



Study on the simultaneous lipids transesterification and cellulosic matter liquefaction of oleaginous seeds of *Pistacia atlantica*



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ABSTRACT

In this work, simultaneous transesterification and thermochemical liquefaction of *Pistacia atlantica*, an oleaginous plant, using ethanol as a reagent and a solvent was studied. Experiments were performed under temperatures ranging from 300 to 450 °C and pressures ranging from 40 to 85 bar. Effects of temperature and pressure on conversion rate, bio-oil and gas yield were studied. The maximum conversion rate and bio-oil yield were 82% and 56% respectively and were obtained at 375 °C and 43 bar. Gaseous products were mainly composed of carbon oxides at low temperatures. At 450 °C the percentage of light hydrocarbons increased to more than 40% due to secondary cracking of bio-oil. Bio-oil was compared to conventional biodiesel and it was concluded that products have higher viscosity and oxygen to carbon ratios and lower hydrogen content and heating values. Products obtained at 300 °C and 46 bar recorded the lowest viscosity that reached 6.8 mPa·s.

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1. Introduction

The rapid increase in the world energy demands, associated with the depletion of fossil energy resources and their impact on the environment, requires the use of renewable energy. As one of the used renewable resources, biomass is the fourth largest source of energy in the world providing about 14% of primary energy [1], and the most promising resource as it is carbon neutral and abundant [2]. Biomass designates any organic matter derived from plants and animal materials such as wood from forests, crops, seaweed, material left over from agricultural and forestry processes, and organic industrial, human and animal wastes [3].

The second generation biofuels, which are derived from non-food lignocellulosic biomass, are getting attention especially that they don't compete with food supply [4]. Oleaginous plants are vegetable oil-producing plants, which have a wide range of chemical compositions and able to produce more than 20% of their biomass as triglycerides [5].

There are several methods available to convert biomass into useable form of energy. The four main routes of conversion are: chemical conversion (transesterification), thermochemical (such as direct combustion, pyrolysis, gasification, liquefaction),

biochemical/biological (such as anaerobic digestion, fermentation and enzyme catalyzed transesterification) and mechanical extraction [6].

To date, more than 95% of biodiesel is produced using edible oleaginous plants by extracting their oil before undergoing a transesterification process [7]. The extraction phase is energy and time consuming process and it increases production costs. In the case where residues, that can constitute up to 80% of the pressed fruits, can be used as animal food source, protein cake can enhance the economic viability of the process. However, in some cases, where the animal feeding with residues is not feasible (low nutrition grade, toxicity, high costs of packing and transportation), alternative methods should be adopted. Several studies investigated in-situ transesterification of feedstock, this process consists on a simultaneous oil-extraction and transesterification using a single step method like using microwaves and ultrasonication [8–10], or reactive extraction [11]. Despite the efficiency of these methods, they do not offer solutions for non-edible residues that should undergo other conversion processes. Thus, an in situ transesterification coupled to a thermochemical process could be the most suitable solution to maximize the conversion rate of non-edible oleaginous biomass.

Among the different processes, thermochemical liquefaction offers the possibility of economizing energy due to its high humidity tolerance, low reaction temperature and residence time [12]. In fact, this process converts the biomass to bio-oil in presence of a

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solvent, with or without a catalyst and under high pressure and temperature ranging between 250 °C and 400 °C [12–14], and with a residence time between 5 and 60 min [15]. Furthermore, the presence of solvent prevents the cross-linking and reverse reactions by diluting the products.

Supercritical ethanol is a very efficient thermochemical liquefaction solvent [12,14], which could be attributed to its hydrogen donor capability. On the other hand, supercritical alcohol acts as a transesterification reactant [16,17]. The hydrogen bonding between the alcohol molecules decreases dramatically in the supercritical region, allowing alcohol molecules to act as an acid catalyst in the transesterification reaction [18]. In addition, liquid products obtained in supercritical alcohol liquefaction and transesterification, form a single phase, whereby biocrude can be recovered by simple alcohol drying.

Regarding these characteristics, the present study aims the conversion of total biomass of a non edible plant, *Pistacia atlantica*, by extracting the oil content from the kernel, and transforming lignocellulosic materials to liquids, through simultaneous application of supercritical transesterification and thermochemical liquefaction. Reaction took place at different temperatures and reaching pressures at sub/supercritical medium. Ethanol was used as a solvent because of its hydrogen donor capability, with a relatively low critical point (243 °C, 6.39 MPa). A sufficient amount of bio-oil was produced, which allowed the measurement of viscosity and heating value. In addition, reaction products were analyzed in order to understand the effects of both pressure and temperature on the reaction and on the liquid products properties related to its use as a diesel fuel extender.

2. Materials and methods

2.1. Raw materials

Pistacia atlantica is a deciduous tree of the family Anacardiaceae, semi-evergreen and growing to 20 m. Its seeds are rich with lipids: the oil content in ripe fruits is 32.8–45% [19], and the most predominant fatty acid (FA) in oil is oleic acid with a mean value of ~51% [20].

Pistacia atlantica used as the biomass feedstock in this study was collected from the mountains in Lebanon. The moisture content was 6.2 wt% and the oil content, determined using a soxhlet extractor, was 45%.

Before being used in the experiments, the biomass feedstock was dried in an oven at 105 °C for 12 h.

2.2. Experimental setup

The experiments were conducted in a 1000 ml cylindrical reactor designed by ATOMECA, SACMO society. The reactor is made of stainless steel and can operate at a maximum temperature and pressure of 450 °C and 190 bar respectively, and an installed heating capacity of 20 kW. The reactor set-up is shown in Fig. 1.

The reactor is equipped with an injection system of inert gas (N₂); manual valves are used to control N₂ feed. A manometer and two thermocouples are introduced in the reactor to measure its pressure and its bulk and wall temperatures respectively. In each experiment, the reactor was loaded with a constant biomass/ethanol ratio of 1/2.5. Then, the reactor was purged for 15 min with nitrogen to remove the inside air. After that, the reactor was heated to desired reaction temperature (300 °C, 375 °C or 450 °C), reaching different maximum pressures at sub and supercritical medium. Reaction time was set to 60 min. Once the desired reaction time was elapsed, the reactor was cooled down to room temperature.

Each experiment was realized in three replicates, mean value was adopted and standard deviation was used to represent the standard error.

2.3. Extraction and product separation procedures

The procedures for separation and extraction of reaction products are shown in Fig. 2. Once the reactor was cooled to room temperature, the gaseous fraction was taken from the upper vent of the reactor to be analyzed, and then the autoclave was opened. The solid/liquid products were extracted from the reactor. The solid residue was washed with acetone and then filtered with a pre-weighed filter paper. Then, the solid residue was dried at 105 °C in an oven for 24 h. The collected liquid was distilled to remove the ethanol and acetone. After distillation, light fraction was distilled with acetone/ethanol mixture and the remaining heavy fraction (bio-oil) was recovered for analysis.

Solid residue (SR) and bio-oil were weighed for the calculation of product yields.

$$\text{Bio - oil yield (wt\%)} = \frac{\text{mass of bio - oil (g)}}{\text{mass of atlantic pistacia (g)}} \times 100 \quad (1)$$

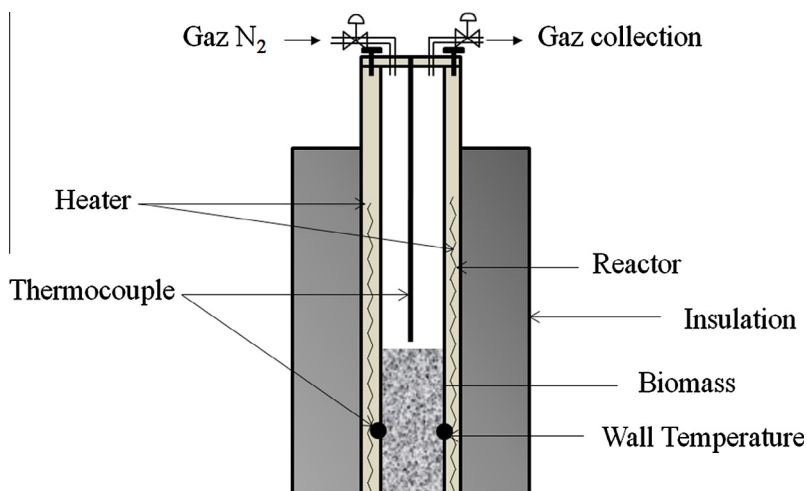


Fig. 1. Schematic diagram of reactor set-up.

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