



# Hydrothermal treatment of antibiotic mycelial dreg: More understanding from fuel characteristics

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## HIGHLIGHTS

- Hydrothermal treatment (HTT) decreases moisture adsorption ability of solid matters from mycelial dreg.
- HTT of mycelial dreg leads to biofuel with higher heating value and lower N and Cl contents.
- Reactions like dehydration, decarboxylation, hydrolysis and so on occur in HTT of mycelial dreg.
- HTT removes alkali metal oxides and sulfate but retains alkaline earth oxides and phosphate.
- HTT converts hard burning heavy volatiles into easily releasing and burning light volatiles.

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## ABSTRACT

Antibiotic mycelial dreg is a hazardous pollutant that is difficult to deal with because of its high contents of moisture and biomass pectin. Our previous work has demonstrated that the hydrothermal treatment (HTT) provides a feasible way to produce safe solid biofuel by reducing its moisture and nitrogen contents and decomposing its residual antibiotics. This study is devoted to further investigating the HTT through characterizing the HTT produced solid biofuels using oxygen bomb calorimeter, XPS, FTIR, XRF, TGA, DTA and so on. The results showed that the higher heating value (HHV) of the solid biofuel can reach about 26.5 kJ/g, which was much higher than the HHV of dried raw antibiotic mycelial dreg made by conventional drying (only 19.3 kJ/g). Moreover, the N content was lowered from 7.7 wt.% in the dried raw antibiotic mycelial dreg to 5.6 wt.% in the 200 °C HTT produced solid biofuel, while most alkaline metals and chlorine in ash of antibiotic mycelial dreg were leached out with centrifugation after HTT. The atomic ratios of H/C and O/C in the solid biofuels both decreased with increasing the temperature of HTT, indicating the occurrence of dehydration, decarboxylation and hydrolysis of solid matters in the dreg. The HTT also greatly improved the combustion reactivity of the solid matters in the antibiotic mycelial dreg. All of these show that HTT provides indeed an effective way to convert the antibiotic mycelial dreg into high-quality solid biofuel.

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## 1. Introduction

The exploration and usage of antibiotics were started centuries ago, and now its production has already become a big industry. For instance, the production of primary antibiotics was 0.3 million tons in China and 0.4 million tons worldwide in 2012 [1]. However, the

production of antibiotics generates more than ten times of mycelial dreg as residue which reached 4 million tons in China in 2012. Moreover, the residue amount keeps increasing at a rate of about 10% per year. Antibiotic mycelial dreg has not only high moisture content above 80 wt.% [2] but also high fraction of organic substances. The residual antibiotics in the dreg further make it hazardous and difficult to be tackled with. In fact, antibiotic mycelial dreg is on the list of the most dangerous industrial wastes in many countries [2]. The pharmaceutical companies are forced to treat, even recycle this waste in an environmental friendly way. The conventional treatment via landfill or open disposal has to

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cause serious secondary pollution by its moisture and residual antibiotics.

Considering the high protein content in the waste, many efforts have been made to recycle antibiotic mycelial dreg into animal feedstuff or organic fertilizer [1]. Apart from the high energy consumption by drying the dreg into solid feedstuff or fertilizer, the potential impact of its residual antibiotics on the security of the food chain is definitely an issue or a risk. Thus, this kind of direct uses of antibiotic mycelial dreg is being forbidden in many countries [2]. We then have to consider the incineration of it after high-efficiency moisture removal, which is expected to provide the feasible way for safe treatment as well as utilization of the waste [3].

For incineration of antibiotic mycelial dreg, the big obstacle is its high contents of moisture (80–90 wt.% in wet basis), oxygen (42.1 wt.% in dry basis) and nitrogen (7.7 wt.% in dry basis), which would cause low efficiency, low energy density and high  $\text{NO}_x$  emission, respectively. In the raw antibiotic mycelial dreg, the colloid, which is basically protein (30–40 wt.%) [2], leads to the tight bond of water with organic substances. Consequently, the water is hard to be deeply removed through various traditional mechanical dewatering technologies such as compressing, centrifugation and so on. Furthermore, the full vaporization of its water requires too much energy to make this technical way impractical. We have proposed hydrothermal treatment (HTT) as a feasible pretreatment technology to overcome the problems mentioned above [3].

Basically, hydrothermal treatment (HTT) of a biomass waste involves dehydration and decarboxylation of the waste to raise its carbon (C) content and heating value. HTT has been widely used to simulate the natural coalification processes for a century, and from last decade the application has been further extended to the pretreatment of biomass feedstock for making high-quality biofuel or biomaterial [4]. Many previous studies have demonstrated the effectiveness of HTT for producing clean solid biofuel from high-moisture wastes such as sewage sludge, municipal solid waste (MSW), low-rank coal and so on [5–12]. Namioka et al. [5,6] conducted studies to develop a two-step system consisting of HTT and mechanical dewatering to replace the traditional thermal drying for producing solid biofuel from sewage sludge. Further verification was made by Zhao et al. [7,8] and Meng et al. [9] on improvement of the dewaterability for several kinds of sludges by HTT. Upgraded fuel was made also by Mursito et al. [10] through HTT of tropical peat, while Areeprasert et al. [11] made an effort to produce an alternative solid biofuel from paper sludge via HTT.

As for antibiotic mycelial dreg, there was limited study on HTT of the waste and HTT produced solid product except for our recent work [3] on how HTT improves the dewaterability of the antibiotic mycelial dreg and how it decomposes its residual antibiotics. In succession to that previous work, this study is devoted to fully understanding the HTT produced solid biofuels and thereby to further investigating the mechanism of HTT of antibiotic mycelial dreg for solid biofuel. Detailed characterization of the fuel was performed through fuel analyses, for example, oxygen bomb calorimeter, X-ray photoelectron spectrometer (XPS), Fourier transform infrared spectrophotometer (FTIR), X-ray fluorescence (XRF), thermo gravimetric analyzer (TGA) and differential thermal analyzer (DTA). Understanding fuel properties and their correlations with the HTT conditions are expected to further clarify the mechanism of HTT for upgrading the antibiotic mycelial dreg. The examined solid biofuels were made by, in succession, HTT of raw antibiotic mycelial dreg in a bench-scale autoclave, centrifugation of the resulting HTT suspension in a centrifugal machine and finally drying of the solid matters in an air-drying oven.

## 2. Experimental section

### 2.1. HTT experiment and materials

The HTT test in this study was conducted in a 1.5-L bench-scale autoclave reactor shown in Fig. 1. The reactor was electronically heated, and a condenser was attached to the outlet of the reactor to cool down the exhaust gas. A rotator was set to enhance the mixing of sample and the heat transfer inside the reactor. Two impingers in series were used in the downstream of the condenser to absorb the exhausted gas.

HTT in real practice uses saturated steam to heat the mixture of waste and water [12], meaning that additional water is added. Thus, in this study the HTT test was performed by premixing the raw antibiotic mycelial dreg with distilled water in mass ratio of 5:2, which means 80 g water was added into 200 g the raw antibiotic mycelial dreg. Before experiment, argon was introduced to purge out air from the closed autoclave reactor. Then, the reactor was heated up to the targeted temperature (160–220 °C) and held there for 30 min. The rotator inside the reactor was kept stirring at a speed of 100 rpm during the heating and holding period. In turn, the heating was stopped and the autoclave reactor was carefully opened through a valve to reduce its pressure. The vaporized water in this process was discharged into the condenser to recover the condensable liquid. When the temperature in the autoclave was lower than 100 °C, the cover of the autoclave was opened to take out the hydrothermally treated suspension. The released ammonium with the exhausted gas from the reactor was determined by measuring the liquids recovered in the condenser and downstream impingers.

After the HTT was completed, the solid biofuels for analysis were made by centrifugation of the suspension from the autoclave reactor at 3000 rpm for 5 min and drying the centrifuged solid samples in an air-drying oven at 105 °C for 48 h. Besides, the raw dried antibiotic mycelial dreg was prepared via the same procedure but without HTT. All dry solid biofuels were ground into powders and sieved into sizes below 200 meshes for characterization.

The antibiotic mycelial dreg used in this work was from a cephalosporin production process in a pharmaceutical factory in Hebei Province, China. The basic properties of it were shown in Table 1, indicating that its original moisture content is high as 83 wt.% and the dried solid matter has no fixed carbon (FC) content so that the volatile matter content is over 90 wt.% (in dry basis). Of the element composition, its N is extremely high (7.7 wt.% in dry-ash-free basis), while C and O contents are equivalent in 41–44 wt.%. These properties indicate that the antibiotic mycelial

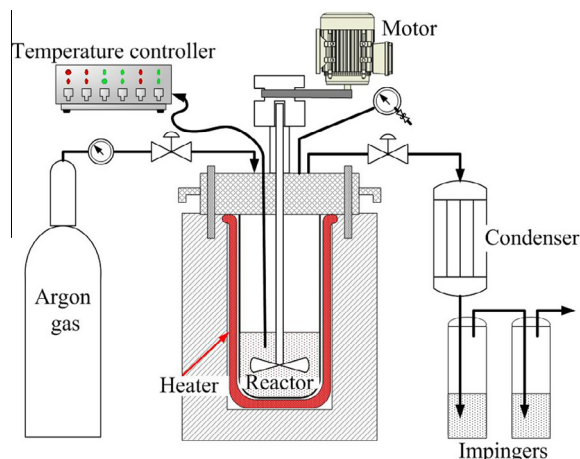


Fig. 1. A schematic diagram of the autoclave system for performing HTT test.

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