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Application of experimental design techniques in the optimization of the ultrasonic pretreatment time and enhancement of methane production in anaerobic co-digestion

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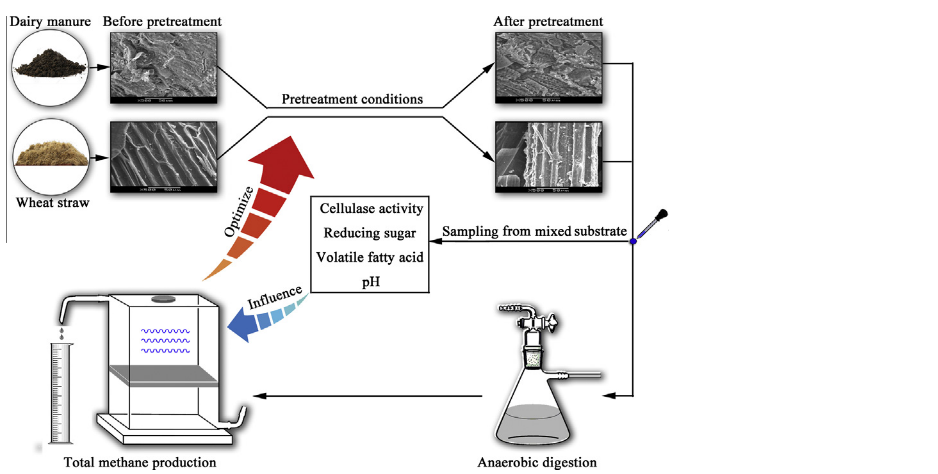
HIGHLIGHTS

- UP is a high efficiency method for application in AD.
- UP can improve TMP, net energy and energy benefit in AD.
- OED was more suited for design and optimization UP conditions than CCD.
- The maximum TMP and energy benefit were 186 mL/g TS and 2.88 kJ/g TS in OED.
- Cellulose activity contributed the maximum direct influence on TMP.

GRAPHICAL ABSTRACT



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ABSTRACT

In this study, wheat straw (WS) and dairy manure (DM) were ultrasonically pretreated to optimize the time for ultrasonic pretreatment (UP), identify the most appropriate optimization method, clarify whether UP is a high efficiency method for application, and explain why UP improves total methane production (TMP). The pretreatment conditions were designed using orthogonal experiment design (OED) and central composite design (CCD) and were optimized using the direct measurement method and response surface method, the relationships among the initial digestion characteristics and TMP were subsequently analyzed, the net energy, and energy benefit were calculated. The OEM results showed that mixed DM pretreated for 30 minutes (min) and WS pretreated for 20 min ($DM_{30}WS_{20}$) produced the maximum TMP, net energy, and energy benefit of 186 mL/g TS (total solid), 6.04 kJ/g TS, and 2.88 kJ/g TS, respectively. For CCD, the maximum TMP, net energy, and energy benefit were 146.61 mL/g TS, 4.91 kJ/g TS, and 1.75 kJ/g TS, respectively, and were obtained with DM_9WS_{25}. The maximum TMP, net energy, and energy benefit of OED were greater than those of CCD, indicating that OED is more suitable for UP in anaerobic digestion (AD). When pretreated with UP, the surface of DM showed an increasingly

Abbreviations: UP, ultrasonic pretreatment; AD, anaerobic digestion; WS, wheat straw; DM, dairy manure; TMP, total methane production; OED, orthogonal experiment design; CCD, central composite design; min, minute(s); VFA, volatile fatty acid; CK, untreated sample; TS, total solid; ANOVA, analysis of variance; R^2, coefficient of determination.

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uniform distribution and WS became increasingly rough and displayed fractures of different degrees. Cellulose activity contributed the maximum (0.3856) direct decision influence to TMP, and the comprehensive decision influence at a pH of 0.328 was the highest in TMP. This study concluded that the direct measurement method in OED was best suited for the design and optimization of pretreatment conditions by UP in AD. UP improved TMP, net energy, and energy benefit by changing the initial environment of AD. Therefore, UP is a high-energy benefit method and worth popularizing for AD.

1. Introduction

Anaerobic digestion (AD) is a biological process that converts complex substrates into methane via digestion by microbial action in the absence of oxygen [1]. Methane is a renewable energy source that can mitigate the fossil energy crisis [2,3], and can be used to produce solid and liquid residues for soil conditioning and fertilization [4]. Thus, AD is an efficient method that uses manure and straw to avoid environmental problems and generate clean energy. The wax layer on the epidermis of straw slows the absorption of water and limits the disintegration of lignocelluloses [5], which increases the retention time and reduces the degradation efficiency [7]. To address these issues, mechanical, ultrasonic pretreatment (UP), microwave, thermal, acid, and alkaline pretreatment methods are often used in AD to improve total methane production (TMP) [1,6,7]. Pretreatment increases the surface area and porosity of lignocelluloses, partially modifies and depolymerizes hemicelluloses and lignin, and reduces the crystallinity of cellulose, which changes the structure of the lignocellulose [8,9].

UP is a physical pretreatment method for methane projects that is a simple, time-saving and environmentally friendly, because it enlarges the reaction boundary of the lignocelluloses for degradation of highly polymeric matter and bond breaking at high temperature [10,11]. Previous studies have suggested that UP positively affects AD through the occurrence of acoustic cavitation phenomena [12]. The energy input affects the ultrasonic process performance [13], and energy amounts ranging from 1000 to 16,000 kJ/kg TS (total solid) have been reported [14]. For example, previous results have shown that when dairy manure (DM) was pretreated by ultrasound at 3600 kJ/kg TS combined with crude glycerine for AD, TMP was increased by 121% due to the increase in dissolved organic carbon [15]. UP at 3380 kJ/kg TS was used on activated sludge, and TMP was enhanced by more than 50% [16]. The pretreatment time and pretreatment power determine the energy input, and specific energy inputs in the range of 31–93 W h/L improved the disintegration degree by 22–53%, with an increase in specific biogas volumes of 46–71% [17]. Our published research showed that UP of maize straw for 30 min at 250 W improved total biogas production by 69.65% in anaerobic co-digestion of DM and maize straw [18]. Although UP can improve TMP, it requires energy input, and therefore, an examination of the net energy and the energy benefit of UP for AD are important. However, previous studies lack of calculating the energy benefit [17–19], which has resulted in arguments as to whether UP is a high-efficiency method for application. Certain research studies concluded that ultrasound did not offer an energy incentive for AD because the enhanced methane yields were not sufficient to compensate for the required energy in the absence of test data [20–23], without experiments or were only based on supposition. The UP is a good pretreatment method because this method is efficient and environmentally friendly, but applications of this method are difficult to develop in the current background. Thus, to investigate this process more fully, the energy input gradient of UP must be considered in future research to calculate the energy input and output difference. Moreover, the cellulose activity, reduced sugar content, volatile fatty acids (VFA) content and pH effects on TMP in the initial AD liquid are unclear.

Various experimental design methods can be used to design pretreatment conditions in AD, such as orthogonal experiment design (OED) [19] and central composite design (CCD) [20]. OED is an important statistical method used to study multi-factor and multi-level experiments and analyze the factor design [37]. CCD is a mathematical method used to analyze the relationship among variable factors that is well suited for multi-factor tests and thus applied widely [22]. Many previous studies have shown that OED combined with the direct measurement method could be used to design and optimize AD conditions [23–25]. For example, OED combined with direct measurement was used to design and optimize pretreatment conditions for the NaOH concentration, temperature and pretreatment time for wheat straw (WS) [26] as well as enzymatic pretreatment conditions, enzymatic temperature, enzymatic time, initial pH and substrate concentration in AD [27]. In recent, CCD combined with the response surface method has been applied to the design and evaluation of the interactive effects of pretreatment conditions for AD. For example, CCD and response surface methods have been applied to advanced pretreatment of olive oil, processing of wastewater using Fenton's peroxidation [28], selective optimization in thermophilic acidogenesis of cheese–whey wastewater to acetic and butyric acids [29], and evaluation the influence of the pH, temperature and substrate concentration on the acidogenesis of sucrose-rich wastewater [30], among others. However, no studies have conducted a comprehensive comparison of OED and CCD in AD with materials pretreated by UP.

To address these gaps, OED and CCD were used to design UP conditions for WS and DM, the direct measurement method and response surface method were used to optimize the pretreatment time, and OED and CCD were evaluated based on TMP, net energy, and energy benefit to identify the most appropriate optimization method. Path analysis was conducted to analyze the effect of the initial AD liquid on TMP, The net energy, and energy benefit were calculated to explore whether UP can improve the TMP and energy benefit. This research creates an experimental database of energy benefit for the practical application and further promotion of UP and also offers theoretical guidance for the choice of UP conditions for AD in real applications as well as a theoretical direction for the experimental design of scientific research.

2. Materials and methods

2.1. Origin and characterization of substrates

In this study, WS was obtained from a farm and cut into lengths of 1–2 cm by a 9Z-2.5 straw material shredder. DM was collected from a livestock farm located in Yangling, China. The inoculums of methane slurry with methanogens and nutrients were obtained from household methane digesters. Chemical characterization data for each substrate and the inoculums are presented in Table 1. All samples were collected in triplicate, and averages of the three measurements are presented.