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上海交通大学 Shanghai Jiao Tong University

Optimization of Sodium Hydroxide Pretreatment Conditions to Improve Biogas Production from Asparagus Stover

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There are 4 main parts

1. Introduction to Shanghai JiaoTong University(SJTU)
2. Biomass Energy Research in SJTU
3. Biogas
4. Selected Publications(SCI)

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上海交通大学
Shanghai Jiao Tong University

1.Shanghai JiaoTong University(SJTU)

- Established in 1896
- **There are 20 schools, including school of Agriculture and Biology, School of Mechanical and Power Engineering, School of Environment Engineering, etc.**
- **Students:44020; Teachers:2851**
- **Area of campus: about 333 ha.**

2.Biomass Energy Research in School of Agriculture and Biology (SJTU)

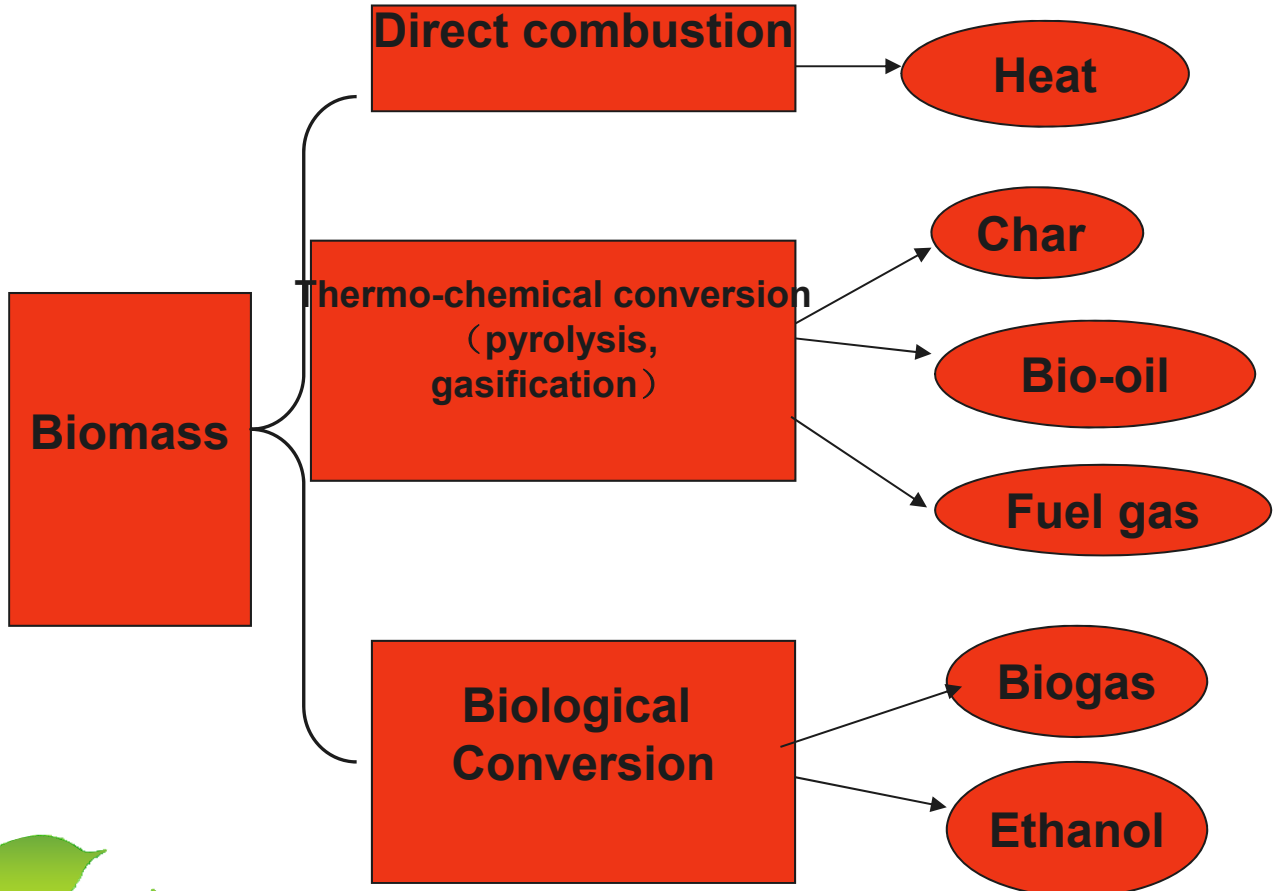
上海交通大学 Shanghai Jiao Tong University

Biomass Energy Engineering Research Centre,

School of Agriculture and Biology , Shanghai JiaoTong University has a lot of experiences in the field of biomass energy and environment.

Including characterization of biomass, biogas, biomass pyrolysis, biochar, gasification, bioethanol, etc.

Biomass energy conversion technologies



2.1 Biomass Fast Pyrolysis for Bio-oil production:
Project of Ministry of Science and Technology of China

Project Title: Development of Equipment for Biomass Fast Pyrolysis for Bio-oil Production and its Demonstration in Thousand Ton Scale

Organizer: Shanghai JiaoTong University

Partners:1) Zhejiang University

2)Shandong University of Technology

3)Guangzhou Institute of Energy Conversion, Chinese Academy of Science

4)University of Science and Technology of China

5) University of Science and Technology of South China

6)Liaoyang Hengxing Company Ltd

Coordinator of the project: Ronghou Liu

Period: January 2011-December 2013

Budget from MOST: 11.76 Million RMB Yuan

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A demonstration plant of biomass fast pyrolysis with bio-oil yield:10000 t/a in Shaanxi has been jointly built by Shanxi Yingjiliang Company and Shanghai Jiao Tong University, China

Fuel 182 (2016) 677–686

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journal homepage: www.elsevier.com/locate/fuel

Full Length Article

Performance of a commercial-scale biomass fast pyrolysis plant for bio-oil production

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ABSTRACT

A commercial-scale biomass fast pyrolysis plant, based on downdraft circulating fluidized bed technology with biomass throughput of 1–3 T h^{-1}, has been developed for bio-oil production and its performance has been investigated. The technological process consists of six parts: a feeding system, a heat carrier system, a reactor system, a cyclone system, a condensation system and a carbon separating system. The plant has four circulation systems: circulation of a heat carrier, quenching materials (bio-oil), cooling water and non-condensable gas. The bio-oil, raw material (rice husks), char and non-condensable gas samples were analyzed using GC–MS, FTIR, and SEM to characterize the physical properties and chemical composition. Results showed that the operation of the plant was stable. At 550 °C, the highest yield of bio-oil obtained was 48.1 wt% with char, and non-condensable gas yields of 26.0 wt% and 25.9 wt%, respectively. GC–MS results revealed that the composition of the bio-oil was complicated and the most abundant compound category was phenolics (14.92%). The char had complex pore structure by SEM analysis, which can be collected as a resources for further comprehensive utilization.

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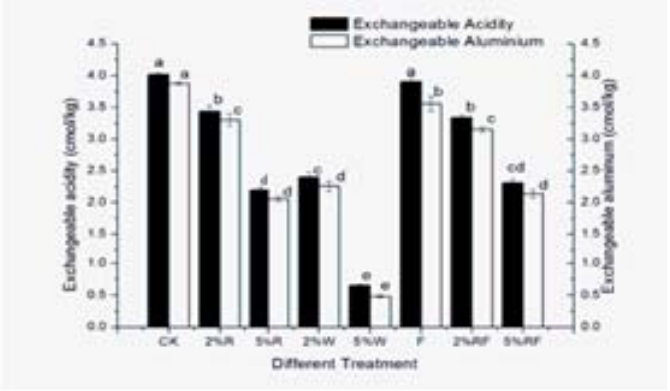
2.2 Biochar Application for Soil Amendment

-863 Project by MOST

1)Developed a biochar application machine:

Scale： 4753.8-34185.2 kg/h

2) The effect of biochar on soil and plant growth



wood sawdust biochar could reduce the exchangeable acidity and aluminum by 84% and 88%, respectively at the 5% biochar amendment level.

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2.3 Bioethanol

Available online at www.sciencedirect.com
ScienceDirect
Bioresource Technology 99 (2008) 847–854
BIORESOURCE TECHNOLOGY

Impacts of main factors on bioethanol fermentation from stalk juice of sweet sorghum by immobilized *Saccharomyces cerevisiae* (CICC 1308)

Ronghou Liu *, Fei Shen

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Received 17 October 2006; received in revised form 17 January 2007; accepted 17 January 2007
Available online 13 March 2007

Abstract

In order to attain a higher ethanol yield and faster ethanol fermentation rate, orthogonal experiments of ethanol fermentation with immobilized yeast from stalk juice of sweet sorghum were carried out in the shaking flasks to investigate the effect of main factors, namely, fermentation temperature, agitation rate, particles stuffing rate and pH on ethanol yield and CO_2 weight loss rate. The range analysis and analysis of variance (ANOVA) were applied for the results of orthogonal experiments. Results showed that the optimal condition for bioethanol fermentation should be $A_4B_3C_3D_4$, namely, fermentation temperature, agitation rate, particles stuffing rate and pH were 37 °C, 200 rpm, 25% and 5.0, respectively. The verification experiments were carried out in shaking flasks and 5 L bioreactor at the corresponding parameters. The results of verification experiments in the shaking flasks showed that ethanol yield and CO_2 weight loss

This paper has been Top 20 Articles, in the Domain of Article 17360181, Since its Publication (2008)

1. Introduction

Ethanol production as an alternative fuel energy resource has been a subject of great interest since the oil crisis of the 1970s (Tao et al., 2005). Therefore, a strong need exists for efficient ethanol production with low cost in raw material and production process. The varied raw materials used in the production of ethanol via fermentation are conveniently classified into three main types of raw materials: sugars, starches, and cellulose materials. Sugars (from sugarcane, sugar beets, sweet sorghum, molasses, and fruits) can be converted into ethanol directly. Starches (from corn, cassava, potatoes, and root crops) must firstly be hydrolyzed to fermentable sugars by the action of enzymes from malt or molds. Cellulose (from wood, agricultural residues, waste sulfite liquor from pulp, and paper mills) must likewise be converted into sugars, generally by the action of mineral acids. Once simple sugars are formed, enzymes from microorganisms can readily ferment them to ethanol (Lin and Tanaka, 2006). As for materials, one of the prime sources being investigated for ethanol is sweet sorghum. Sweet sorghum {*Sorghum biocolor* (L.) Moench} is a high biomass- and sugar-yielding crops (Bryan, 1990), meantime, the stalk of sweet sorghum contains quite a few quantities of soluble (glucose and sucrose) and insoluble carbohydrates (cellulose and hemicellulose) (Jasberg et al., 1983). Therefore, of many crops currently being investigated for energy and industry, sweet sorghum is one of the most promising, particularly for ethanol production (Gnansounou et al., 2005).

The advantages of immobilized cells over free cell sys-

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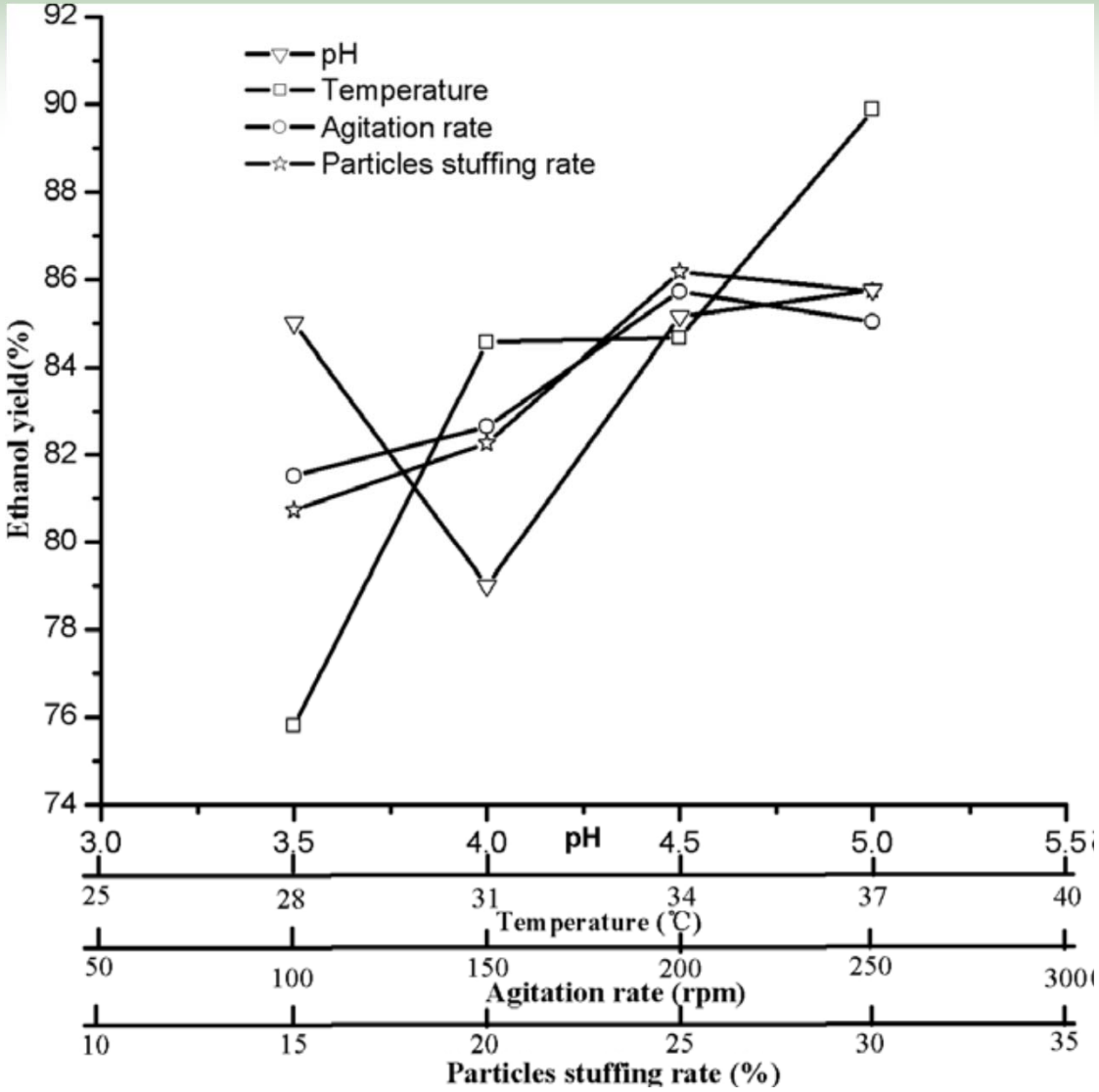
Optimal condition for bioethanol fermentation:

Fermentation temperature:37 ° C , agitation rate 200 rpm, particles stuffing rate:25%,
pH 5.0.
Ethanol yiedl:98.07%,
Fermentation time 11

h.



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3.Biogas

1. Introduction
2. Materials and Methods
3. Results and Discussion
4. Conclusions
5. Related work

Introduction

- The annually asparagus stover yield in Chongming county, Shanghai,China

  1000 ton

- How to deal with?

  Sodium hydroxide pretreatment + anaerobic digestion (AD)

- How to evaluate the effectiveness of
- pretreatment?

  Degree of lignocellulose degradation

  Biogas yield in subsequent AD

  Response surface method (RSM)

- The previous study of ‘change-one-factor-at-a-time’ test showed that asparagus stover can be used for biogas production after alkaline pretreatment.
- Appling the response surface method (RSM) to optimize the pre-treatment conditions.

- The objectives of this work is to investigate :
  - The pretreatment effectiveness on lignocellulose removal
  - The interactions among the pretreatment factors
  - The optimized conditions to improve biogas yield

Materials and Methods

Feedstock:

- naturally air-dried asparagus stover;
- grinded to partical size of ≤2.5cm;
- oven dried at 105° C for 6h before pretreatment.

Inoculum:

- Sludge from a pilot scale CSTR reactor treating pig manure (mesophilic)

Materials

The physical and chemical characteristics of asparagus stalk and inoculation sludge

	TS (%)	VS (%TS)	TOC (%)	TN (%)	Hemicellulose (%)	Cellulose (%)	Lignin (%)	pH
Feedstock	88.12	80.10	75.1	2.88	18.22	33.52	11.10	-
Inoculum	5.78	63.62	3.09	0.24	-	-	-	8.13

Air-dried asparagus stover

Grinded asparagus stover

Inoculum

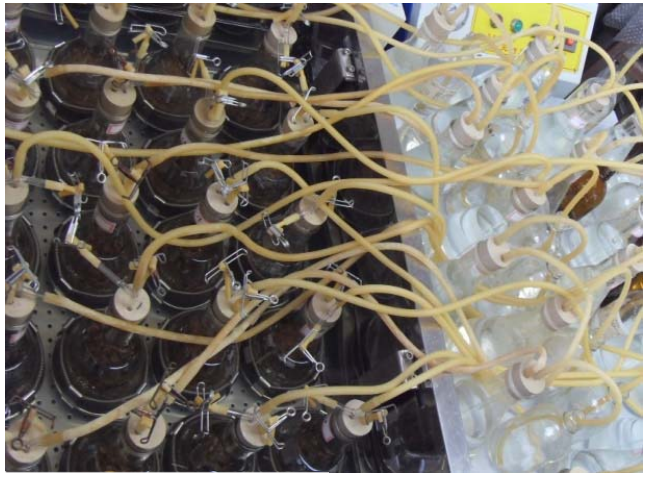
Materials and Methods

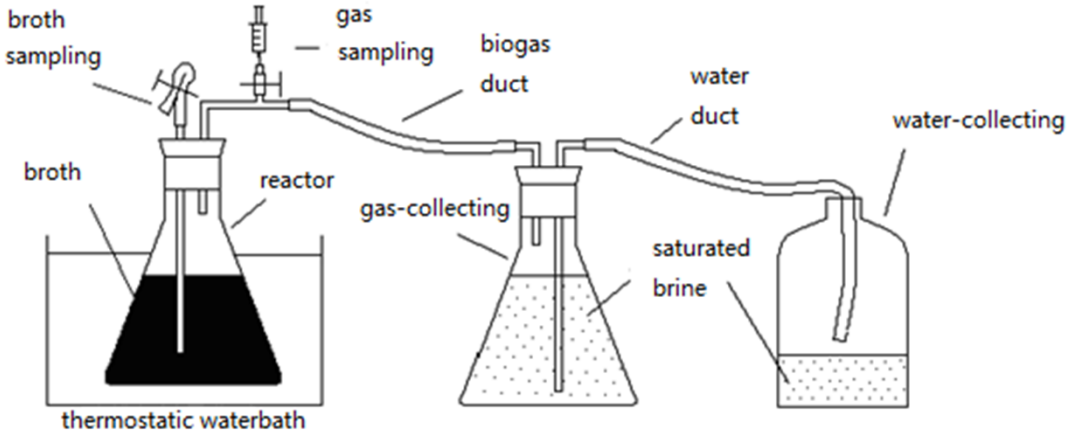
- TS of feedstock： 100 g
- 20 runs, duplicated, 25±1° C

2.5 L plastic buckets, sealed by vaseline and preservative film to avoid moisture change and rot fungi infection

Materials and Methods

- **Biogas production conditions：**
  - **flask reactor: 1 L**
  - **working volume: 0.8 L**
  - **temperature: 35° C**
  - **TS: 6%**
  - **Inoculum percentage: 30%**





Sketch of the biogas digester

Analysis Methods

- Daily biogas production: water displacement method.
- Methane content: gas chromatograph (GC-14B, shimadzu, Japan).
- Hemi-cellulose, cellulose and lignin: Van Soest method.
- TS, VS: Standard Methods (APHA, 1995).
- Total organic carbon: organic carbon analyzer (multi C/N 3000, Jena, Germany).
- Total nitrogen: Kjeldahl method.
- pH value: pH meter (PHS-3C, Leici, Shanghai).

Test Design

Independent variables：

- Pretreating time(d)，
- NaOH concentration (%
- NaOH solution dose(m

Response value:
Biogas yield

Experimental range and central point values of the independent variables

- Face-centered Central Composite Design;
- 3 factors, 3 levels;
- 20 runs;
- 6 replicates of the centre point.

Independent variables	Symbol		Coded level		
	Uncoded	Coded	-1	0	+1
Time /d	A	X_1	10	18	25
concentration/%	B	X_2	2.5	5	7.5
dose/ml	C	X_3	20	60	100

The second-order polynomial formulation：

$$Y=A_0+A_1X_1+A_2X_2+A_3X_3+A_{12}X_1X_2+A_{13}X_1X_3+A_{23}X_2X_3+A_{11}X_1^2+A_{22}X_2^2+A_{33}X_3^2$$

Results and Discussion

The experimental design and results for biogas yield

	X_1	X_2	X_3	Biogas yield	
Run	Pretreatment time	NaOH concentration	NaOH Solution dose	Test result	Predicted
	d	%	mL	mL/g VS	mL/g VS
1	-1	1	-1	102.7	91.5
11	0	0	0	292.5	266.8
12	0	1	0	178.8	190.8
13	1	-1	-1	124.9	114.8
14	1	0	0	235	235.4
15	1	-1	1	207.3	211.0
16	0	0	1	260.6	245.3
17	0	0	0	301.3	266.8
18	1	1	1	97.8	113.5
19	0	0	0	262.7	266.8
20	-1	-1	-1	105	91.8

regression analysis

The regression model:

$$Y=-381.79+29.39x_1+89.68x_2+5.35x_3-0.13x_1x_2$$

$$-7.92\times10^{-3}x_1x_3-0.24x_2x_3-0.763x_1^2-8.25x_2^2-0.03x_3^2$$

Results and Discussion

ANOVA for the response surface quadratic model for biogas yield

Source	Sum of squares	df	mean square	F-value	P-value	significance
Corrected model	89595.2	9	12785.72	14.05	0.0001	**
x_1	1317.90	1	1317.90	1.86	0.2025	
x_2	5978.02	1	5978.02	8.44	0.0157	*
x_3	5664.40	1	5664.40	7.99	0.0179	*
x_1x_2	50	1	50	0.07	0.7959	
x_1x_3	45.125	1	45.125	0.06	0.7959	
x_2x_3	4723.92	1	4723.92	6.67	0.0273	*
x_1^2	5070.78	1	5070.79	7.16	0.0233	*
x_2^2	7319.46	1	7319.46	10.32	0.0093	**
x_3^2	5653.44	1	5653.45	7.98	0.0180	*
Residual	7180.93	10	708.58			
Lack of fit	4775.67	5	936.11	1.95	0.2413	
Pure error	2405.26	5	481.05			
Total	96681.00	19				
SD	26.6192	R^2	0.92671	pred- R^2	0.6519	
CV/%	13.5184	adj-R^2	0.8607	Adeq precision	9.5565	

the fitting model is highly significant

The regression model is fairly fit

more than 92.67% variability of the response can be explained by the model

Results and Discussion

- 3.1 Statistical Analysis

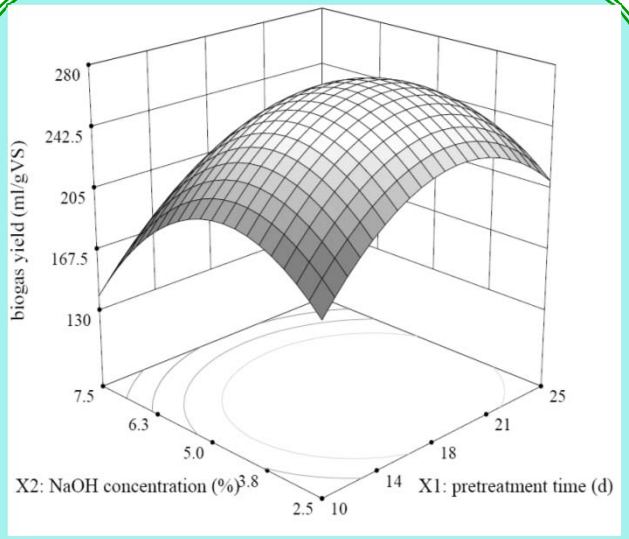
- According to ANOVA, The model could be reduced to:

$$Y=-361.81+28.25x_1+87.35x_2+5.21x_3-0.24x_2x_3-0.76x_1^2-8.25x_2^2-0.03x_3^2$$

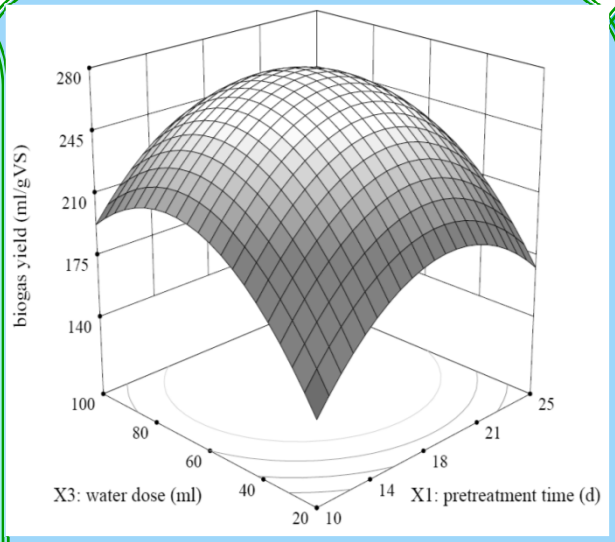
The credibility test showed that:

- Adeq Precision=9.5565, greater than 4 is desirable
- $R^2=0.927$, more than 92.67% variability of the response can be explained by the model
- $R^2_{Adj}=0.861$ is in agreement with $R^2_{Pred}=0.652$
- C.V=13.52% ,>10% implies biogas production from lignocellulosic feedstock is lack of stability

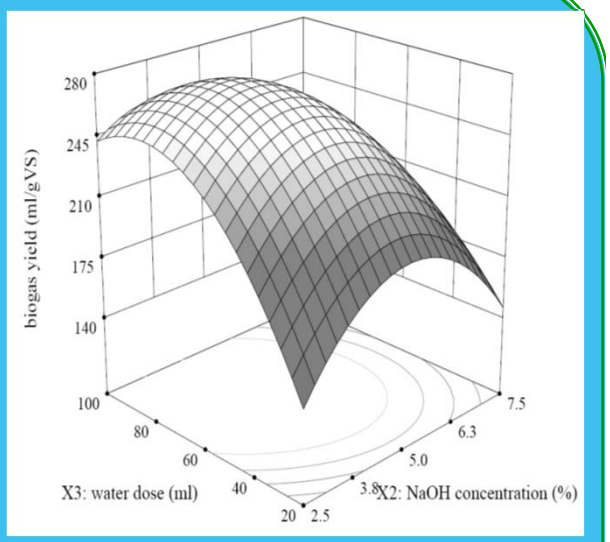
Interactive effect of NaOH concentration and NaOH solution dose on the biogas yield



the interactive effects of x_1 and x_2 on biogas yield



the interactive effects of x_1 and x_3 on biogas yield



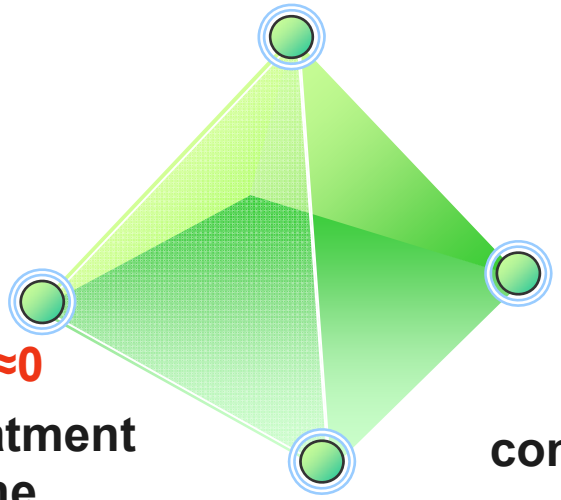
the interactive effects of x_2 and x_3 on biogas yield

At low NaOH concentration, high solution dose resulted in more biogas yield;

High NaOH concentration and low solution dose would not benefit biogas yield.

Optimization Analysis

Optimization Choices



≈0
tment
ne
d

X_2=0.35
NaOH
concentration
4.2%

X_3=-0.32
solution dose
74ml

Maximum predicted biogas yield(Y)

Y_{max}=275.65 mL/g VS

Verification Test

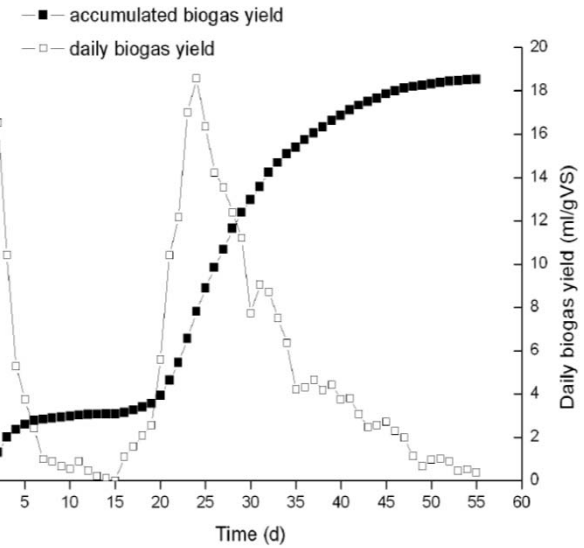
ne pretreatment conditions of **19 d, 4.2% NaOH concentration** **l 74 g NaOH solution dose** was used for verification test.

iplicate batch AD test was carried out for **55d**.

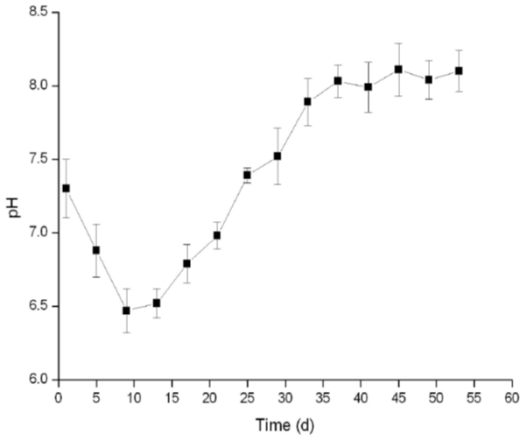
The lignocelluloses content before/after pretreatment

	Hemi-cellulose (wt.% dry basis)	Cellulose (wt.% dry basis)	Lignin (wt.% dry basis)
ore pretreatment	18.22	33.52	11.1
ter pretreatment	6.34	23.78	9.60

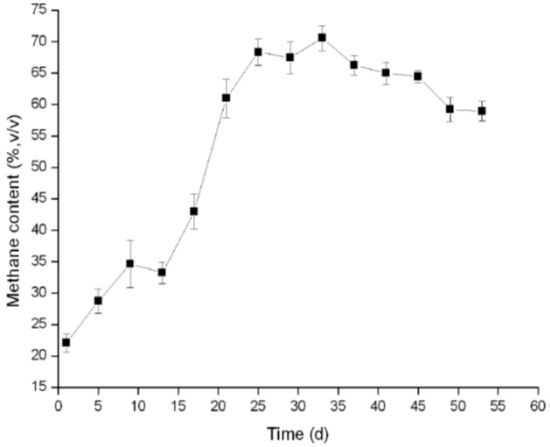
Verification Test



he daily and accumulated
ogas yields from asparagus
tover pretreated by NaOH



pH value



methane content

e observed biogas yield of 277.86
g VS was close to the predicted
ue of 275.65 mL/g VS with the

- A acidification stage was showed by pH dropping to 6.5

Conclusions

1. The optimized NaOH pretreatment conditions were pretreatment time of 19d, NaOH concentration of 4.2%, NaOH solution dose of 74g.

2. At the optimized conditions, the maximum biogas yield of 277.86 mL/g VS was acquired in verification test with the relative error of 0.80% compared with the predicted value of 275.65 mL/g VS.

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ts of Alkaline Hydrogen Peroxide Pretreatment on Chemical
osition and Biochemical Methane Potential of Agricultural
Stalks

,†,‡,§ Ronghou Liu,*,†,§ Weixing Cao,§ Renzhan Yin,∥ Yuanfei Mei,†,‡,§ and Le Zhang†,§

energy&fuels

Impacts of Alkaline Hydrogen Peroxide Pretreatment on Chemical Composition and Biochemical Methane Potential of Agricultural Crop Stalks

Chen Sun,†,‡,§ Ronghou Liu,*,†,§ Weixing Cao,§ Renzhan Yin,§ Yuanfei Mei,†,‡,§ and Le Zhang†,§

ctive:

increase bio-digestibility and methane yield from crop residues via
eatment

ts and conclusions:

e AHP pretreatment could:

- break down esterified and etherified linkage in lignocellulose
- recover 90% of glucose and 80% of xylose;
- remove 30-50% of lignin;
- increased methane yield and bio-digestibility for certain

Selected Publications(SCI)

anju Chen, Ceng Wu, Ronghou Liu, et al. Effect of hot vapor filtration on e characterization of bio-oil from rice husks with fast pyrolysis in a uidized bed reactor[J]. Bioresource Technology, 2011, 102, 6178-6185.

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Thank You!