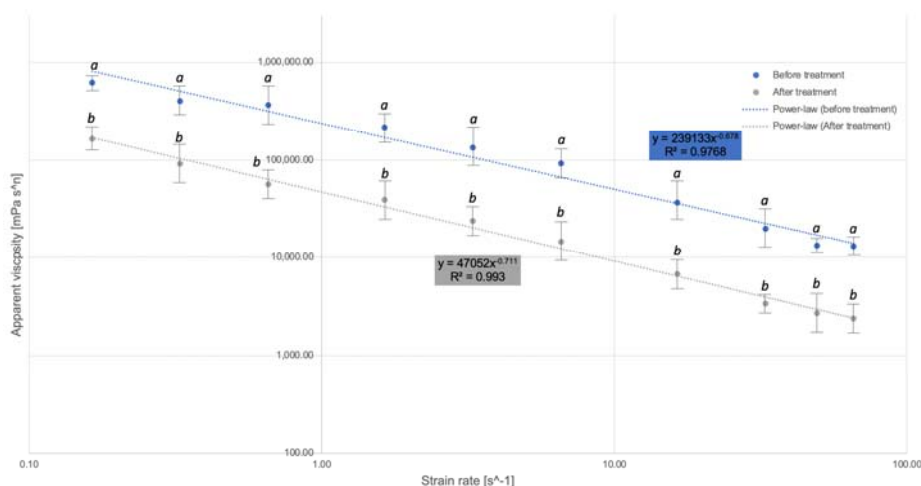


Figure 4: Apparent viscosity vs strain rate in log-log scale before and after the combined treatment for olive pastes of Peranzanao variety

The significant reduction of the consistency index, and therefore of the apparent viscosity after the treatment, maybe due to the combined effect of temperature increasing and physical damage of vegetable tissues by means of microwave action. In fact, the heat exchanger raised the temperature of the paste and operated a kind of kneading through the inner mixing helix, producing an additional effect on the reduction of the paste viscosity. In addition, the MW, with its volumetric way of heating, heated up the moisture present in the matrix until its evaporation, generating high pressure on the cell wall and causing its ruptures.

The significant reduction of the viscosity of the olive paste thanks to the combined machine leads to two positive aspects: (i) allows to greatly reduce the malaxation time and consequently allows to improve the olive oil quality; (ii) increases the decanter's extractability and consequently the oil yield.

#### 4. Conclusions

A rheological investigation on olive paste, of different cultivars (Cima di Bitonto, Frantoio and Peranzana), conditioned with a new technology coupling a heat exchange and microwave machine (HE-MW) was carried out in this study. Using a HE-MW machine a significant reduction of the viscosity and the consistency index is observed. The use of a machine capable of treating the olive paste before the passage in the malaxer machine, causing a significant reduction in viscosity, is positively evaluated since the reduction in viscosity appears to be an important parameter for the subsequent centrifugation phase. In fact, one of the main physical parameters influencing the centrifugation process is the olive paste viscosity, which affects the velocity gradients and, therefore, the motion of suspended solid particles that must be removed. This machine allows to reduce the viscosity by exploiting the thermal and mechanical action due to the particular constructive devices, such as the internal spiral of the heat exchanger. This allows to reduce the malaxing times which would normally have allowed a reduction in viscosity. Investing in rheological changes when new machines are incorporated into food processes is essential for process and plant optimization.

#### Acknowledgments

All authors contributed equally to this work.

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