



## Short communication

## Potential biofuel from liquefied cork – Higher heating value comparison



Maria Margarida Mateus, João Carlos Bordado, Rui Galhano dos Santos\*

CERENA, Departamento de Engenharia Química e Biológica, Torre Sul, Instituto Superior Técnico, Av. Rovisco Pais, 1049-001 Lisboa, Portugal

## HIGHLIGHTS

- Comparison of HHV between cork liquefied products.
- Improvement of calorific values of cork liquefied products.
- Organic extract of with HHV comparable to that from natural gas.

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## ABSTRACT

Herein the improvement of liquefied cork for the production of a bio-oil, which can be faced as a potential bio-fuel, is presented. Cork liquefied products were therefore subjected to water extraction to separate the most polar compounds, in particular, those arising from cellulose and hemicellulose hydrolysis. After drying the organic extract, the higher heating value (HHV) was estimated. The results clearly revealed that the extraction process led to a bio-oil with an upgraded HHV indicating, thus, this refined product is more likely to be used as a biofuel for hot steam furnaces, for example, than the liquefied cork or even cork in its primitive form.

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## 1. Introduction

The high demand for the fossil fuels allied with the growing concerns about the effects of gas emissions and sustainable alternatives energy sources have gained attention from researchers all over the globe. Comparatively with fossil fuel, biomass energy has countless advantages such as its renewable nature, relative abundance (annually the produce amounts can be set at around eight times higher than world fossil energy consumption), carbon neutral ability, low sulfur emission during combustion, and its easy transportation and storage. Biomasses are the putative resource for the production of new liquid fuels, synthetic gas, hydrogen, solid fuel, and valuable chemicals [1].

Being a well-known concept, lately, liquefaction of biomass, in particular, lignocellulosic residues, is a process that has been largely investigated [2–5]. Overall, this process consists in the depolymerization and solubilization of biomass at high temperatures [6–9].

In the last couple of years cork by-products, have been reported as raw materials for liquefaction in polyhydric alcohols by conven-

tional [3], microwaves mediated [2] and ultrasounds-assisted methods [9]. Actually this waste, proved to be suitable for the production of biomaterials based on the polyols obtained from its liquefaction [10,11]. Chemically cork is mainly composed by polysaccharides, lignin, suberin, and extractives (20, 22, 40, and 15 wt.%, respectively) [12,13].

Cork is an environmentally friendly and sustainable biomaterial harvested from Cork oak tree (*Quercus suber* L.). The bark is stripped with the preservation of the tree allowing a new bark to regrow. That process happens every 9–14 years, with the improvement of cork quality, until the tree is approximately 200 years old [14]. The cork industry generates a residue considered problematic, the so called “cork dust”, an important fraction of the total amount of wastes with low commercial value. The inclusion of that residue in cork products is only possible under small amounts, therefore not incorporating significant volumes when compared to its production. Currently, it is mostly applied as fuel for burning in furnaces.

Biomass properties are usually classified into three main classifications: physical, chemical, and thermal. One of the most important properties within the thermal ones is the higher heating value (HHV), which is, probably, the characteristic that better defines the suitability of solid biomass to be used as a fuel [15].

\* Corresponding author.

E-mail address: [rui.galhano@ist.utl.pt](mailto:rui.galhano@ist.utl.pt) (R.G. dos Santos).

HHV is a measure of the chemical energy released during combustion of biomass [16].

Within this context, we envisaged to study the liquefaction of cork to produce a bio-oil that can be further used as bio-fuel. In this study, we disclosed the work conducted concerning the use of liquefaction to produce the referred bio-oil and its HHV analysis.

## 2. Materials and methods

### 2.1. Materials and chemicals

Cork powder from corks rectification operation was used for the tests. This biomass for liquefaction was dried in an oven set to 120 °C until no change in weight was observed. The reagents used were chemical grade (Sigma–Aldrich).

### 2.2. Liquefaction procedure

A glass reactor was loaded with a mixture of solvents, 660g of 2 ethyl-hexanol and 220g of DEG. 33g *p*-Toluene sulfonic acid was added along with 220g of cork dust. The mixture was then stirred at 160 °C for the 90 min. After liquefaction, the reactor was allowed to cool at room temperature [9].

### 2.3. Measurement of liquefaction extent

The conversion was gravimetrically evaluated based on the residue content (unreacted raw material). The reaction mixture was diluted with acetone and then filtered and washed with acetone and methanol. The filtrate was evaporated under reduced pressure by rotary evaporator system at 40–50 °C temperatures to recover the bio-oil, which was then kept for further analysis and extraction. The obtained solid residue was then dried in an oven set to 120 °C until no change in weight was observed. The liquefaction yield was calculated according to Eq. (1).

$$\text{Liquefaction yield (\%)} = \left(1 - \frac{M_R}{M_C}\right) \times 100 \quad (1)$$

where  $M_C$  is the initial mass of cork,  $M_R$  the mass of the residue obtained.

### 2.4. Extraction

The bio-oil was extracted with distilled water (10 × 500 mL); the combined fractions were evaporated under reduced pressure affording the aqueous extracted.

### 2.5. Attenuated total reflectance (ATR) analysis

ATR spectra were collected using an ATR accessory. Spectra were collected on Thermo Nicolet Nexus instrument (128 scans with a resolution of 4 cm<sup>-1</sup>).

### 2.6. HHV determination

The calorific values were determined with an LECO AC500.

## 3. Results and discussion

The work conducted was divided into two main steps as indicated in Fig. 1. A former stage is the cork liquefaction. This first step was conducted with no problems whatsoever leading to the expected products in quantitative yield (>90%). Afterward, filtration, washing with acetone and concentration were performed and a blackish crude was obtained. The latter step consisted in

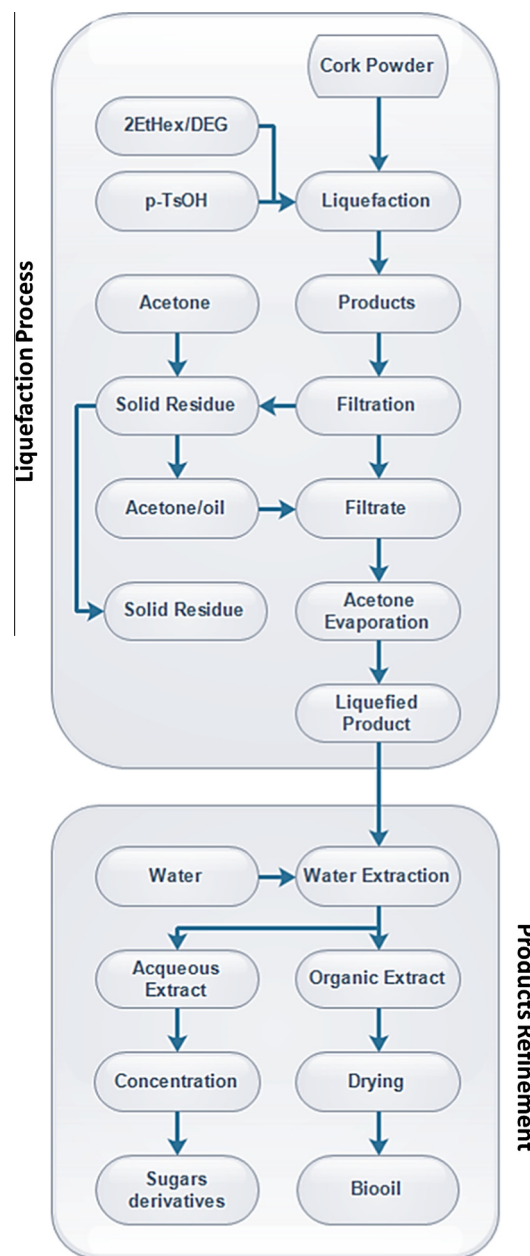


Fig. 1. Cork liquefaction and its further refinement – procedure flowchart.

the water extraction of the most polar constituents of the concentrated oil. After extracting several times with distilled water until the aqueous layer becomes almost clear, the organic extract was dried to remove any residual water leading to a dark brown oil. On the other hand, the aqueous extracts were, then, combined and concentrated under vacuum affording an amber colored syrup/gel.

### 3.1. ATR-FTIR analysis for the liquefied products

The normalized ATR-FTIR absorption spectra of each extract as well as of the reaction crude and the starting raw material were used to screen the functional groups of the obtained products, as presented in Fig. 2, demonstrating the liquefaction success of cork. A strong OH bond vibration is seen at 3500–3000 cm<sup>-1</sup> and two prominent C–H stretching vibration 2917 and 2850 cm<sup>-1</sup>. Clearly, in the aqueous extract (Fig. 2D) the O–H stretch band at

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