Study of a New Process for the Preparation of Butyl Levulinate from Cellulose
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ABSTRACT: Butyl levulinate (BL) is a versatile chemical utilized widely in food and chemical industries, the production of which by using cellulose in biomass resources is of great significance to its sustainable development. Traditional synthesis processes for n-butyl levulinate are confronted with various problems such as high cost of raw materials, difficulty in separating products, etc. In this paper, a novel process for the preparation of BL from cellulose is proposed. The process is composed of five main unit operations including fed-batch hydrolysis, decolorization, extraction, esterification and purification. A 171.63 g/L concentration of the intermediate levulinic acid was obtained at the fifth feeding through the fed-batch hydrolysis process. The resin-activated carbon secondary decolorization method was adopted to remove the soluble humin byproducts with an accumulative decolorization rate of 89%. In the extraction process, the product BL was chosen as the extractant to avoid the introduction of new impurities. After purification, the purity of the final product BL reaches up to 98 wt %. The proposed technique allows for cost-effective and eco-friendly production of BL from biomass resources.

1. INTRODUCTION
Biomass resources are known as low cost, renewable, extensive, and innocuous to the environment, the utilization of which to produce correlative chemicals is of great significance to sustainable development.1,2 Cellulose, as a typical biomass resource, can be converted into biofuel and chemicals through biological or chemical methods3,4 such as bioethanol,5 levulinic acid (LA),6 LA esters,7 etc. Among them, LA esters are very promising chemicals, which can be used not only as fragrances, food additives, and plasticizers8-10 but also as important intermediates in chemical and pharmaceutical industries.11,12 In addition, due to similar properties to biodiesel, LA esters can be applied as blend fuels and additives (usually with low LA ester concentrations) for gasoline, petrochemical diesel, and biodiesel, owing to their advantages such as low toxicity, stable flash point, excellent lubricity, and good low-temperature fluidity.13,14

Butyl levulinate (BL), as a kind of LA ester, has been widely applied in food and chemical industries. At present, there are three methods for the synthesis of BL. The first is direct esterification of LA with n-butanol. Dharne et al.15 synthesized BL by esterification of LA and n-butanol with a heteropoly acid catalyst. The conversion rate could reach 97%. Maheria et al.16 developed acidic zeolites for synthesizing BL through esterification of lignocellulose-derived LA and n-butanol. Direct conversion of LA has the advantages of simple process, high conversion rate, and high purity of the BL product. However, most of previous studies merely focus on the process of the conversion of various types of feedstocks to LA.17,18 Only a few studies have reported the separation and purification processes of extraction,19,20 electro dialysis,21 and direct distillation22 to obtain LA from the hydrolysate. Considering the cost and effect of separation, extraction is the most widely studied process with complex techniques, including cellulose-hydrolysis, concentration, extraction, back-extraction, and vacuum distillation operation. These complex processes result in considerable cost in extracting LA. Besides, the addition of an extractant in the extraction process brings other impurities, which is not conducive for product separation and purification. The second method is furfuryl alcohol conversion. Furfuryl alcohol can produce LA esters by heating alcoholysis with an acidic catalyst. Chang et al.23 converted the furfuryl alcohol to BL in n-butanol with ferric sulfate as the catalyst with the yield of BL up to 86.7%. Mohammadbagheri et al.24 obtained BL from the furfuryl alcohol alcoholysis in n-butanol with KCC-1/Pr-SO3H as the catalyst. An yield of BL as high as 81.9% was achieved with some side reactions. Besides, the reaction conditions and the catalyst have a great influence on the yield of the product. However, as the raw material for BL production, furfuryl alcohol is also an important platform chemical, which needs to be converted from hemicellulose in the biomass. Hence, high prices of raw material limit BL production on a large scale. The third method refers to the synthesis of BL by adding biomass raw materials, alcohols, and catalysts to the reactor simultaneously for one-step alcoholysis—esterification under high temperature conditions. Deng et al.25 adopted the alcoholysis of cellulose in n-butanol to synthesize BL catalyzed by metal sulfate. Hishikawa et al.26 studied the direct synthesis of BL from cellulose catalyzed by a sulfuric acid catalyst in butanol. This
method has various advantages such as simple process, wide source of raw materials, low cost, short reaction route, and easy control of process conditions. However, the disadvantages of this method are the formation of more byproducts and a complex separation process in the later stage. At present, there is no suitable industrial production process for preparing BL from low cost raw materials. A completely new process method with low cost and wide source of raw materials, high product purity, and capability for large-scale production is therefore required.

In this paper, cellulose is used as a raw material to study the new process of producing BL through direct acid hydrolysis. The final product BL is chosen as the extractant to avoid introducing other impurities. Unit operations including fed-batch hydrolysis, decolorization, and extraction in the whole process are discussed, whereas optimal conditions for each unit operation are given. The recycling of materials in the process is further optimized, allowing for reduced energy consumption and waste emission.

2. MATERIALS AND METHODS

2.1. Materials. All experimental reagents utilized in this study are listed in Table 1.

2.2. Experimental Procedure. The experimental process is shown in Figure 1. Note that to avoid the introduction of other organic impurities, the crude product BL was selected as the extractant and was recycled for extraction operation after the esterification reaction. Process system engineering was applied in this process, as most of the materials were recycled.

2.2.1. Fed-Batch Hydrolysis. The experiment was carried out in a 500 mL reaction vessel. The inner wall of the reactor, which was contacted with raw materials, was made of Hastelloy (C276) for preventing corrosion under acidic conditions. Cellulose (45 g) and 300 mL of 0.5 mol/L sulfuric acid solution were placed into the reaction vessel. The reaction was maintained at 453.15 K for 50 min, and the vessel impeller speed was set to 400 rpm. The reaction under the above conditions could ensure that the cellulose was fully converted. After the reaction, the filter residue was separated and the volume of the filtrate was measured. The same volume of 0.5 mol/L sulfuric acid solution as the hydrolysate adsorbed by the residue was added for washing. Later, the washing solution was added into the filtrate to recover the reaction solution to 300 mL for the next hydrolysis reaction. The reaction was continued to hydrolyze new batches of cellulose by adding the recovered hydrolysate from the previous step while maintaining the same solid−liquid ratio. The reaction conditions were kept the same as those for the first hydrolysis step to ensure that the cellulose would be completely converted. This process was referred to as the second batch hydrolysis. Based on this method, the hydrolysate was continuously adopted to hydrolyze the cellulose.

2.2.2. Decolorization by Resin and Resin Regeneration. For this, 15 g of AB-8 macroporous adsorption wet resin was placed in a glass column. Then, 75 mL of the hydrolysate (solid−liquid ratio is 1:5) was pumped into the column with a flow rate of 1 BV/h at room temperature, and the effluent was collected. The hydrolysate before and after decolorization was diluted 25 times, respectively. Then, the absorbance of the hydrolysate before and after decolorization was measured by a spectrophotometer (V-5100B, YuanXi Instrument Analysis Co., Ltd., Shanghai) at the wavelength of 420 nm. The aforementioned decolorization experiment was repeated afterwards by switching the solid−liquid ratio to 1:10, 1:15, and 1:20, respectively, and the absorbance of hydrolysates was measured. After decolorization, the resin was eluted with 15 mL of 333.15 K hot water for removing the LA and formic acid adsorbed on the resin. The eluent was mixed with the hydrolysate for subsequent operations, and the used resins were regenerated. For resin regeneration, the used resin was

| reagent                        | specification  | manufacturer                      | CAS number |
|-------------------------------|----------------|-----------------------------------|------------|
| microcrystalline cellulose    | chromatographically pure | Sinopharm chemical, Shanghai | 9004-34-6  |
| sulfuric acid                 | 98 wt %        | Sinopharm chemical, Shanghai      | 7664-93-9  |
| deionized water               |                | homemade                          | 7732-18-5  |
| levulinic acid                | chromatographically pure | Aladdin, Shanghai  | 123-76-2   |
| ethyl benzoate                | chromatographically pure | Sinopharm chemical, Shanghai | 93-89-0    |
| n-buty1 levulinate            | chromatographically pure | Aladdin, Shanghai  | 2052-15-5  |
| AB-8 macroporous adsorption resin | analytically pure | Nankai University Chemical Factory, Tianjing | --        |
| ethanol                       | analytically pure | Sinopharm chemical, Shanghai      | 64-17-5    |
| activated carbon              | analytically pure | Aladdin, Shanghai  | 7440-44-0  |
| sodium bicarbonate            | analytically pure | Sinopharm chemical, Shanghai      | 144-55-8   |

Figure 1. Overview of the experimental process.
added into 30 mL of ethanol and placed in a magnetic stirrer at 333.15 K stirring for 30 min. To completely regenerate the resin, the resin was separated from ethanol by a sand core funnel and then was added into 30 mL of fresh ethanol to repeat the above operation. Finally, the regenerated resin was fully washed with deionized water to remove ethanol and used for the next decolorization. The used ethanol was distilled and collected for recycling. The remaining solid humins after evaporation of ethanol were mixed with the residue of the feed-hydrolysis reaction for preparing activated carbon raw materials.

2.2.3. Decolorization by Activated Carbon. For further decolorization, 50 mL of the hydrolysate obtained from resin decolorization was mixed with 0.25 g of activated carbon and placed in a magnetic stirred vessel at the temperature of 333.15 K (the best decolorization performance of activated carbon was found experimentally at a temperature of 333.15 K) and stirred for 30 min. After decolorization, the mixture was filtered through a 0.45 μm filter to obtain the clear hydrolysate and the used activated carbon. The absorbance of the hydrolysate after decolorization was measured. The above experiment was repeated by changing the amount of activated carbon to 0.125 g, 1.25 g, 0.5 g, and 0.375 g with the resin decolorized hydrolysate, respectively (while keeping the solid—liquid ratio of the resin and hydrolysate at 1:15). The used activated carbon was mixed with the residue for producing microporous activated carbon.

2.2.4. Extraction Operation. For this, 200 mL of the final product butyl levulinate and 200 mL of the decolorized hydrolysate were thoroughly mixed for 30 min. After shaking, the solution was poured into a 1000 mL separatory funnel and allowed to stand for 15 min. The upper extractant and the lower raffinate were separately discharged and collected. A large amount of LA retained in the raffinate solution was collected and used as the solvent for the hydrolysis process. The extractant was reserved for the subsequent step operation.

2.2.5. Estimation Reaction. The extractant including LA was flashed under 373.15 K to distill water and formic acid. After flash evaporation, excess n-butanol and 0.5 mL of sulfuric acid (served as the catalyst) were added into the extractant. The reaction was heated in an oil bath heating apparatus and was carried out under the following conditions: the reaction temperature was maintained at 373.15 K; the nitrogen gas rate was controlled to 100 mL/min; the stirring speed was set to 100 rpm. Nitrogen was adopted to bring the water—n-butanol azeotrope into the condenser. Then, the water—n-butanol azeotrope was cooled back to the lower storage tube in the condenser and stratified. As the reaction progresses, the liquid level in the storage tube rises up continuously, whilst the upper layer of n-butanol can be returned from the liquid storage tube to the flask to participate in the reaction. The lower layer of water can be discharged through the valve, ensuring that the esterification reaction continues. The reaction was conducted for 3 h until no more water was evaporated. The evaporated water in the esterification reaction could be recycled to the hydrolysis operation as new solvent.

2.2.6. Purification of Butyl Levulinate. After the esterification reaction, the solution mainly contains butyl levulinate, unreacted n-butanol, sulfuric acid catalyst, soluble humins, and humin esters. To obtain pure butyl levulinate, further refining of the solution is required. The solution was first distilled to remove the unreacted n-butanol at a temperature of 403.15 K. Part of the crude product, i.e., BL, was used as the extractant to circulate to the extraction process. The remaining crude ester solution was washed successively with saturated sodium carbonate solution and water to remove the sulfuric acid and salt, respectively. The crude ester solution after washing was distilled at an absolute pressure of 0.02 MPa and a temperature of 413.15 K to obtain a transparent BL product.

2.3. Analytical Method. The absorbance of the hydrolysate was measured by a spectrophotometer (Jingke V-5100B, Shanghai, China) at a wavelength of 420 nm. The decolorization rate E can be expressed as

\[ E = \left( \frac{A_0 - A}{A_0} \right) \times 100\% \]  

where \( A_0 \) denotes the absorbance of the hydrolysate and \( A \) is the absorbance of the hydrolysate after decolorization.

Gas chromatography (Shimadzu) with a FFAP (30 m × 0.32 mm × 0.33 μm) capillary column was used to determine the content of LA and BL in the sample. \( N_2 \) (High purity, 99.99%) was used as the carrier, and the flow rate of the carrier was selected as 15 mL/min. Ethyl benzoate was used as the internal standard. The initial temperature of the oven was set to 363.15 K and was maintained at this temperature for 3 min. Then, the column oven was heated at a rate of 10 K/min and the sample was separated in the temperature programming process. After the temperature reached 503.15 K, the process was maintained at this temperature for 2 min. The temperature of the sampler was 513.15 K, and the temperature of the detector was 523.15 K. A standard curve was plotted by using the chromatographic pure chemical. The concentration of each component in the samples was measured thereafter by standard curves.

3. RESULTS AND DISCUSSION

3.1. Study of the Fed-Batch Hydrolysis. The traditional hydrolysis process yields low concentrations of levulinic acid, which is not conducive with respect to LA separation. In our previous study, a new fed-batch hydrolysis process for increasing the concentration of LA in the hydrolysate was developed. This new fed-batch hydrolysis process was adopted in this work. As shown in Figure 2, the concentration of the LA increased with the number of hydrolysis batches. A 171.63 g/L concentration of the LA was obtained at the fifth feeding, which effectively reduces the amount of acid catalyst used in the hydrolysis. As a result, the fed-batch hydrolysis can effectively reduce the energy consumption for the subsequent separation and purification processes in industrial production.

Figure 2. Variation of the LA concentration with feeding times of the hydrolysis.
However, with increasing feeding time, the increment of the LA concentration exhibited a gradual moderate decline with a greater decline for the fifth feeding. In particular, the concentration increment was only 15.98 g/L at the fifth feeding, compared to that of 27.84 g/L at the fourth feeding. Since the yield of LA decreased significantly between the fourth and fifth feedings, a four times hydrolysis feeding is considered to be reasonable, as it not only obtains relatively high concentrations of levulinic acid but also effectively reduces the operating costs.

3.2. Effect of Resin Content on Decolorization of the Hydrolysate. The cellulose hydrolysate contains a large amount of red-brown soluble humins.29,30 As shown in Figure 3, extraction of the hydrolysate without decolorization causes serious pollution on the extractant (upper layer solution). In addition, soluble humins extracted by the extractant may react with n-butanol to form humin esters. Consequently, n-butanol is excessively consumed and byproducts humin esters are not conducive to the separation and purification of butyl levulinate. Therefore, it is necessary to decolorize the hydrolysate before extraction to remove the soluble humin impurities.

Due to the weak adsorption of short-chain organic acids on macroporous adsorption resin, the adsorption capacity of organic acids is generally low. Nevertheless, soluble humins have large molecular weight, the molecular structure of which includes a double bond, benzene ring, and furan ring. These functional groups render the large affinity of soluble humins to macroporous adsorption resin, resulting in the high adsorption capacity of soluble humins on the resin. As a kind of weak polar macroporous adsorption resin, AB-8 resins have strong adsorption capacity on soluble humins. Moreover, adsorbed soluble humins are more difficult to be eluted from AB-8 resin as compared to levulonic acid. Therefore, AB-8 macroporous adsorption resin was selected to decolorize the hydrolysate.

The amount of AB-8 adsorbent has a direct effect on the decolorization rate. As shown in Table 2, when the solid–liquid ratios (the ratio of AB-8 resin to hydrolysate) are 1:5, 1:10, 1:15, and 1:20, the corresponding decolorization rates are 82.6, 78.6, 71.2, and 60.7%, respectively. The decolorization rate of the hydrolysate increases with increased amounts of resin. When the solid–liquid ratio increased from 1:10 to 1:5, the decolorization effect of the hydrolysate was not significantly improved. The reason may be due to the variation in the molecular weight of soluble humins in the hydrolysate.

Macroporous resin has an obvious adsorption effect on soluble humins with relatively large molecular weight, rather than that on soluble humins with small molecular weight.

In view of the small molecular weight of the remaining soluble humins, activated carbon with small pore size was selected to further decolorize the hydrolysate. The hydrolysate after resin decolorization with a solid–liquid ratio of 1:15 was employed as the decolorizing solution. Then, different doses of activated carbon were selected for the secondary decolorization. The results are shown in Table 3.

From Table 3, the decolorization rate of the hydrolysate improves with increasing the amount of activated carbon. However, when the amount of activated carbon increased further to 0.375 g, the improvement in the decolorization rate (3.9%) was lower as compared to the former step change (7.4%). This may be attributed to the fact that most majority of soluble humins have been adsorbed by activated carbon at the solid–liquid ratio of 1:200, while more activated carbon added into the hydrolysate would not be an obvious benefit for the decolorization. Therefore, a 1:200 solid–liquid ratio was selected as the optimal condition for the secondary decolorization of the hydrolysate.

To evaluate the combined decolorization of resin and activated carbon on soluble humins, three different amounts of macroporous resins, i.e., solid–liquid ratios of 1:10, 1:15, and 1:20, were selected to decolorize the hydrolysates, respectively. After resin decolorization, the hydrolysates were further decolorized by identical amounts of activated carbon (0.25 g, solid–liquid ratio is 1:200). The results detailed in Table 3 show that the secondary decolorization rate reduces with a decreasing amount of resin added for the first decolorization. This may be attributed to larger amounts of soluble humins with large molecular weight remaining in the hydrolysate when lower amounts of resins were used in first decolorization. During the secondary decolorization, partial pore channels of the activated carbon were likely occupied by those large macromolecular humins, and thus soluble humins with small molecule weights have less opportunity to get into the micropore structure of the activated carbon, resulting in a relatively low secondary decolorization rate of the hydrolysate.

As shown in Table 4 and Figure 4, when the ratio of macroporous resin to hydrolysate is 1:15 and the ratio of activated carbon to hydrolysate is 1:200, a good decolorization effect can be obtained, which not only removes most of the soluble humins in the solution but also reduces the amount of activated carbon and resin.

3.3. Recycling of the Resin. To recycle the resin after decolorization, the resin regeneration process is needed. The resin regeneration process can be referred to Section 2.2.2. The decolorization rate is adopted to represent the decolorization efficiency of the regenerated resin. As shown in Figure 5, the decolorization rate decreased slightly with increasing the times of regenerated resin usage. In particular, a 68.6% decolorization rate.

| Hydrolysate (mL) | Amount of Resin (g) | Solid–Liquid Ratio (g/mL) | Decolorization Rate (%) |
|-----------------|---------------------|---------------------------|-------------------------|
| 75              | 15                  | 1:5                       | 82.6                    |
| 150             | 15                  | 1:10                      | 78.6                    |
| 225             | 15                  | 1:15                      | 71.2                    |
| 300             | 15                  | 1:20                      | 60.7                    |
rate of the hydrolysate can be reached at the seventh use of resin, a bit lower than that of the hydrolysate at the first time (71.4%), indicating that resins still have good decolorization efficiency on hydrolysate after the seventh usage.

### 3.4. Extractant Selection

After decolorization, the LA needs to be extracted from the hydrolysate to the extractant. A large variety of physical solvents have been mentioned for organic acid extraction such as ketones, fatty acids, alcohols, ethers, and halogenated hydrocarbons. In addition, aliphatic amines, organophosphorus, and compound extractants have been used to extract carboxylic acids with high efficiency. However, for LA extraction, all aforementioned extractants are acting as impurities, which give rise to extra difficulties for the subsequent extractant separation from final products. Based on this, the final product BL was chosen as the extractant, due to its specific characteristics such as low solubility in water, safety in use, chemically stable, and especially no extra impurities introduced during the extraction process.

The extraction effects of different amounts of the extractant were investigated, as shown in Table 5. When the ratio of the extractant to the hydrolysate was 1:1, the extraction rate of LA was 44.8%, and the concentration of LA in the extractant was 63.2 g/L. Upon increasing the amount of extractant added, the extraction rate of LA increases, whereas the concentration of LA in the extractant reduces. When the amount of extractant increases to 2:1, the extraction rate reached 61.8%, while the concentration of LA in the extractant was 44.27 g/L. Considering the concentration in the extractant and the extraction rate of levulinic acid, a reasonable ratio of the extractant to the hydrolysate is given as 1.5:1.

### 3.5. Development of the Whole Esterification Process

A detailed process flow chart of the BL production from cellulose is presented in Figure 6. First, the cellulose was hydrolyzed with the fed-batch hydrolysis process to obtain a high concentration of LA up to 155 g/L. Second, the LA hydrolysate was decolorized by both resin and activated carbon to remove the soluble humins, and the accumulative decolorization rate reached 89%. After decolorization, the final product BL, as the extractant, was added to the hydrolysate for extracting LA from the hydrolysate. Then, excess n-butanol was added to the flashed extractant for the esterification reaction. By continuously steaming the byproduct water out of the reactor during esterification, a conversion rate of LA as high as 99.5% was achieved. Finally, the product BL was obtained with a purity of 98 wt % after dealcoholization and purification.

### Table 3. Relationship between Different Solid–Liquid Ratios of Activated Carbon and the Secondary Decolorization Rate

| hydrolysate after resin decolorization (mL) | amount of activated carbon (g) | solid–liquid ratio of secondary decolorization (g/mL) | secondary decolorization rate (%) | accumulative decolorization rate (%) |
|------------------------------------------|--------------------------------|-------------------------------------------------|-----------------------------------|--------------------------------------|
| 50                                       | 0.125                          | 1:400                                           | 36.1                              | 81.6                                 |
| 50                                       | 0.25                           | 1:200                                           | 62.7                              | 89.0                                 |
| 50                                       | 0.375                          | 1:133                                           | 75.5                              | 92.9                                 |

### Table 4. Decolorization Rate of Resin and Activated Carbon

| hydrolysate after resin decolorization (mL) | solid–liquid ratio of resin decolorization | amount of activated carbon (g) | secondary decolorization rate (%) | accumulative decolorization rate (%) |
|------------------------------------------|-------------------------------------------|--------------------------------|-----------------------------------|--------------------------------------|
| 50                                       | 1:10                                      | 0.25                           | 63.1                              | 92.1                                 |
| 50                                       | 1:15                                      | 0.25                           | 62.7                              | 89.0                                 |
| 50                                       | 1:20                                      | 0.25                           | 59.8                              | 84.2                                 |

### Table 5. Extraction Rate with Different Amounts of Extractant

| extractant (mL) | hydrolysate (mL) | LA concentration in the hydrolysate (g/L) | LA concentration in the extractant (g/L) | extraction rate (%) |
|-----------------|------------------|------------------------------------------|------------------------------------------|---------------------|
| 100             | 100              | 150.5                                    | 63.2                                     | 44.8                |
| 150             | 100              | 150.5                                    | 51.8                                     | 54.7                |
| 200             | 100              | 150.5                                    | 44.2                                     | 61.8                |
In the proposed new process, BL was directly synthesized from cellulose. Raw materials can be acquired from cellulose-rich biomass that is widely available, avoiding the use of expensive LA or furfuryl alcohol. High concentrations of the LA hydrolysate through the fed-batch hydrolysis were obtained, thereby reducing energy consumption as compared to ordinary processes with low concentrations of levulinic acid. To remove soluble humins in the hydrolysate, a two-step decolorization method was adopted, using resin-activated carbon, which reduces the burden of the extractant in the extraction operation and avoids other impurities of the esterification reaction. The AB-8 resin can be continuously regenerated and the ethanol regenerant can be recycled in the regeneration process. The activated carbon used in the decolorization process and the distilled solid humins in the regeneration process could be mixed into the hydrolysis residue for further use. In the extraction process, the product BL was chosen as the extractant, avoiding the introduction of other organic impurities. Using BL as the extractant can also avoid separation of LA from the extractant with reducing the separation energy consumption compared to the traditional process for preparing the LA product first and then esterification. A large amount of LA remained in the raffinate, and thus the raffinate can be recycled to the next hydrolysis reaction. The distillate obtained from flash distillation could also be put into the raffinate for the subsequent hydrolysis. Dehydration in esterification can ensure a higher conversion rate and unreacted n-butanol can be used for the next esterification reaction. Overall, most of the materials, including the BL product, can be continuously recycled while the waste residue can be used to produce other chemical products such as microporous activated carbon, allowing for cost-effective preparation of BL by cellulose with reduced waste emission.

4. CONCLUSIONS

In this paper, a new process for the preparation of BL from cellulose was proposed. The process mainly includes unit operations such as fed-batch hydrolysis, decolorization, extraction, esterification, and purification. The fed-batch hydrolysis process can obtain a relatively high LA concentration of 171.63 g/L at the fifth feeding. A two-step decolorization method, i.e., decolorize by AB-8 resin and activated carbon, respectively, was employed to remove soluble humins with an accumulative decolorization rate of 89%. The final product BL was chosen as the extractant to extract LA for the hydrolysate. After esterification and purification, a high purity of 98 wt % was obtained with a yield of BL around 31 mol %. During this process, most of the materials including the final product BL were continuously recycled, while the waste residue can be used to produce other chemical products such as microporous activated carbon, allowing for cost-effective preparation of BL by cellulose with reduced waste emission.

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The authors declare no competing financial interest.

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