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ScienceDirect

Energy Procedia 105 (2017) 75 – 81

Energy Procedia

The 8th International Conference on Applied Energy – ICAE2016

Non-catalytic hydrothermal liquefaction of biomass: An experimental design approach

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Abstract

A face centered central composite design was used for determining the effects of reaction temperature (180–260 °C), reaction time (0–2 h) and sawdust concentration (9.1–25 wt%) on conversion and recovered product yields during hydrothermal liquefaction of pine sawdust. The determined conversion and aqueous product (AP) yield were in the range 23.1–57.2% and 14.6–43.4%, respectively. The results showed that all linear model terms and some interaction terms were statistically significant for sawdust conversion and liquid product yields, whereas all quadratic terms were found statistically insignificant. In general, increasing reaction temperature increased sawdust conversion and AP formation, while increasing sawdust concentration led to a reduction in conversion and liquid product yields. The determined model revealed that the decomposition reactions of lignocellulosic constituents may have occurred competitively, thus resulting in a certain trend in recovered AP and heavy oil (HO) yields.

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Peer-review under responsibility of the scientific committee of the 8th International Conference on Applied Energy.

Keywords: hydrothermal liquefaction, pine sawdust, central composite design

1. Introduction

The development of biomass-based technology to produce chemicals, fuels and energy has become a necessity to counter the dependency on fossil fuels, meet energy demand and reduce carbon dioxide emissions [1]. Biomass is defined as the renewable organic material originating from plants, including their derivatives [2]. It is abundant and is considered as neutral for respective carbon emissions [1,3]. In particular, lignocellulosic biomass has great potential to be utilized since it does not compete with food resources [4,5].

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Peer-review under responsibility of the scientific committee of the 8th International Conference on Applied Energy.
doi:10.1016/j.egypro.2017.03.282

Hydrothermal liquefaction (HTL) is a promising process to convert high moisture lignocellulosic biomass into liquid chemicals and fuels. The merits of liquid fuels are widely known due to their high energy content, transportability and applicability. Among other thermochemical conversion processes, HTL allows biomass to be processed without drying at a temperature of a few hundred degrees lower than the one of required for pyrolysis [3,6]. It is currently well-known that reaction temperature and reaction time affect biomass conversion and product yields during HTL, while adjusting the biomass concentration has correlated with energy savings for further upgrading steps (e.g. gasification of the HTL product) [7].

Experimental design offers an efficient and powerful method to evaluate multiple experimental variables, including their interactions [8–11]. In this study, a face centered central composite design at three different levels was utilized to determine the effects of reaction temperature, reaction time and sawdust concentration on the conversion and recovered product yields during HTL of pine sawdust.

2. Materials and Methods

2.1 Materials

Pine sawdust obtained from Northern Sweden (600–710 µm) was dried overnight at 105 °C prior to use. Added water was purified using Milli-Q water purification (0.22 µm Millipak Express 40 filter). The elemental composition (dry ash-free) of pine sawdust was 50.5% carbon, 6.76% hydrogen and 42.8% oxygen. Filter paper Whatman No.3 was dried and pre-weighed before use. Acetone (min 99.5%, Wako Chemical, Japan) was used as received. High purity Argon (>99.9%, Tomoe Shokai) was used during the gas chromatography (GC) analysis and purging the reactor.

2.2 Methods

The experimental run order was randomized to prevent systematic errors. In each experiment, 12 g of sawdust and a certain amount of water were loaded into a laboratory scale stirred batch reactor (400 ml effective volume, MMJ-500, OM Labo Tech, Japan) equipped with electric heating (average heating rate is 7 °C/min). The stirrer was set at 120 rpm to ensure adequate mixing. The reactor was sealed and the air inside was replaced by argon. The initial reaction pressure was atmospheric. During the heating, the system may enter the reactive temperature for biomass reaction. As soon as the reactor reached a predetermined target temperature, this temperature was maintained stable (±5 °C from the target temperature) for a certain reaction time. When the reaction time was complete, the reactor was cooled down (about 100 min) using electric fan until the temperature reached 35 °C. The heating and cooling period are excluded in the reaction time.

The generated gas product was collected using a sampling bag and its volume was measured. The recovered liquid and solid products were separated by a two step filtration procedure [12]. During the first filtration step, 100 g of water was added for rinsing. The water-soluble filtrate, denoted as aqueous product (AP), was collected. The heavy oil (HO) sticking to the solid residue (SR) was collected during the second filtration step by rinsing the SR with 200 g acetone. The acetone in the HO mixture was removed by using a rotary evaporator. The remaining water and solvent in the SR and HO fractions were furthermore removed by means of oven drying (105 °C) for at least 15 h and 12 h, respectively. An unavoidable loss of light components might have occurred during the drying.

2.3 Feed and product analysis

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