

Research on the Conversion of Waste Straw into High-octane Gasoline

废弃秸秆转化高辛烷值汽油实验研究

Research Paper

研究论文

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1. RATIONALE

With the economic and technological development, demand for high-octane gasoline is constantly increasing. The majority of the current energy supply comes from fossil fuels, which are non-renewable and can contribute to pollution. In addition, waste straw is piled up beside roads or burnt, further contributing to air pollution. This straw represents a renewable biomass resource which is a potential source for the production of alternative fuels. Therefore, the conversion of waste straw into high-octane gasoline is a potential solution to the depletion of fossil fuels and pollution associated with their extraction and combustion.

China is a major producer of agricultural products with 0.73 trillion tons of waste straw produced in 2015, containing over 12 EJ of potential energy. Over the past two years, the government has pledged to solve the problem of waste straw and banned burning of the straw. Recycling of waste straw is difficult and remains a major concern. In other countries, solutions to manage and convert waste straw into alcohol or fertilizer have been developed. Conversion of waste straw (and other biomass resources) into high-octane gasoline using industrial processes has been proposed as another way of dealing with waste straw. Compared to other solutions, conversion of waste straw into gasoline is a fairly efficient way to solve the problems from two completely different fields at the same time.

2. PROPOSAL

2.1 Research Question

Current production methods suffer from drawbacks, including poor product quality and the inability to use the gasoline separately or directly. Thus, possible solutions to optimize or develop a better approach for conversion of biowaste to gasoline are urgently needed.

2.2 Hypothesis

The conversion of waste straw to gasoline can be optimized, or a better approach can be developed.



2.3 Engineering Goal and Expected Outcomes

Objective: The objective is the complete low-cost conversion of waste straw to gasoline that can be used directly in transportation. I plan to fulfil this objective through three main methods.

- (1) Catalysts: Ru/C, sulfuric acid, and other simple catalysts instead of expensive catalysts or those requiring strict conditions, such as Pt/Au, to reduce costs while maintaining reaction efficiency.
- (2) Parameter optimization: Effectively reduce the energy consumption of the conversion to fit industrial practices and adhere to the fundamental principles of green chemistry.
- (3) Reduce pollution caused by heavy metals, both in the catalytic process and in the product.

3. EXPERIMENTAL AND ANALYTICAL METHODS

3.1 Method Choice

Cellulose can be extracted from straw powder, and the conversion of straw biomass to high-octane gasoline can be achieved from cellulose as a raw material. According to previous studies, using chemical compounds as bridges to convert cellulose to gasoline with effective catalysts is promising.



Fig. 3.1 Scientific Principle and Technique Flow Chart for the Conversion Process



3.2 Experimental Procedure

The procedure can be divided into four primary components: (1)cellulose to levulinic acid (LA), (2)LA to γ -valerolactone (GVL), (3)GVL to butene, and (4) butene/isobutane to gasoline (Fig. 3.1).

3.2.1 Cellulose to LA



LA is an important polysaccharide hydrolysis product. Using various acids, including HCl, HBr, and H_2SO_4 as catalysts, is a common and useful way to convert cellulose to LA. Cellulose is first hydrolyzed to glucose with subsequent isomerization of glucose to 5-

hydroxymethylfurfural (HMF). Because HMF is unstable at high temperatures, rearrangement occurs to produce formic acid (FA) and LA (Fig. 3.2).

$$C_6H_{10}O_5 + H_2O == C_6H_{12}O_6 == C_5H_8O_3 + CH_2O_2$$



Fig. 3.2 Reaction Mechanism of the Conversion from Cellulose to LA

This experiment will employ a heating mixing control instrument and 50-mL autoclave with a polytetrafluoroethylene (PTFE) lining tube as the reaction vessel to simulate an industrial experiment. Autoclaves will be placed in a 600-r/min magnetic stirring heating furnace. This experiment will investigate an experimental group using straw powder as the reactant and control group using pure glucose/cellulose. Using sulfuric acid (2-3%) as a catalyst and adjusting the catalyst-to-reactant ratio from 1:200

to 1:800 at 190 to 210 °C and 0.5 to 2 MPa pressure N₂, the process is expected to last for 20–60 min. Rapid cooling will be required after reaction, and the products will be transferred to a test tube and diluted to 2–5 g, and solid–liquid separation will be performed by centrifugation. Further purification will then be performed using liquid chromatography.

3.2.2 LA to γ-Valerolactone (GVL)



 γ -Valerolactone (GVL) is an important chemical that is known to form a green reaction solution. It is nontoxic and biodegradable, making GVL a focus of biochemical conversion studies. Based on thorough comparison, we chose to use a Ru/C catalyst instead

of Au to catalyze the hydrogenation reaction of LA to GVL (Fig. 3.3).



Fig 3.3 Reaction Mechanism of the Conversion from LA to GVL

To better test and determine the yield and selectivity of the conversion, this experiment will not use the product of the last step. This experiment will use a heating mixing control instrument and a 50-mL autoclave with a PTFE lining tube as the reaction vessel to simulate an industrial experiment. Autoclaves will be placed in a 600-r/min magnetic stirring heating furnace. Using Ru/C as a catalyst and adjusting the catalyst-to-LA ratio from 1:200 to 1:800, at 180 to 280 °C and 5 MPa H₂, the process is expected to take 0.5 to 2 h. Rapid cooling will be



required after the reaction. After transferring the products to a test tube and diluting them to 4–5 g, solid–liquid separation will be performed by centrifugation. Further separation and detection will then be performed using gas chromatography.

3.2.3 GVL to butene

A ANN PARAMINE Butene consists of three possible isomers that are important components of fuel (Fig. 3.4).

$$C_5H_8O_2 == C_4H_8 + CO_2$$

Chemical Formula: C4H8



Fig 3.4 Reaction Mechanism of the Conversion from GVL to Butene

Experiment 1: This experiment will employ a heating mixing control instrument and a 50-mL autoclave with a PTFE lining tube as a reaction vessel to simulate an industrial experiment. Autoclaves will be placed into a 900-r/min magnetic stirring heating furnace. Then, 2 g of GVL as reactant and 5 wt% HZSM-5 catalyst will be added, and the temperature adjusted while collecting the gaseous products from the reaction at regular time intervals. After the reaction, the autoclave will be cleaned with ethyl alcohol to collect the remaining product, and then it will be mixed with the previously collected gaseous products. Further analyses will be performed using gas chromatography.

Experiment 2: This experiment will be performed in a fixed-bed reactor using HZSM-5 as a catalyst. The fixed-bed reactor has an interior 60-cm-long reaction tube 1 cm in diameter. The tube is filled with SiO_2 and catalyst from the bottom to the top. For the reaction, 4-MPa N2 pressure will be required. After 20 min of preheating, liquid reactant will be pumped into the reactor at 6 mL/h. The product will be collected at regular time intervals. Further analyses will then be performed using gas chromatography.

Butene/isobutane to gasoline 3.2.4

Gasoline is a mixture of hydrocarbons and is judged mostly by the octane number criterion. This step of the experiment aims to produce gasoline with a fairly high research octane number (RON) (Fig. 3.5).



Fig. 3.5 Reaction Mechanism of the Conversion from Butene to Fuel with Sulfuric Acid as Catalyst

This experiment will be performed in a fixed-bed reactor and autoclave using sulfuric acid as a catalyst. N2 pressure will be required for the reaction to liquefy the reactants. After precooling to 5 °C, the reactant will be pumped into the reactor with 37.2 mL of isobutane and 12.8 mL of butene and allowed to react for 20 min. Finally, the product will be extracted from the reaction liquid, and further analyses will be performed using gas chromatography.

3.2.5 Analytical experiments

This research will use a Shimadzu gas chromatograph. The InertCap-FFAP 0.25- $mm \times 0.25$ - $mm \times 30$ -m column for gas-phase analysis will be used for sampling and analyses. In this study, the vaporizing chamber temperature will be set to 260 °C, and a flame ionization detector (FID) at 260 °C will be employed. To improve the detection speed and accentuate the peaks, the following temperature program will be used: 40 °C for 1 min, increase at 15 °C/min to 150 °C, increase at 10 °C/min to 230 °C, and hold at 230 °C for 10 min.

A Shimadzu liquid chromatograph will also be used. The Shodex Sugar SC1011 $0.25 \text{ mm} \times 0.25 \text{ mm} \times 30 \text{ m}$ phase analysis column will be used for sampling and analysis. This study will use water as the mobile phase at a flow rate of 0.8 mL/min, measuring temperature of 80 °C, and testing time of 30 min. A diode array detector (UV), will be used to eliminate differential detection to determine the chemical components of the collected products.

3.3 Risk and Safety

Because this project is mainly chemistry-based, the risk of the hazardous chemical compounds used must be considered, along with relevant safety measures. This project will ensure the safety during experimentation and provide solutions to emergency situations in two ways.

3.3.1 Precaution

- ANN FURIN (1) All reactants will be obtained from legal and safe companies with stellar reputations: straw powder (corn straws, countryside, Beijing), glucose (AR, Sinopharm Chemical Reagent Co., Ltd.), cellulose (column chromatography, Sinopharm Chemical Reagent Co., Ltd.), levulinic acid (LA, 98%, Alfa Aesar Chemical Co., Ltd.), γ-valerolactone (GVL, 98%), isobutane and butene (Beijing Yongsheng Gas Technology Co., Ltd.), sulfuric acid (H₂SO₄, 2–3%, Beijing Beihua Fine Chemicals Co., Ltd.), ruthenium/carbon (Ru/C 5%, 50% water, Tokyo Chemical Industry), H-ZSM5 (Tianjin Nankai catalyst), and mesoporous molecular sieves (SiO₂/Al₂O₃).
- (2) Every reagent will be properly stored in the laboratory. Before performing the experiment, laboratory rules and procedures should be reviewed. The researcher must wear specific personal safety equipment, including masks, gloves, glasses, and suit when performing experiments. After finishing the experiment, reagents should be placed in their designated storage areas to avoid risk and to make them readily available for other experiments.

3.3.2 **Emergent solutions**

- (1) Flammable and explosive products may be generated during the experiment under high temperature and pressure. Thus, a detailed procedure should be in place prior to beginning the experiment to prevent injury and other dangers. Butene and butane used in the project are combustible gases. The experiments should be performed in a draught cupboard to reduce the risk of combustion. A fire extinguisher should be placed near the experimental site, and an alarm apparatus should be installed in the room. When the alarm rings, researchers should leave the room and staff will assess and mitigate the risk. (According to laboratory safety procedures of the Institute of Process Engineering, CAS)
- (2) When unexpected injury occurs, first-aid measures should be performed immediately. A first-aid bag should be made available in the laboratory with useful tools and bandages in a location that is easy to access.

3.4 Data Analysis

Data analysis will consist of four parts in this project. First, software provided with the liquid and gas chromatographs will be used to obtain relevant data, such as peak areas, that can be used to determine concentration. Second, standard reagents will be used to generate standard curves to calculate the yield and selectivity of the reaction. Third, statistical methods will be used to compare each reaction approach based on predetermined factors. Last, the results will be synthesized to propose a complete conversion methodology.

4 RESULTS AND ANALYSIS

4.1 Straws (Cellulose) to LA (levulinic acid)

The conversion of waste straws into high-octane gasoline consists of four principle organic experiments, which will be discussed separately in this section.

According to the hydrolysis reaction of cellulose, the product is first glucose, which produces high yield and will turn into LA afterward: $(C_6H_{10}O_5)n$ (cellulose)+ $nH_2O = nC_6H_{12}O_6$ (glucose). Therefore, this research addresses the hydrolysis of glucose to simplify the research procedure.



Fig. 4.1 Liquid Chromatogram of Liquid Products

(1) High LA content is found in the product of hydrolysis of glucose

Fig. 4.1 is the liquid chromatogram of the liquid products of 0.05-g glucose and 2g–3% sulfuric acid under 210° C/1-MPa N₂ condition for 30 min. In the left-hand side, the peak with a retention time of 9.09 min is defined as the catalyst sulfuric acid, and the peak with a retention time of 13.06 min is predicted to be levulinic acid. After adding a certain amount of levulinic acid to the product, the area of the peak at approximately 13 min increased noticeably, which proved the existence of levulinic acid.



Fig. 4.2 Liquid Chromatogram of Liquid Products + LA





Fig. 4.3 Peak Areas of LA in Conversion of Glucose under Different Pressures

From Fig. 4.3, we can see that no levulinic acid can be found in the product without any pressure added to the reaction, whereas, when certain pressure is applied, there is no obvious difference of the amount of levulinic acid in the product as the pressure increases. Therefore, a certain pressure provides the experiment with a certain pushing force.

(3) Ratio between catalyst and reactant affects the result greatly

Using the equivalence between the area of peaks and the concentration calculated according to the standard concentration curve (Fig 4.5), the index in the conversion to LA under different reaction conditions is provided in Table 4.1.

	Reactant	Mass (g)	Selectivity	Concentration	Yield
			(%)	(g/g)	(%)
А	cellulose: 2g catalyst	0.05	82.3	0.01000	40.0
В	Straw: 2g catalyst	0.05	72.3	0.00750	30.0
С	Straw: 2g catalyst	0.10	85.2	0.00865	17.3

Table 4.1 Conclusion of Index in the Conversion to LA under Different Reaction Conditions

After calculation, we get three samples of levulinic acid's selectivity, concentration, and yield, shown in Table 4.1 (A, B, C). The cellulose content in the straw is 40~50%, so the same mass of straw and cellulose converts into levulinic acid and should result in a product with a ratio of 1:2 in theory. Figure 4.4 shows, under different reaction conditions, a liquid chromatogram analysis of the conversion of levulinic acid.

When comparing Figs. 4.4 A and 4.4 B, the proportion is higher than 1:2, showing that the reaction of straw fiber has a higher yield, but poor selectivity. A possible reason is that the straw and other ingredients are converted into levulinic acid under this condition, so the yield increases; because of the complexity of straw composition, which also can convert into more-complex products, the selectivity reduces.

Comparing Figs. 4.4 A and 4.4 C, A is smaller. The concentration and yield in Fig. 4.4 C are significantly lower than in Fig. 4.4 A, proving that the catalyst's relative amount is too small and the yield of the reaction is lower. When the catalyst is 1:40, selectivity is lower than the catalyst in the case of 1:20 and the concentration close to that when the yield of 1:40 is nearly two times that of 1:20. In industrial reactions, the dosage of raw materials must be a high and the importance of yield affects more than selective, so a 1:40 catalyst ratio is chosen, which can save raw materials and better achieve benefits. At the same time, in the experimental results shown in Fig. 4.4 B, in the catalyst ratio 1:40, sulfuric acid catalyst in reaction products that have not been used completely in the catalyst in the reaction process. If used for an appropriately longer reaction time, the use of the catalyst could improve in the subsequent reaction.





Fig. 4.4 Liquid Chromatogram of Liquid Products in the Conversion to LA under Different Reaction Conditions (A, B, and C)

In conclusion, when the temperature reaches 210° C with 1-MPa N₂ and reacts for 30 min with a ratio between catalyst and reactant of 1:40, the best performance is obtained, with a yield of 30%.



Fig. 4.5 Concentration Peak Area Standard Curve of LA

4.2 LA to GVL



(1) GVL exists in the product

Gas chromatography was used to analyze the product of this experiment. A preexperiment was done to determine whether the conversion is achievable. In the preexperiment, 2 g of LA and 0.01 g of Ru/C with 5 MPa H₂ at 220 °C and reacted for 30 min were converted into GVL. The result is shown in Fig. 4.6, in which the ratio of GVL content in the product is obviously higher than any other components at a conversion rate of nearly 100%. The conversion of LA to GVL was proved to be achievable.

(2) Effect of the ratio between catalyst and reactant

 H_2 is one of the reactants in this experiment, so pressure has little range to be adjusted as a parameter. In recent research, the approach that reached the best result was to use 0.1% Au catalyst to obtain a 90% yield. Although using precious metals has the drawback of losing activity in a short period, Pt/Au catalyst appears to be fairly expensive for industrial uses. Therefore, this research attempts to use the less-expensive Ru/C to achieve the highest possible yield.

Fig. 4.8 shows the result of different ratios between catalyst and reactant (Ru/C and LA) with 5-MPa H_2 at 220°C and reacted for 30 min to convert into GVL. With the increase of the ratio, the increase of yield slows down. Because the yield and the conversion rate rise at the same time, when the ratio is 1:400, a turning point occurs. No apparent improvement of yield and conversion rate appears when the ratio is 1:200, which can be the evidence of a waste of catalyst when the ratio is more than 1:400. Because the selectivity of the reaction are all higher than 95%, a ratio of 1:400 is considered to be the best solution.



Fig 4.6 Gas Chromatogram of Liquid Products in Conversion to GVL



Fig. 4.7 Concentration Peak Area Standard Curve of GVL

Fig. 4.8 Conversion Rate and Yield with different consumption of catalyst

In conclusion, when the temperature reaches 220°C with 5-MPa H2 and reacts for 30 min with a ratio between catalyst and reactant of 1:400, the best performance results, offering a yield of 75.2% and a conversion rate of 91.4%. Although the amount of Ru on earth is poor, its price is the lowest among all precious metals and is one-tenth that of Au. Using 0.25% Ru/C can produce a yield of 75.2%, which is more economical than the method of using Pt/Au.

4.3 GVL to Butene

Fig. 4.9 shows the conversion rate from GVL to butene and the percentage of each component in the product. The reaction requires high pressure, so this stage of research did not consider pressure as a main factor. The aim is to produce trans-2-butene, 1-butene, and cis-2-butene. Fig. 4.9 shows that, as the temperature increases, the yield and conversion rate rise, while the selectivity drops. When the temperature was 350°C, the highest yield was 97% with 30% trans-2-butene, 39% 1-butene, and 28% cis-2-butene. When the temperature was more than 350°C, the yield decreases, and when the temperature was 400°C, the yield was less than 95%. This result is probably caused by the increase of carbonization and splitting decomposition.



Fig 4.9. Percentages of Different Products in the Conversion of GVL to Butene at Different Temperatures

When the temperature reaches 350°C with 4-MPa N2 and the speed of pumping was 6 mL/h with 10mL H-ZSM5 catalyst, a yield of 97% can be reached.

4.4 Butene/Isobutane to Gasoline

In this stage of the research, the focus is the catalyst. After doing several experiments with different catalysts, the best results obtained are shown in Table 4.2.

Table 4.2 shows that all three catalysts produced effective results. The equal of RON is: $RON = \sum_{i=1}^{n} w_i RON_i$ Table 4.2 Index with Different Catalysts Involved

Table 4.3 shows that whether the gasoline is highly effective or not largely depends on the percentage of TMP, C8, and C9 in the product. In the three reactions above, the percentage of C8 included in the product catalyzed by sulfuric acid was higher than that of the other two reactions, while the differences between C5 able 4.2 malex with Different Catalysis involved

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Catalyst	Pyridine- HF	BMIM]PF ₆ -HF	H2SO4
olefin/alkane	1:10	1:10	1:10
time (min.)	10	10	10
T (°C)	35	15	10
C ₅ -C ₇	12.4	9.4	5.31
C ₈	76.1	59.1	83.88
TMP	46.8	31.7	58.5
C_9^+	11.5	31.5	10.81
RON	88.9	91.2	94.9

and C7 are not that obvious. Although sulfuric acid performs the best among the three, it still cannot compensate for the non-environmentally-friendly factor. However, the production of [BMIM]PF6-HF is much more complex and time consuming than that of sulfuric acid. Sulfuric acid remains the best, but research of [BMIM]PF6-HF might hold the key to the future.



When the temperature reaches 10° C with N₂ pumped into the system during the process, 37.2mL isobutane and 12.8mL butene reacting for 10 min can produce gasoline with a high octane number of 94.9 (RON).

4.5 Improved approach

1. When the temperature reaches 210° C with 1-MPa N₂ and reacts for 30 min with a ratio between catalyst and reactant of 1:40, the best performance is obtained, offering a yield of 30%.

2. When the temperature reaches 220° C with 5-MPa H₂ and reacts for 30 min with a ratio between catalyst and reactant of 1:400, the best performance yields 75.2% and has a conversion rate of 91.4%. 3. When the temperature reaches 350°C with 4-MPa N₂

Table 4.3 Octane Number of Different Components					
С		gasoline alkylate	RON		
C ₅		2- Dimethylbutane	92.3		
C ₆		2,3- Dimethylbutane	103. 2		
$C_6 \sim C_7$			80.0		
		2, 2, 4-Trimethylpentane	100. 0		
	тмр	2, 2, 3-Trimethylpentane	109.6		
C	IMP	2, 3, 4-Trimethylpentane	102.7		
		2, 3, 3-Trimethylpentane	106.1		
C_8	DMH	2, 5- Timethylhexane	55.2		
		2, 4-Timethylhexane	65.2		
		2, 3-Timethylhexane	71.3		
		2, 2-Timethylhexane	72.0		
C ₉		2, 2, 5-Rimethylhexane	91.0		
C ₉ +			80.0		
C ₁₂ +			80.0		

and the speed of pumping is 6 mL/h with 10Ml H-ZSM5 catalyst, a yield of 97% is obtained.

4. When the temperature reaches 10° C with N₂ pumped into the system during the process, 37.2mL isobutane and 12.8mL butene reacting for 10 min can produce gasoline with an octane number of 94.9 (RON).

Table 4.4 Index During the Conversion from	/asted Straw to Fuel with High Octane Number
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Turne of Fur	Temp	Time	Pre.	Material		Catalyst	
Type of Exp.	°C	min	MPa/N ₂	Туре	Quant.	Туре	Quant.
Cellulose to LA	210	30	1	Cellulose	0.05 g	$\mathrm{H}_2\mathrm{SO}_4$	2 g-3%
LA to GVL	220	30	5	LA	2.00 g	Ru/C	0.005 g
GVL to Butene	350	-	4	GVL	6. 0 mL/h	H-ZSM5	10.0 mL

Butene/Isobutane Butene 37.2 mL							THE A	M. S.
to Gasoline Isobutane 12.8 mL	Butene/Isobutane to Gasoline	10	20	Butene Isobutane	37.2 mL 12.8 mL	H ₂ SO ₄	10.0 mL	

After evaluation, 100 mass straw can produce 21.86 mass 94.9-octane gasoline, with a consumption of 0.075 mass of Ru/C and 4000 H_2SO_4 , which is reusable, and costs only come from 0.075 mass of Ru/C under ideal circumstances.

5 CONCLUSIONS AND INNOVATION

5.1 Conclusions and innovation

- (1) High-octane-number gasoline conversion was implemented. A complete conversion method was developed, and it obtained high-octane gasoline with an octane number of 94.9 and a yield of 21.86%.
- (2) The cost and energy consumption were reduced. The use of nonnoble-metal ruthenium/carbon (Ru/C) as reaction catalyst was studied. It replaced gold/platinum (Pt/Au) catalyst for the catalytic reaction, and the conversion rate reached 91.4% producing a yield of 75.2%, resulting in a high catalytic conversion effect and effectively reducing the cost and energy consumption in the transformation process.
- (3) The conversion parameters were optimized. The key factors in the process were studied, and a parameter setting and optimized scheme were presented.
- (4) Heavy-metal pollution was reduced. Using straw biomass as the raw material and Ru/C as catalyst effectively reduced heavy-metal pollution in the process and in the product (such as gold, silver, copper, iron, and lead).

5.2 Suggestions and Outlook

Suggestion: On-site incineration of agricultural products or packing of the straw should be replaced by the government or relevant organizations' unified dealing.

Outlook: This study provided solutions for testing using laboratory simulation, and not a full-scale of operation. Hence, the next step for research should focus on:

- (1) Environmental factors, through catalyst recycling to reduce the impact of environmental pollution
- (2) Maintaining the advantage of the model state reaction and conversion rate. Test specification should be gradually expanded, through large-scale experiments, ranging from small to pilot to demonstration.



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