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Evaluation of *Eremurus spectabilis* for production of bio-oils with supercritical solvents

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ABSTRACT

Eremurus spectabilis samples were liquefied in organic solvents (methanol, ethanol and acetone) with (sodium hydroxide and ferric chloride) and without catalyst in a cylindrical reactor at temperatures of 270, 290 and 310 °C under supercritical conditions. The effects of liquefaction parameters such as temperature, catalyst and solvent on product yields were investigated. The liquid products were extracted with diethyl ether and benzene using an extraction procedure. The product yields in supercritical methanol, ethanol and acetone were found to as 41.6%, 53.8% and 64.3% in the non-catalytic runs at 310 °C, respectively. The highest conversion was obtained in supercritical acetone in the presence of ferric chloride (10%) at same temperature in the catalytic runs. The produced liquids in acetone were analyzed and characterized by elemental, Fourier transform infrared spectroscopy (FT-IR), gas chromatography–mass spectrometry (GC–MS). The liquid products (bio-oils) obtained with acetone contained various types of components including aromatics, nitrogenated and oxygenated compounds. As the bio-oils obtained exhibit high heat values, *E. spectabilis* is presented as a potential feedstock candidate for production of bio-fuels or valuable chemicals.
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Keywords: Biomass; Bio-fuel; Liquefaction; *Eremurus spectabilis*; Thermo-chemical process; Supercritical organic solvent

1. Introduction

In recent years, biomass has attracted much attention as a renewable energy resource since fossil fuels are accounted for 88% of the global primary energy consumption (Küçük and Demirbaş, 1997; Demirbaş and Demirbaş, 2010). Energy is consumed at a higher rate because of the technological progress, leading to energy insecurity and climate change. Greenhouse gas emissions have increased steadily due to a rise in the exploitation and already exceeded the high limit threshold of 450 ppm CO_2. It is widely accepted that the use of fossil fuels is unsustainable due to depleting resources (Sakthivel et al., 2011). Since large scale usage of fossil fuels for electricity, transport, and thermal energy generation has caused an increase in greenhouse gases and depletion of fossil fuel reserves, it is important to develop new techniques and adopt policies to promote renewable energy sources capable to sequester the atmospheric CO_2, and to minimize the dependency on fossil fuels maintaining the environmental and economic sustainability (Brennan and Owende, 2010; Singh et al., 2011; Hossain et al., 2008). At present, utilization, conversion and accessing of energy are the basis of many of the great challenges, including those related with environmental quality, security, poverty and sustainability. Biomass is an attractive alternative to current petroleum based fuels which can be utilized as transportation fuels with little change to current technologies and have significant potential to improve sustainability and reduce greenhouse gas emissions. Recently, biomass utilization researches have been intensified for both economical and ecological concerns, especially for its use as an alternative to petroleum based fuels. If environmental and economic sustainability are considered carefully,

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biomass could play an important role in lowering long term carbon dioxide emissions and in reaching targets to replace petroleum based transportation fuels with a viable alternative. In recent years, worldwide use of liquid bio-fuels in transportation has been growing rapidly. This has been mostly driven by policies focused on achievement of secure energy and mitigation of greenhouse gas emissions (IEA, 2007).

Bio-energy is considered as one of the most important components for mitigating greenhouse gas emissions and substitute of fossil fuels (Aresta et al., 2005). Energy need is continuously increasing because of global industrialization and population growth. Today, basic sources of energy are supplied from petroleum, natural gas, coal, hydro and nuclear. The major disadvantage of using petroleum based fuels is atmospheric pollution created by the use of petroleum diesel. Petroleum diesel combustion is a major source of greenhouse gas. Apart from these emissions, petroleum diesel is also a major source of other air contaminants including NOx, SOx, CO, particulate matter and volatile organic compounds. Biomass is one of the better sources of energy. Large-scale utilization of biomass energy can contribute to sustainable economic, environmental, and social development of countries worldwide. It has been reported that bio-fuels obtained from canola, soybean, palm, sunflower oil, algal oil can be used as a diesel fuel substitute (Alp and Cirak, 2012; Demirbaş, 2011; Aysu et al., 2012; Şener et al., 2010; Aysu and Küçük, 2013a). Biodiesel is a non-toxic and biodegradable alternative fuel that is obtained from renewable sources. Biodiesel fuel can be prepared from waste cooking oil such as palm, soybean, canola, rice bran, sunflower, coconut, corn oil, fish oil, chicken fat and algae (Küçük and Demirbaş, 1997). The burning of enormous amounts of fossil fuels has increased the CO_2 level in the atmosphere causing global warming. Biomass has been focused on as an alternative energy source, since it is a renewable resource and fixes CO_2 in the atmosphere through photosynthesis. If biomass is grown in a sustained way, its combustion has no impact on the CO_2 balance in the atmosphere, because the CO_2 emitted by the burning of biomass is offset by the CO_2 fixed by photosynthesis (Demirbaş, 2010).

Biomass, either terrestrial or aquatic is considered as a renewable energy source with quasi zero-emission. Among alternative energy sources, biomass represents the most ready to be implemented on a large scale without any environmental or economic risks. The idea of using biomass as a source of fuel is not new but it is now being taken seriously because of the escalating price of petroleum and more significantly the emerging concern about global warming associated with burning fossil fuels (Zou et al., 2010). Some biomass species for fuel production have received worldwide attention in recent years because of the high growth rate. Direct thermo-chemical conversion of lignocellulosic materials can produce gases and liquid fuels in a quick and efficient way. One of them is *Eremurus spectabilis* which is a hardy perennial plant growing to 1 m. *Eremurus* is a genus of nearly 50 species in the family of Liliaceae. Natural populations of this genus are widely distributed on dry and stony grazed hillsides native to the Central Asia and Middle East, including Afghanistan, Iran, Tajikistan, Lebanon and Turkey. Two of *Eremurus* species, namely *E. spectabilis* and *Eremurus cappadocicus* naturally grow in Turkey. *E. spectabilis* is widely distributed in the provinces of Turkey such as Erzurum, Sivas, Yozgat, Bitlis, Usak, Kars, Agri, Erzincan, Van, Artvin and Ardahan. *E. spectabilis* is called as "Çiriş, Dağ pırasası, Gülük" by local people in Turkey. The leaves of *E. spectabilis* are edible and used as a vegetable and its roots are used to make gum. The young leaves of the plant are consumed as a meal after cooking. *E. spectabilis* is used traditionally used in folk medicine to treat some ailments such as hemorrhoids and diabetics, and also used as antidysuria and antihypertensive (Li et al., 2000; Tosun et al., 2012; Baytop, 1999; Tuzlacı, 1985; Taskin et al., 2012).

Thanks to its location, Turkey has very rich biomass feedstock including bio wastes, lignocelluloses and aquatics. There are approximately 10,000 plants including *E. spectabilis* growing in the lands of Turkey. These plants could be evaluated as renewable energy sources to produce bio-oil or value-added chemicals. The main purpose of the present study was to investigate the possibility of a specific biomass, *E. spectabilis* for bio-oil production using supercritical organic solvents with and without catalyst in a cylindrical reactor (autoclave). Effects of process variables such as temperature, solvent and catalyst on product yields were also investigated.

2. Materials and methods

2.1. Materials

E. spectabilis samples, which are naturally grown in East Anatolia were collected from Van region (geographical coordinates: 38°29′39″ N, 43°22′48″ E) in Turkey in April 2013. They were dried in open air and ground with Perten Instruments LM120 mill to a size of |≤0.425 mm and extracted with petroleum ether (b.p. 40–60 °C) in a Soxhlet extractor for 6 h.

Before liquefaction experiments, the proximate and ultimate (elemental) analyses of the raw material (*E. spectabilis*) were performed. Elemental analyser (LECO CHNS-932) was used to perform the ultimate analysis of the sample. Tappi Test methods (Anonymous, 1998) were used for determining the main characteristics of the *E. spectabilis*. Lignin and cellulose were determined by Tappi T222 and Tappi T202 methods, respectively. Hollocellulose content was determined using the chloride method (Wise and John, 1952). Ash and moisture contents were determined according to Tappi T211 and Tappi T264, respectively. Higher heating value (HHV) was calculated by Dulong's Formula using the results of ultimate analysis. The proximate and ultimate (elemental) analyses of *E. spectabilis* were performed before liquefaction experiments and the results are given below. Ultimate analysis; carbon: 39.27%, hydrogen: 6.54%, nitrogen: 1.28%, oxygen: 52.91%, higher heating value (MJ/kg): 13.17. Proximate analysis; lignin: 30.1%, cellulose: 38.5%, hemicellulose: 20.5%, holocellulose: 59%, moisture: 5.6%, ash: 4.6% and soxhelet extractives: 0.7% as percent of dry feedstock. FT-IR spectrum of the raw material was taken using potassium bromide as transparent pellets with Perkin Elmer Spectrum One FT-IR Spectrometer to identify the structural groups in the raw material. Fig. 1 shows the FT-IR spectra of raw material. The raw material was characterized by FT-IR in the middle region, including the wave numbers between 4000 and 550 cm^{-1}. According to literature (Qian et al., 2007; Sun et al., 2011), the bands in the spectra of raw material (*E. spectabilis*) proves that it is mainly composed of lignin, cellulose and hemicelluloses. The band at 3328.70 cm^{-1} is formed by the hydroxyl group of lignin in *E. spectabilis*. The absorption at wave number of 1728.8 cm^{-1} is the characteristic of xylans of hemicellulose. The absorption peaks at about 2919.68 and 1316.93 cm^{-1} are the characteristics of cellulose. Generally the spectrum of lignin gives the similar absorption peaks. The absorptions at 2919.68, 1606.60 and 1007.57 cm^{-1}

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