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Continuous production of bio-oil by catalytic liquefaction from wet distiller's grain with solubles (WDGS) from bio-ethanol production

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ABSTRACT

Bio-refinery concepts are currently receiving much attention due to the drive toward flexible, highly efficient systems for utilization of biomass for food, feed, fuel and bio-chemicals. One way of achieving this is through appropriate process integration, in this particular case combining enzymatic bio-ethanol production with catalytic liquefaction of the wet distillers grains with soluble, a byproduct from the bio-ethanol process. The catalytic liquefaction process is carried out at sub-critical conditions (280–370 °C and 25 MPa) in the presence of a homogeneous alkaline and a heterogeneous Zirconia catalyst, a process known as the Catliq[®] process. In the current work, catalytic conversion of WDGS was performed in a continuous pilot plant with a maximum capacity of 30 dm³ h⁻¹ of wet biomass. In the process, WDGS was converted to bio-oil, gases and water-soluble organic compounds. The oil obtained was characterized using several analysis methods, among them elementary analysis and GC–MS. The study shows that WDGS can be converted to bio oil with high yields. The results also indicate that through the combination of bio-ethanol production and catalytic liquefaction, it is possible to significantly increase the liquid product yield and scope, opening up for a wider end use applicability.

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1. Introduction

The rapid increase in global energy demand makes the search for new energy sources a major concern, and as a consequence, there is a big interest in the production and use of renewable energy.

Biomass in various forms represents a significant source of renewable energy, and utilization of biomass, in contrast to utilization of fossil resources such as oil and gas, does not cause a net increase in atmospheric CO₂. The increasing level

of CO₂ in the atmosphere is by most scholars considered to cause global warming [1] and [2]. There is globally a large and increasing demand for liquid fuels produced from biomass, not only for environmental reasons, but also for pure economical ones due to increasing oil prices [3]. First generation ethanol production is the largest process for liquid biofuel production today. In 2008, global production of bio-ethanol reached 76.5 hm³, an increase of 32% compared with 2007 [4]. The largest producers are the US and Brazil. The main residuals from starch-based ethanol production are called WDGS

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(wet distiller's grain with solubles) with water content of $w_B = 70\%$, the production rate of WDGS per liter ethanol is in the range $2.4\text{--}4.2\text{ Mg m}^{-3}$ of ethanol, depending on feedstock for the ethanol process, making WDGS a large resource. The WDGS is in most cases further dewatered and dried to DDGS (Dried Distillers Grains with solubles), as indicated in Fig. 1. In a typical first generation ethanol process, the amounts of DDGS produced almost equals the amount of ethanol produced and the energy-intensive drying step adds significantly to both the capital and operating costs [5].

DDGS and WDGS are today mainly used as cattle feed [6]. However, instead the WDGS could be used directly to produce a liquid fuel in a thermal liquefaction process, such as the CatLiq[®] process. This is shown in Fig. 2. This would increase the liquid fuel output and offer a more flexible process, which allows for adjustment of the product profile depending of the price of DDGS and crude oil.

Several other processes for thermal transformation of biomass to liquids with fuel properties exists, however most of them are carried out on dry biomass such as pyrolysis. Pyrolysis is the process of thermochemical transformation of biomass under non-oxidative conditions [7–10]. One of the most used pyrolysis methods is called fast pyrolysis. Typical fast pyrolysis conditions are $500\text{--}520\text{ }^\circ\text{C}$ and residence time of $1\text{--}5\text{ s}$. Pyrolysis oil has a heating value about half that of conventional fuel oil, rather high oxygen content as well as limited chemical stability. Other thermal processes for liquid fuel production include depolymerization (CDP) and biomass gasification combined with Fischer-Tropsch synthesis (BG-FT) [11] and [12].

Many of the above mentioned processes are not optimal for wet biomass. However, many biomass based materials, such as WDGS, contain a large fraction of water. To remove the water by evaporation consumes a considerable amount of energy. In hydrothermal liquefaction processes aqueous biomass is directly converted to oil, water-soluble substances and gas at sub-critical conditions [13–15]. Oils from hydrothermal liquefaction in general have lower oxygen content than pyrolysis oils and also a better chemical stability. Most of these processes operate at pressures and temperatures in the range of $250\text{--}350\text{ }^\circ\text{C}$ and $10\text{--}25\text{ MPa}$ respectively. Alkali catalysts such as NaOH, Na_2CO_3 , KOH and K_2CO_3 are often added

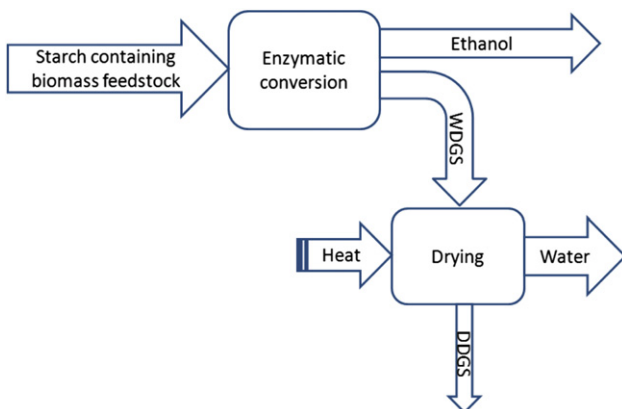


Fig. 1 – Starch based bio-ethanol production with ethanol and DDGS as products.

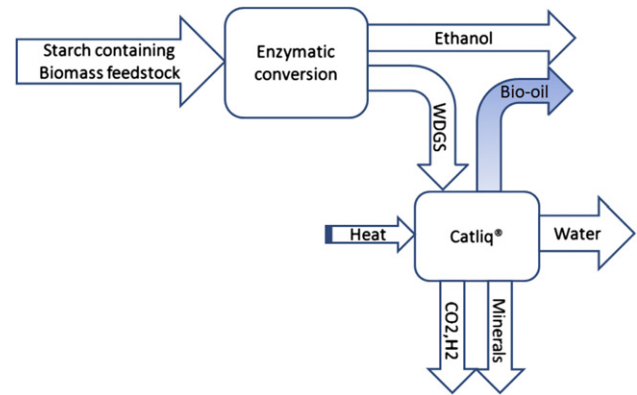


Fig. 2 – Multiple technology bio-refinery with ethanol and bio-oil as products.

to improve gasification and to suppress coke formation [16]. The CatLiq[®] process is similar to these processes, but the use of heterogeneous catalyst, as well as other several process features, is unique. In particular, the heat-up of the feed to process temperature is carried out within seconds. This rapid heat-up is important to optimize oil yields and prevent tar and coke formation [17].

The aim of the current work was to carry out an initial study of the conversion of DDGS to bio oil in the CatLiq pilot plant.

2. Experimental

2.1. The CatLiq[®] process

The Danish company SCF Technologies has developed the CatLiq technology and operates a continuous $30\text{ dm}^3\text{ h}^{-1}$ capacity pilot plant in Copenhagen, Denmark. The study described in this paper was carried out in the pilot plant.

In the CatLiq[®] process the organic fraction of the feed stream is converted to oil in the presence of a homogeneous (K_2CO_3) and a heterogeneous (Zirconia-based) catalyst, at sub-critical conditions ($280\text{--}370\text{ }^\circ\text{C}$ and 25 MPa). The full product consists of a top-phase of bio-oil, a gas-phase mainly consisting of CO_2 , a water phase with soluble organic compounds and a bottom-phase mainly consisting of inorganic salts.

The conditioned feed from the feed tanks is pressurized by a high pressure feed pump. The feed is then preheated in the feed heater. The feed enters a recirculation loop, in which a recirculation pump ensures a high flow rate. As the flow rate within the recirculation loop is higher than the new feed stream by approximately 9:1, the injection of new, colder feed only necessitates the addition of a small amount of heat in the trim heater, giving a very fast heating rate. The flow passes through a trim heater and a fixed-bed reactor filled with heterogeneous catalyst. After the reactor a fraction of the product stream is withdrawn and passed through a cooler. After pressure reduction the oil is separated from the water by centrifugation or gravimetric separation. A schematic flow sheet is shown in Fig. 3.

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