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BIOMASS & BIOENERGY

Lignin yield maximization of mixed biorefinery feedstocks by organosolv fractionation using Taguchi Robust Product Design

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ARTICLE INFO

Article history:
Received 12 August 2014
Received in revised form
24 November 2014
Accepted 19 December 2014
Available online 10 January 2015

Keywords:
Taguchi Robust Product Design
Organosolv fractionation
Lignin yield maximization
Lignocellulosic biomass
Biomass separation

ABSTRACT

Lignin, isolated from switchgrass (*Panicum virgatum*) and tulip poplar (*Liriodendron tulipifera*) using organosolv fractionation is currently being explored for its potential use in the production of value-added chemicals and bio-based polymers. Taguchi Robust Product Design (TRPD) was applied to maximize lignin yield from the fractionation process. The following four controllable design factors were used in the TRPD: process temperature (120 °C, 140 °C and 160 °C), fractionation time (56 and 90 min), sulfuric acid concentration (0.025 M, 0.05 M and 0.1 M), and feedstock type (switchgrass/tulip poplar chip ratios of 10/90, 50/50 and 90/10). Process noise was induced in the experiment by using either the mass- or volume-based feedstock charges of switchgrass and tulip poplar chips. A maximum mean lignin yield of 78.63 wt% and signal-to-noise ratio of 37.90 was found at a 90 min runtime, a process temperature of 160 °C, a sulfuric acid concentration of 0.1 M, and a feedstock composition of 10% switchgrass and 90% tulip poplar. Process temperature was the most significant factor that influenced lignin yield. This study may provide a pathway for industrialists and researchers interested in maximizing lignin yield in the organosolv fractionation process.

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1. Introduction

Lignin, an abundant natural polymer available from fractionation of renewable feedstocks is currently under investigation for the manufacture of carbon fibers, fillers, encapsulants, pharmaceutical intermediates, biopolymers and oxygenated aromatic compounds [1–5]. In this study, a novel organosolv fractionation process for the separation of woody and herbaceous feedstocks was used as a means to isolate lignin as a starting material for biobased chemical production [6]. More broadly, the effectiveness of organosolv fractionation positions it as a leading candidate for use within a biorefinery. By employing organosolv fractionation to provide separate streams of cellulose, hemicellulose and lignin, a biorefinery can integrate manufacture of low value biofuels with high value chemicals to reduce the consumption of nonrenewable resources and simultaneously enhance the portfolio of the biorefining process [2,7].

Operational costs of biomass fractionation are, however, an important factor and account for up to 20% of the total process cost for the conversion of lignocellulosic biomass into ethanol [8]. Moreover, supply logistics associated with feedstock availability, time of harvest, or the impact of storage all

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http://dx.doi.org/10.1016/j.biombioe.2014.12.021
0961-9534/Published by Elsevier Ltd.

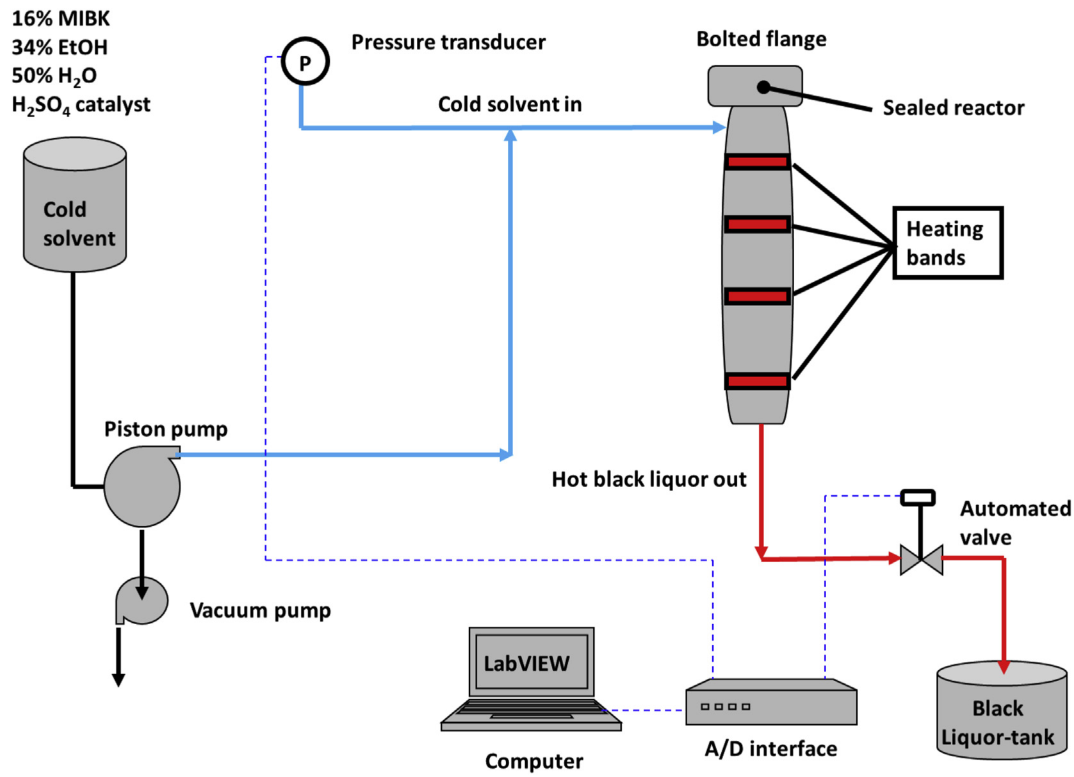

Fig. 1 – Reactor layout and flow diagram.

serve as potential limitations to the effectiveness of a biorefinery. A recent study [9] assessed the delivery cost of raw materials for biorefineries. Lower transportation costs were found when woody and herbaceous biomasses were combined, as compared to single feedstock delivery. This finding supports the importance of mixed feedstocks as a solution for biorefinery supply. Mixed feedstock streams within an economically feasible transportation distance from a biorefinery can be utilized and can tolerate variations in weather conditions (e.g., drought) that other annual agricultural crops such as corn cannot.

As a result of these barriers, the multiple operating parameters associated with lignin isolation from mixed feedstocks make process optimization via full factorial experimental design untenable because of the large number of experiments that would be required. Accordingly, this study uses Taguchi Robust Product Design (TRPD) to minimize the number of optimization experiments necessary to maximize the yield of organosolv-fractionated lignin from mixed feedstock streams of switchgrass and poplar. The experimental design is statistically optimized for processing conditions (temperature, acid concentration, feedstock ratios, and runtime) and evaluated for extracting the highest lignin yield from switchgrass and poplar feedstocks influenced by different bulk densities. Switchgrass and poplar are commonly used feedstocks for organosolv fractionation, which are targeted as bioenergy feedstocks by the U.S. Department of Agriculture (USDA) [10] and are part of a parallel project in energy crop development at the Center for Renewable Carbon, University of Tennessee. In this study, all input factors and levels were selected based on results from previous work [6,11]. This background information was used to assign factors and levels for the Taguchi experimental matrix.

The engineering methodology of TRPD is an important tool for process improvement [12,13] which offers the ability to optimize manufacturing processes through factor settings and make them resistant to sources of variation [14,15]. The Taguchi design uses a minimum number of experiments compared to classical design of experiment (DOE) methods such as response surface methodology, custom design or central composite design, which require a larger number of experimental runs for a given set of process variables and levels. Taguchi's "signal-to-noise" ratio (S/N) provides practitioners with a metric to assess the robustness of a product or process in the presence of controllable and uncontrollable factors. Reduction of process variation with TRPD aims to find an optimal combination of levels by maximizing the S/N ratio [16] and using a fractional factorial design obtained from orthogonal arrays [17]. For practical application in a manufacturing company, reduction of process and product variation through TRPD can be further enhanced by means of powerful statistical tools such as statistical process control (SPC) and design of experiments [18]. A robust manufactured product may improve product reliability and minimize warranty claims.

2. Methods

2.1. *Sample preparation*

Alamo switchgrass (*Panicum virgatum*) and tulip poplar (*Liriodendron tulipifera*) were used as feedstocks and air-dried at