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Short Communication

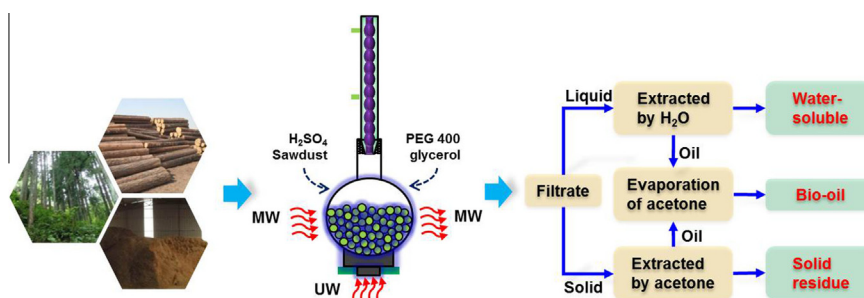
## Rapid and solvent-saving liquefaction of woody biomass using microwave–ultrasonic assisted technology

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## HIGHLIGHTS

- Biomass liquefaction with microwave–ultrasonic assisted was first investigated.
- The heat and mass transfer were intensified by microwave and ultrasonic wave.
- The liquefaction yield of fir sawdust reached 91% under the optimal conditions.
- The liquefaction time was shortened to 20 min and the solvent dosage was halved.
- The assistive technology shows great potential in the field of biomass conversion.

## GRAPHICAL ABSTRACT



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## ABSTRACT

A novel process to rapidly liquefy sawdust using reduced quantities of solvent, was successfully carried out via microwave–ultrasonic assisted technology (MUAT) in a sulphuric acid/polyethylene glycol 400–glycerol catalytic system. The influences of some key parameters on the liquefaction yield were investigated. The results showed that compared with traditional liquefaction, the introduction of MUAT allowed the solvent dosage to be halved and shortened the liquefaction time from 60 to 20 min. The liquefaction yield reached 91% under the optimal conditions. However, the influence on the yield of some parameters such as catalyst concentration, was similar to that of traditional liquefaction, indicating that the application of MUAT possibly only intensified heat and mass transfer rather than altering either the degradation mechanism or pathway. The introduction of MUAT as a process intensification technology has good industrial application potential for woody biomass liquefaction.

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## 1. Introduction

Catalytic liquefaction is a promising technology for converting biomass to produce sustainable liquid biofuels and chemicals (Yazdani et al., 2015). Traditional biomass liquefaction has usually

been carried out in a stirred tank reactor using inorganic acid/alkali as a catalyst at a temperature higher than 150 °C for 60 min or longer. A high mass ratio of solvent/biomass of larger than 6 (Li et al., 2015) has commonly been applied in traditional liquefaction. The large dosage of solvent supplies good transfer property of heat and mass for the desired liquefaction yield. However, the use of large amounts of solvents requires high energy consumption to recycle the excess solvent from the liquefaction products, bringing with it a considerable increase in the cost of the liquefaction.

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Therefore, it is urgent to develop a rapid and solvent-saving liquefaction method to serve as a substitute technology for traditional biomass liquefaction.

Traditional biomass liquefaction, as a liquid–solid heterogeneous catalytic reaction, is very complicated, because it not only involves heat and mass transfer between the inner and interphase of the solvent and the biomass particles, but also involves complicated degradation reactions in the hemicellulose, lignin and cellulose. These components may undergo their degradation only while the acid/alkali catalyst diffuses from the solvent phase into the interior of the biomass particles and as the temperature reaches the level required for degradation reactions. However, the transfer resistance of heat and mass during traditional liquefactions of lignocellulosic biomass is often very large due to such biomass' typically small pore diameter and low thermal conductivity. The heat and mass transfer are the controlling step during traditional biomass liquefaction, especially when only a little solvent is used during the liquefaction. Intensifying the transfer process can save solvent and allow for more rapid liquefaction.

In the past two decades, microwave technology has begun to be used to intensify heat transfer in the field of rapid biomass pyrolysis (Mushtaq et al., 2015; Wang et al., 2015) and biomass liquefaction (Zhuang et al., 2012; Zhang and Zhao, 2010). These efforts indicated that biomass could be liquefied efficiently and rapidly with the incorporation of microwave under suitable reaction conditions, which had good reference significances for the process intensification of biomass liquefaction. Ultrasonic waves as a process intensification technology have also been used in some liquid–solid mass transfer processes. Ultrasonic waves can clean the surface of solid particles in fluids (solvents) by producing impact forces, and can even cause solid particles to break (Hu et al., 2014) or collapse due to the instant high temperature and pressure resulting from the production of the cavity effect when a relatively high ultrasonic power and frequency are used. Therefore, the use of ultrasonic waves as a substitution for mechanical stirring or another intensification technology has been applied to some physicochemical processes for porous biomass, such as enzyme hydrolysis (Shi et al., 2013) and alkali treatment (Subhedar and Gogate, 2014). Until recently, ultrasonic wave technology begins to be introduced into biomass liquefaction.

Combining the above analyses and our previous work (Lu et al., 2013a, 2014), we speculated that if microwave and ultrasonic wave were introduced simultaneously into biomass liquefaction to intensify heat and mass transfer, the result would be a solvent-saving and rapid liquefaction of abundant and renewable woody biomass that would enhance its commercial potential. In recent years, microwave–ultrasonic assisted technology (MUAT) has been used in some treatment processes for biomass with excellent results in process intensification (Lu et al., 2013b). However, this new method for biomass liquefaction has not to date been reported in the published literature. In this paper, the liquefaction process of fir sawdust using MUAT was investigated using sulfuric acid as the catalyst in a solvent blend of polyethylene glycol 400 (PEG 400) and glycerol. The influences of the various parameters on the liquefaction yield were studied and the products were characterized by means of field emission scanning electron microscopy (FESEM), thermal gravimetric analysis (TGA), and elementary analysis.

## 2. Methods

### 2.1. Materials

Samples of fir sawdust were collected from a wood processing factory (Fuzhou, China), oven-dried at 388 K for 12 h, and then

screened into several fractions. The fraction with mean particle size of 1.265 mm (10–20 mesh) was selected and stored in a desiccator for experiments. The composition of the raw material was determined according to China's GB/T 2677 standard as follows: 40.1% cellulose, 26.7% hemicellulose, 31.3% acid-insoluble lignin, and 1.87% ash. All of the chemicals were of analytical grade and were used without any further purification.

### 2.2. Microwave–ultrasonic-assisted liquefaction procedure

10 g of dried sawdust were liquefied in a blend solvent of PEG 400 and glycerol using sulphuric acid as a catalyst. The quartz flask attached a UW transducer was loaded with the raw materials and placed in the liquefaction reactor. The liquefaction reactor (CW2000, Shanghai Xintuo Analytical Instruments Co. Ltd., China), was preprogrammed and equipped with a 2450 MHz MW and 40 kHz UW assisted device in the shell. MW passed through the quartz flask to irradiate the raw materials and UW was transferred from the transducer assembled at the bottom of the flask into the reaction system. The MW power can be set from 10 to 800 W, but the UW has only the option of “off” and “on” at 50 W fixed power. In this study, the liquefaction process was divided into two stages: (i) in the heated stage, the MW power and radiant time for all liquefaction experiments were set at 250 W and 2 min, respectively; (ii) in the liquefaction stage, the MW power was fixed at 60 W and the radiant time was set at the desired value. The ultrasonic generator was always used for the microwave–ultrasonic assisted liquefaction experiment but not for the MW-assisted liquefaction. Each experiment was duplicated three times and the values found were within 5% standard deviation of the mean.

Typically, sawdust (10–20 mesh) of 10 g, 98 wt% HSO<sub>4</sub> of 0.83 g (0.3 mol L<sup>-1</sup>) and a blend solvent (mass ratio of PEG 400/glycerol, 4) of 30 g were mixed together and then loaded into the reactor. Before the reactor was started, the assisted parameters of microwave–ultrasonic wave were preset as follows: for the heat stage, the MW power was 250 W and the radiant time was 2 min; for the liquefaction stage, the MW power was 60 W and the radiant time was 18 min. The UW was always working during the liquefaction process. After 20 min, the reaction was completed, and then the liquefaction products were collected. The non-condensable gases generated in the experiments were not taken into consideration in the mass balance due to their very small quantity.

The separation procedures of the products are described as follows. The collected liquefaction product was separated by filtration, evaporation, and extraction into three fractions: solid residue (SR), water-insoluble bio-oil (BO) and water-soluble products (WS). BO and SR were both dried in a vacuum drying oven at 363 K until they were at constant weight. The liquefaction yield of sawdust was calculated according to Eq. (1)

$$Y_L = \frac{M_S - M_{SR}}{M_S} \times 100\% \quad (1)$$

where,  $M_S$  is the mass of sawdust (10 g), and  $M_{SR}$  is the mass of SR.

### 2.3. Characterization

The sample micrographs were observed by means of a FEI Nova NanoSEM 230 field emission scanning electron microscopy at an accelerating voltage of 5 kV. The TGA profiles of SRs were measured and recorded by a NETZSCH STA 449 F3 Jupiter® simultaneous thermal analyser from 298 to 873 K at 10 K min<sup>-1</sup> using a 30 mL min<sup>-1</sup> N<sub>2</sub> as protective gas. The amounts of carbon, hydrogen, nitrogen, and oxygen in the samples were determined by an elemental analyser (Vario EL, Elementar, Germany). Their weight percentages were calculated according to the assumption that the samples only contained these elements.

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