

# Industrial valorization of *Quercus cerris* bark: Pilot scale fractionation



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## ABSTRACT

Cork-rich *Quercus cerris* bark collected from Turkey was fractionated in a laboratory and in a pilot-scale equipment to obtain cork and phloem fractions. After a primary trituration as a field post-harvest operation, the cork-rich bark granules were mainly concentrated in the big granules (>12 mm) indicating different mechanical properties of cork and phloem. The smaller granules (<4 mm) could be fractionated using water flotation by separating a floating cork fraction. The pilot-scale fractionation consisted of a mechanical grinding of the whole bark fractions followed by a granulometric and a densimetric separation. The operation was quite efficient and as a result, pure cork (8.4% wt) and cork-rich (18.5% wt) fractions were obtained. The colour analysis and FTIR spectroscopy showed the separation efficiency of the cork fractions. The results show the potential of *Q. cerris* bark for production of valuable cork fractions and the efficiency of combined granulation and fractionation.

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## 1. Introduction

*Quercus cerris* L., the Turkish or oriental oak, is a native tree species in the south and central Europe, Balkan Peninsula, Asia Minor, western Syria and Lebanon. This species has its major distribution in Turkey where it grows naturally in almost all parts of the country except in the easternmost regions (Kasapligil, 1981). The *Q. cerris* trees in south-eastern Turkey have a thick bark with a large rhytidome of several superposed periderms that contain substantial amounts of cork (Şen et al., 2011a). The cork layers are conspicuous to the naked eye, making up patches of variable radial width from about 1 to 10 mm.

Cork is a cellular material with structural and chemical features that give it an interesting combination of properties (Pereira, 2015). Cork is the raw material for an economic important industrial chain producing several cork products of which the wine cork stoppers are known worldwide and are the economic pillar of the industry (Pereira, 2007). The commercial cork that is used for wine stoppers is obtained from the cork oak (*Quercus suber* L.), a species with a geographical distribution restricted to the western Mediterranean basin and a total production limited to about 350 thousand tons (Pereira and Tomé, 2004).

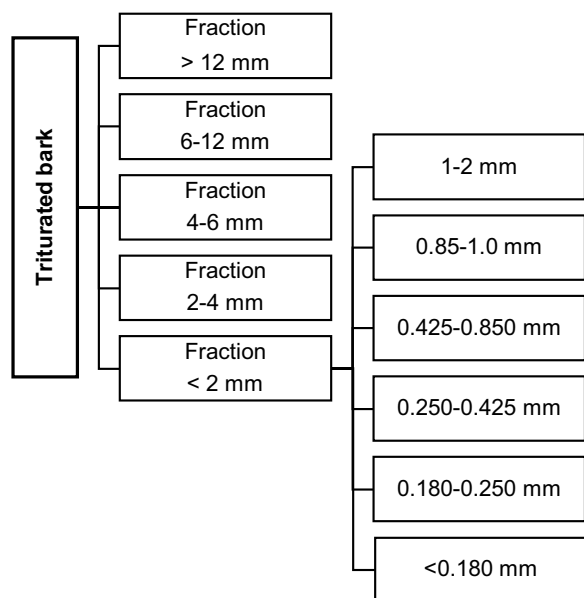
The idea of using cork-rich barks as an additional source of cork is tempting to enlarge the raw-material supply to the industry. *Q.*

*cerris* bark is an interesting candidate and as such has been the subject of recent studies. The cellular structure and chemical composition of *Q. cerris* cork proved to be similar to those of *Q. suber* cork (Şen et al., 2010, 2011b), as well as its thermal behaviour (Şen et al., 2012a, 2014). However *Q. cerris* bark has structural features i.e. anatomical composition, very different from those of *Q. suber* due to their different bark types (Sen et al., 2010). While *Q. suber* bark has no rhytidome and the cork forms a continuous and thick layer around the tree stem that may be removed and processed into cork products e.g. natural cork stoppers, in the case of *Q. cerris* the cork layers are included in a thick rhytidome with several periderms and are therefore separated by phloemic layers. This structure requires that bark has to be trituated and then undergo a fractionation process to efficiently separate the cork from the other lignocellulosic phloemic tissues. For the high value applications of cork e.g. for wine stoppers, it is necessary to have a cork-only fraction with granules of homogeneous granulometry which conditions the fractionation procedure. For instance, a laboratorial grinding and sieving followed by flotation was not sufficient to separate a pure cork fraction (Şen et al., 2012a).

It is therefore important to develop a fractionation methodology that could be adapted to industrial operational conditions and that yields cork granules with dimensions and purity adequate for the cork industry. The demonstration of the technological feasibility for separation of a pure cork fraction that may be used for high value agglomerated cork products is a fundamental step before further efforts towards *Q. cerris* bark valorisation. This was done here, where approximately one ton of *Q. cerris* bark was collected in the

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**Fig. 1.** Schematic granulometric fractionation of the *Quercus cerris* bark after the post-harvest trituration obtained with laboratorial sieving equipment.

forest, trituated and fractionated in industrial pilot scale equipment, allowing to determine fraction yields and characteristics.

## 2. Material and methods

### 2.1. Samples

The bark of *Q. cerris* was collected manually from the stumps of recently harvested trees at the Amanos Mountains in the Dörtöl province near the city of Hatay (Antioch), in the south of Turkey. The trees were cut to be used as firewood and for small-scale construction applications. The sampling site was a natural *Q. cerris* coppice stand with approximately 60-year-old trees.

### 2.2. Post-harvest trituration

In order to facilitate transport, the bark samples were coarsely trituated locally in a hammer-type mill designed to granulate biomass for essential oil extraction. The trituration yielded bark pieces with varying dimensions, amounting to approximately 980 kg that were collected in four industrial big bags and shipped to our laboratory in Lisbon, Portugal.

### 2.3. Laboratory fractioning

The result of the post-harvest trituration was evaluated upon arrival by fractionation in the laboratory. The trituated bark was manually fractionated using a vibratory sieving device with sieves with the following sizes: 12 mm, 6 mm, 4 mm and 2 mm. The fraction that passed the 2-mm sieve was further screened using a vibratory sieving apparatus (Retsch AS 200basic) with the following sieves: 1 mm, 0.850 mm, 0.425 mm, 0.250 mm and 0.180 mm. After sieving, the mass retained on each sieve was weighed and the corresponding mass fraction yields were determined (Fig. 1). Three independent samples with approximately 15 kg were taken and the result is shown as the mean. The moisture content of the bark particles was determined and averaged 12%.

### 2.4. Laboratory separation by water flotation

A water flotation experiment was used to test its efficacy to obtain cork fractions with higher purity after laboratory scale fractioning. The fractions with 2–4 mm, 1–2 mm and 0.85–1.0 mm granules obtained from the initial post-harvest trituration were used. Approximately 30 g of each fraction were separated by density difference in distilled water with 15 min settling time after an initial quick mixing. The separation of the samples resulted in a floating fraction of cork-enriched granules (subsequently named supernatant) and a submerging fraction of phloem-enriched granules (subsequently named sediment). Both fractions were separated, dried and weighed. Two replicate experiments were performed for each fraction.

### 2.5. Pilot-scale fractioning

The trituated bark samples were further fractionated in the pilot plant for cork processing of Cincork, Portugal. The pilot plant is designed to process the present commercial cork raw-materials and includes two cutting mills, sieve separation and densimetric tables; the equipment requires a minimum raw-material feedstock of approximately 600 kg.

The *Q. cerris* bark that underwent the pilot scale fractionation amounted to 680 kg. A stepwise fractionation process was followed: the bark samples were passed through two mills, screened by a vibratory screens into five dimensional fractions (5–7 mm, 3–5 mm, 2–3 mm, 1–2 mm, 0.5–1 mm) followed by density separation on gravimetric separators into two density fractions (low density and high density) as schematically shown in Fig. 2. Fines were separated after each mill, and the oversized particles were recirculated in the second mill. After density separation, and following the practice used for the commercial cork, the granules were labelled as good (low-density and that can be directly used by the cork industry) and weak (high-density and rejected).

### 2.6. Bulk density

The bulk densities of the fractionated bark samples were determined for each sieve fraction using a cylindrical glass container (25 mm diameter × 29 mm height) as the ratio of mass sample in the container to its volume.

### 2.7. UV/VIS spectroscopy and colour measurement

The reflectance spectra of the laboratory-fractionated and pilot scale-fractionated bark fractions were measured with a Minolta CM-3630 (d/0°) spectrophotometer at wavelengths between 360 and 720 nm with a 10 nm spectral resolution (Şen et al., 2012b). The CIELAB colour parameters  $L^*$ ,  $a^*$  and  $b^*$  were determined for each granulometric fraction obtained by the two fractionation methods. Total colour differences ( $\Delta E^*$ ) between cork-rich and phloem-rich bark granules after pilot scale fractionation were calculated using the following formula (Bekhta and Niemz, 2003; Mononen et al., 2005; Fan et al., 2010):

$$\Delta E = (\Delta L^2 + \Delta a^2 + \Delta b^2)^{1/2}$$

The conversion of a reflectance spectrum into an absorbance spectrum was made by applying the Kubelka-Munk equation where  $R$  is the measured reflectance value,  $k$  is the light absorption coefficient and  $s$  is the light scattering coefficient.

$$\frac{k}{s} = (1 - R)^2 / 2R$$

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