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Maximizing phosphorus and potassium recycling by supplementation of lignin combustion ash from dry biorefining of lignocellulose

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HIGHLIGHTS

- Dry biorefining processing maximized water-soluble P and K recycling.
- Recycling of lignin combustion ash significantly reduced use of P and K fertilizer.
- MESP of cellulosic ethanol was reduced by 2.1–3.3%.

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ABSTRACT

Lignocellulose biorefinery produces fuel ethanol and lignin residue byproduct. The combustion ash of lignin residue after electricity generation contains high amount of phosphorous and potassium elements and the recycling to farmland provides a sustainable nutrients supply. This study demonstrated a maximum scenario of water- soluble phosphorous and potassium recycling through a dry biorefining process by reduction of wastewater generation to an extremely low level, in which the soluble phosphorus and potassium compounds were preserved. For biorefining one metric ton of five different lignocellulose biomass, averagely 51–87 kg of lignin combustion ash, 0.75–1.12 kg of phosphorous or equivalent 1.75–2.57 kg of P_2O_5, and 1.03–2.26 kg of potassium or equivalent 1.24–2.73 kg of K_2O were obtained. The recycling of the ash to farmland covered 78–90% and 100% (with surplus) of the world average phosphate and potash fertilizer use, respectively. Even for the need of farmland in China with the heavily overused phosphate and potash fertilizers, the lignin combustion replaced 17–19% of phosphate fertilizer and 30–70% of potash fertilizer. The minimum ethanol selling price was also reduced by 2.1–3.3% by the fertilizer replacement.

1. Introduction

Phosphorus and potassium are essential nutrients to the farmland. Crop cultivation removes considerable amount of phosphorus and potassium from the farmland into crop straw and edible grains, therefore the re-supplementation of phosphorous and potassium is required [1,2]. Currently, chemical phosphate and potash fertilizers are used for supplementing phosphorus and potassium to the farmland in the modern agriculture industry. This is certainly not sustainable because the phosphate and potash fertilizers come from either natural mineral deposits or petroleum based resources [3]. An old tradition in developing countries such as China is to burn crop straws on-site in the farmland after grain harvest and leaves the phosphorus and potassium containing ash in the farmland. However, the straw burning creates heavy air pollution and transportation blockage. Therefore the on-site straw burning has been gradually banned in many countries [4]. An alternative of crop straw burning is to return chopped straws into the farmland directly and simultaneously with grain harvest. Again, this practice has been proved not a proper way for agricultural operation because the decay of the straws is slow [5]. The accumulation of excessive straw in soil negatively affects the crops growth and roots distribution, and consequently reduces the grain yield [6]. Besides, either burning or direct return of crop straw causes the waste of valuable sugars in cellulose and hemicellulose, which is may be used for cellulosic ethanol or bio-based chemical production.

Biorefinery technology provides a different way of phosphorus and potassium recycling without generating air pollution and negatively affecting agricultural operation. Lignocellulose biomass is firstly pretreated, detoxified, then the cellulose and hemicellulose are hydrolyzed and fermented into ethanol [7]. The lignin component is recalcitrant to

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bioconversion and left behind in the stillage slurry of ethanol distillation column. Solid lignin residue is obtained after solid-liquid filtration and its high heating value property makes it an ideal fuel for steam or electricity generation by combustion [8–10]. However, the conventional biorefinery technologies don't work efficiently on phosphorus and potassium recycling because the highly soluble phosphorus and potassium compounds are easily lost along with the great amount of waste water release in each step [3,11–13]. To give the solution for the problem, a recent study proposed a new biorefinery processing technology, dry acid pretreatment and biodetoxification (DryPB) process, to reduce the phosphorus and potassium loss [11]. In this dry biorefining, not only the cellulosic ethanol titer and yield were close to that of corn ethanol, but also the wastewater generation is completely removed from the first step of pretreatment till the end of distillation, in which the lignin rich stillage slurry is the only liquid form of the dry biorefining process [15]. Plenty of the phosphorus and potassium elements still exist inside the combustion ash. The re-supplementation of lignin combustion ash after cellulosic ethanol production to the farmland could provide significant amount of phosphorus and potassium back to the farmland.

In this study, five typical lignocellulose biomass including corn stover, wheat straw, rice straw, sugarcane bagasse and poplar sawdust were selected as the feedstocks for fuel ethanol production. The output of water-soluble phosphorus and potassium elements in lignin combustion ash after dry biorefining (DryPB) from the lignocellulose feedstocks were accurately measured and balanced. The potentials of reducing chemical fertilizers usage and cellulosic ethanol production cost by the application of lignin combustion ash were rigorous estimated. The results provided a practical method for water-soluble phosphorous and potassium recycling by combining biorefinery technology and agriculture operation.

2. Materials and methods

2.1. Raw materials

Corn stover, wheat straw, rice straw, sugarcane bagasse and poplar sawdust were collected from their dominant growing regions in China, Bayan Nur, Inner Mongolia in 2015, Dan Cheng, Henan in 2013, Chang Zhou, Jiangsu in 2014, Bei Hai, Guangxi in 2014 and Yan Cheng, Jiangsu in 2015, respectively. Poplar sawdust was used directly without grinding. The others were ground coarsely using a beater pulverizer and screened through a mesh with the circle diameter of 1.0 cm. The composition of the lignocellulose feedstocks was measured by the two-step acid hydrolysis method according to National Renewable Energy Laboratory (NREL) protocols [16,17], as shown in Table 1.

2.2. Dry acid pretreatment and biodetoxification (DryPB) processing

Cellulosic ethanol was produced from the five feedstocks by dry acid pretreatment and biodetoxification (DryPB) processing technology, including the dry acid pretreatment, solid-state biodetoxification, and high solid loading simultaneous saccharification and co-fermentation (SSCF) [14]. As shown in Fig.1, the feedstocks were pretreated at 175 °C for 5 min with 1.5–2.5 g sulfuric acid per 100 g dry biomass in the 20 L pretreatment reactor after feedstock handing [18–20]. The pretreated feedstock contained approximately 50% (w/w) solid and without wastewater generation. After neutralized to pH of 5.5 with 20% (w/w) calcium hydroxide suspension slurry and briefly milled, the solid state pretreated biomass was inoculated with *Amorphotheca resinae* ZN1 for biodetoxification at 28 °C with aeration of 0.8 vvm for 36–48 h in the 15 L bioreactor [21,22]. The major inhibitors including furfural, 5-hydroxymethylfurfural (HMF), and acetic acid were completely degraded while xylose was well preserved without observable loss.

Table 1
Composition of the five lignocellulose feedstocks on the dry weight base (w/w).

Feedstocks	Cellulose (%)	Hemicellulose (%)	Lignin (%)	Ash (%)
Corn stover	35.4	24.6	16.1	3.5
Wheat straw	38.7	25.9	14.9	5.2
Rice straw	35.3	18.4	22.5	9.3
Sugarcane bagasse	38.8	23.9	26.4	1.3
Poplar sawdust	39.7	16.6	29.4	3.2

Then the feedstock solids were pre-hydrolyzed at 50 °C, pH 4.8 for 12 h at the solid loading of 30% (w/w) and cellulase enzyme dosage of 10 mg protein/g cellulose (Cellic CTec 2.0, Novozyme China) in 5 L fermenter [14,23]. The shortly adapted *Saccharomyces cerevisiae* XH7 seed was inoculated and fermented at 30 °C for 96 h. Samples were periodically withdrawn and for analysis of glucose, xylose, ethanol, glycerol, acetic acid, furfural, and HMF on HPLC (LC-20AT, Shimazu, Kyoto, Japan) equipped with YMC-Pack ODS-A column (YMC, Tokyo, Japan) and an SPD-20 A UV detector (Shimadzu, Kyoto, Japan). The fermentation broth was distillated to obtain 35% (w/w) ethanol stream in glass distillation column with the inner diameter of 40 mm filling with stainless theta ring packings (3 mm in diameter). The stillage from the distillation column was filtrated by a press filter through a fabric cloth to yield the wastewater stream and the lignin residue cake with the 35% water content.

2.3. Assay of elemental content of lignin residue

Lignin residue cake was dried at 105 °C until constant weight and completely burned in a muffle furnace at 573 °C for 3 h. The generated combustion ash was assayed to determine the content of nitrogen content by organic elemental analysis apparatus (Vario EL III, Elementar, Shanghai, China). Water-soluble phosphorus and potassium contents were measured by ICP-OES (Agilent 725ES, Agilent, CA, USA). All assays were done in aqueous solution of combustion ash. The weight change of the solid residues in burning was accurate measured and calculated.

2.4. Process model establishment and techno-economic analysis

The Aspen plus models were established (AspenTech Co., Cambridge, MA, USA) based on the NREL model [8], including ten process areas of feedstock handing (A100), pretreatment (A200), detoxification (A250), hydrolysis and fermentation (A300), cellulase enzyme production (A400), product recovery (A500), wastewater treatment (A600), storage (A700), combustor-boiler-turbogenerator (A800), and utilities (A900). The general process diagram on Aspen plus platform of DryPB biorefining process for cellulosic ethanol production was shown in Fig.2. And the detail process designs for each process area were shown in Figs. A.1(a)–(j). The models were varied in pretreatment, biodetoxification, hydrolysis and fermentation, wastewater treatment according to the DryPB process and the experimental results [15]. The main process input data for the established Aspen plus simulation model of cellulosic ethanol production from five lignocellulose feedstocks was shown in Table A.1. The main reactions in pretreatment, biodetoxifaction, saccharification and fermentation were also shown in Table A.2.

The technical economic evaluation of cellulosic ethanol production was based on the “*n*th-plant” assumption [8]. The process was 2000 metric tons dry lignocellulose feedstock daily and operated for 8,410 h annually. The year of 2013 is used as the reference year for adjusting the quotes of equipment, chemicals and labor in a certain year. The exchange rate from US dollar ($) to Chinese Yuan (CNY) is set to 1:6.2 according to the official announcement in 2013 (http://data.stats.gov.cn/). The prices of the specific equipment in this study including the pretreatment reactor, fermenter and the helical agitator are cited according to the quotes from the related Chinese market, shown in Table A.3 and Table A.4 [24]. The mass and energy balance data are

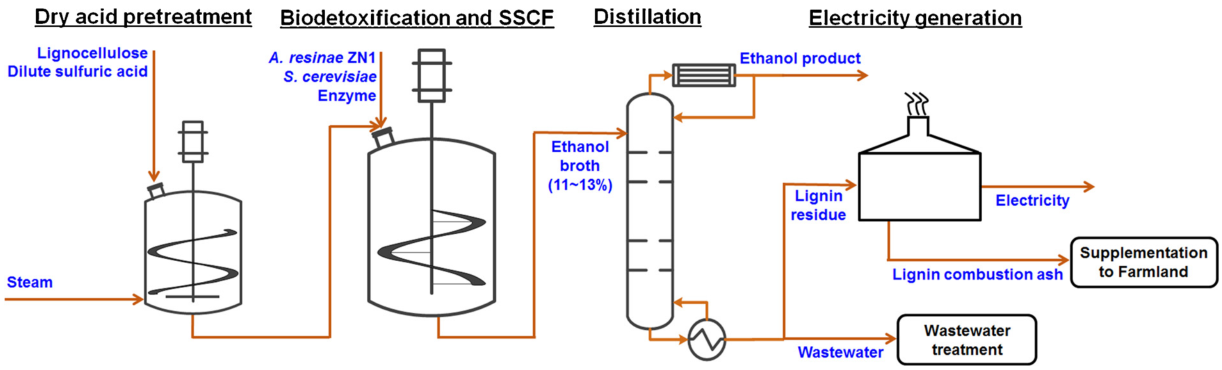

Fig. 1. Flowsheet of DryPB biorefining process for cellulosic ethanol production.

calculated based on Aspen plus simulation. The total capital investment is calculated based on the total equipment cost, and then variable and fixed operating costs are determined according to plant capacity. With these costs, a discounted cash flow rate of return analysis to determine the minimum ethanol selling price (MESP, \$/gal) required to obtain a net present value of zero with 10% internal rate of return after taxes. The calculation process was performed in the Microsoft Excel. Table A.5 shows the assumptive parameters in the discounted cash flow analysis.

3. Results and discussion

3.1. Water-soluble phosphorus and potassium preservation during cellulosic ethanol production process

Five lignocellulose feedstocks were dry acid pretreated and biodetoxified to yield the inhibitor free feedstocks and then the simultaneous saccharification and co-fermentation (SSCF) was followed for ethanol production. The SSCF at high solid loading of 30% (w/w) achieved the high ethanol titer of 85.1, 87.0, 71.9, 78.3, and 79.1 g/L (9.1–11.0% in volumetric percentage) from corn stover, wheat straw, rice straw, sugarcane bagasse, and poplar sawdust, respectively [14]. The feedstocks input and the products output were shown in Table 2: one metric ton of dry biomass feedstock yielded 215–260 kg of ethanol and 355–428 kg of dry lignin residue. The burning of lignin residues yielded 129–203 kg of combustion ash per metric ton dry lignin residue, equivalent to 213–405 kg of combustion ash per metric ton cellulosic ethanol, or 51–87 kg of combustion ash per metric ton of biomass feedstock (Table 3). The contents of nitrogen, phosphorus and potassium elements in combustion ash were determined according to the Materials and Methods section. The nitrogen content in the combustion ash was below the detection line perhaps due to the nitrogen oxides existed in gaseous phase and dispersed during the combustion (data not shown). The contents of water-soluble phosphorus and potassium elements were 9–22 kg and 18–43 kg per metric ton of combustion ash, respectively. These were equivalent to the effective content of fertilizer components, P_2O_5 and K_2O, of 21–50 kg and 22–52 kg per metric ton of combustion ash, respectively (Table 3). For biorefining one metric ton of lignocellulose biomass, averagely 51–87 kg of lignin combustion ash, 0.75–1.12 kg of phosphorous or equivalent to 1.75–2.57 kg of P_2O_5, and 1.03–2.26 kg of potassium or equivalent to 1.24–2.73 kg of K_2O were obtained.

Most of phosphorus and potassium compounds are dissolved in water after pretreatment. In conventional biorefining processes, producing one ton of ethanol is accompanied with approximately several tens of tons to one hundred tons of wastewater generation [15]. This means a considerable amount of phosphorus and potassium compounds are moved to the wastewater stream and less are obtained from lignin residue. This study applied the dry acid pretreatment and

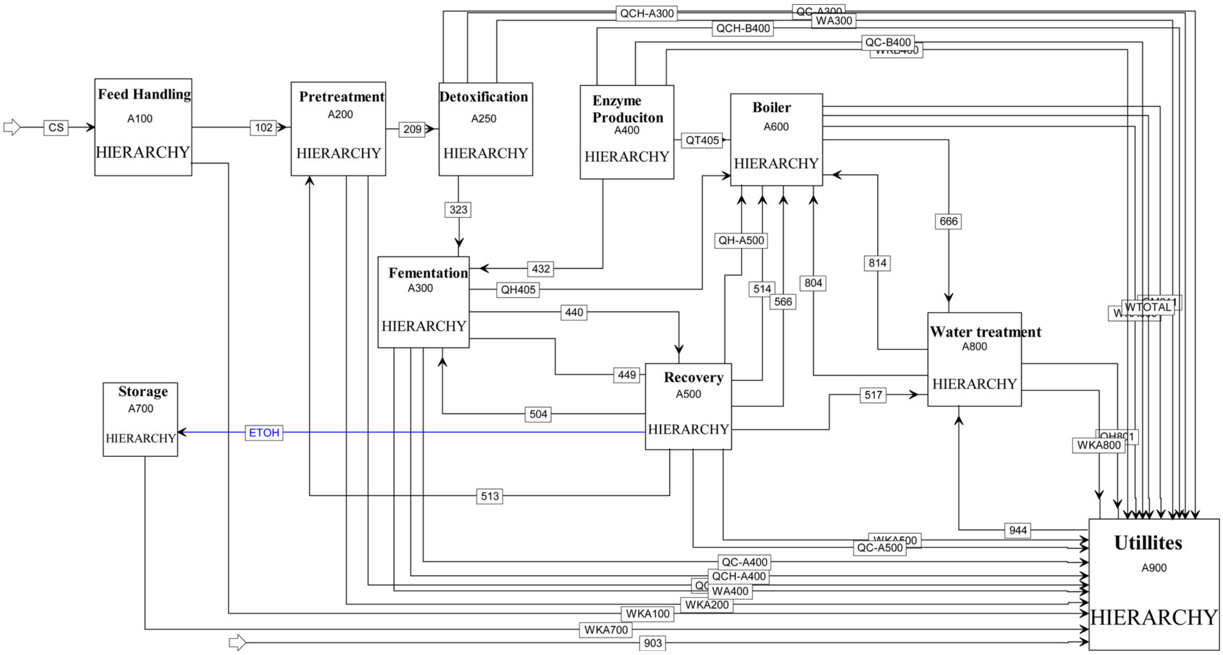

Fig. 2. Process diagram on Aspen plus platform of DryPB biorefining process for cellulosic ethanol production.

Table 2
Feedstocks input and products output from five lignocellulose feedstocks (based on per metric ton of dry biomass feedstock).

	Feedstocks (metric ton)				Products (metric ton)			
	Biomass (dry)	Water	Enzyme	Others	Ethanol	Lignin residue (dry)	Distilled water	Waste water
Corn stover	1.00	1.978	0.041	0.045	0.254	0.384	0.433	2.227
Wheat straw	1.00	1.965	0.044	0.045	0.260	0.355	0.442	2.250
Rice straw	1.00	2.046	0.040	0.045	0.215	0.428	0.365	2.204
Bagasse	1.00	2.009	0.044	0.045	0.234	0.395	0.398	2.216
Poplar sawdust	1.00	2.004	0.045	0.045	0.236	0.388	0.402	2.227

Note: Raw lignocellulose biomass contains 20% (w/w) of water. Wet lignin residue contains 35% (w/w) of water. The other components include sulfuric acid, calcium hydroxide, nutrients and so on.

Table 3
Phosphorous and potassium content in lignin combustion ash.

	Combustion ash generation		Phosphorous in combustion ash	Equivalent P_2O_5	Potassium in combustion ash	Equivalent K_2O
	(kg/metric ton of dry feedstock)	(kg/metric ton of dry lignin residue)	(w/w, %)	(kg/metric ton of dry ash)	(w/w, %)	(kg/metric ton of dry ash)
Corn stover	54	141	1.9%	44	1.9%	23
Wheat straw	60	169	1.6%	37	3.1%	37
Rice straw	87	203	0.9%	21	2.6%	31
Bagasse	51	129	2.2%	50	4.3%	52
Poplar sawdust	58	149	1.3%	30	1.8%	22

biodetoxification (DryPB) processing, and the phosphorus and potassium recycled was maximized by reduction of wastewater generation in orders of magnitude (8.65–10.25 metric tons of wastewater generation per metric ton of fuel ethanol).

3.2. Replacing chemical phosphate and potassium fertilizer use by lignin combustion ash supplementation

The outputs of corn, wheat and rice yield per unit area in China in 2015 were estimated to be 5893 kg/ha, 5393 kg/ha and 6891 kg/ha, respectively (the statistics of the National Statistics of China, http://www.stats.gov.cn/). The estimated output of corn stover, wheat straw and rice straw were 7661 kg/ha, 5123 kg/ha and 8614 kg/ha, respectively, based on the general ratios of straw to grain of 1.25, 0.95 and 1.30 for corn, wheat and rice, respectively [25] (Table 4). According to the calculation in Table 3, 7661 kg of corn stover, 5123 kg of wheat straw, and 8614 kg of rice straw contained 18 kg, 11 kg, 15 kg of P_2O_5 and 9 kg, 11 kg, 23 kg of K_2O, respectively, used for the return to the farmland after biorefining of crop straws for cellulosic ethanol production. Furthermore, the phosphorus and potassium recycling was practically doubled in the agricultural regions with crop rotation system, such as the wheat-maize rotation (wheat in spring and maize in fall) in North and Northwest China, the wheat-rice rotation (wheat in spring and rice in summer/fall) in middle and lower Yangtze River China, and the double rice cultivation (in spring and fall) in middle and lower Yangtze River and Southeast China [26]. In these regions, the phosphorus and potassium supplementation from lignin combustion ash could reach to equivalent fertilizer of 26–30 kg/ha of P_2O_5 and 20–46 kg/ha of K_2O.

Table 4
Supplementation potential of phosphorus and potassium nutrients by lignin combustion ash.

	Crop straw production (kg/(ha year))	Combustion ash generation (kg/(ha year))	P_2O_5 output (kg/(ha year))	K_2O output (kg/(ha year))
(a) Potential of P_2O_5 and K_2O production from biomass				
Corn stover	7661	414	18	9
Wheat straw	5123	307	11	11
Rice straw	8614	749	15	23
(b) Supplementation from combustion ash based on cropping system				
Wheat-maize rotation (corn stover and wheat straw)			29	20
Wheat-rice rotation (wheat straw and rice straw)			26	34
Double cropping rice (double rice straws)			30	46

The usage of P_2O_5 and K_2O per hectare farmland in China in 2014 was 156 kg and 66 kg, respectively, approximately three to five folds greater than that of the world average (33.2 kg and 20.4 kg per hectare, respectively) [27]. The 26–30 kg/ha of P_2O_5 and 20–46 kg/ha of K_2O from the lignin combustion ash covered 78–90% of phosphate fertilizer and 100% of potash fertilizer (with surplus) of the world average of the need of farmland nutrients by dry biorefinery process. Even for the need of farmland in China with the heavily overused phosphate and potash fertilizers, the lignin combustion replaced 17–19% of phosphate fertilizer and 30–70% of potash fertilizer. The utilization of lignin combustion ash significantly reduces the application amount of chemical phosphate and potash fertilizer.

3.3. Reduction of cellulosic ethanol cost by lignin combustion ash supplementation

The techno-economic analysis on cellulosic ethanol production from corn stover, wheat straw and rice straw were performed according to the NREL model and our modification on the DryPB process [8,15]. The production cost of cellulosic ethanol was indicated by the term of minimum ethanol selling price (MESP, $/gal). The MESP from corn stover, wheat straw and rice straw were $1.79, $1.75 and $2.26 per gal of fuel ethanol, respectively (Table 5). The cost contribution detail of cellulosic ethanol from corn stover for each operation was shown in Fig. A.2. In the analysis, the lignin combustion ash after electricity generation was treated as solid waste [8]. If the lignin combustion ash is used as the phosphorus and potassium supplements to the farmland, the reduced use of chemical fertilizer could be considered as the credit added to the cellulosic ethanol product. Chemical phosphate and potash fertilizers are mainly provided in the forms of ammonium dihydrogen phosphorus and potassium chloride. The value of lignin combustion ash was calculated based on the phosphorus and potassium contents and the actually selling price of chemical phosphate and potash fertilizers in market [28]. The calculation process was shown in footnote of Table 5. The prices of the lignin combustion ash from corn stover, wheat straw and rice straw were $59.35, $78.61 and $60.87 per metric ton,

Table 5
MESP reduction by lignin combustion ash supplementation.

Feedstocks	Ash selling price (\$/metric ton)	Original MESP (\$/gal)	Reduced MESP (cents/gal)	Reduction ratio (%)
Corn stover	59.35	1.79	3.78	↓2.1%
Wheat straw	78.61	1.75	5.42	↓3.1%
Rice straw	60.87	2.26	7.36	↓3.3%

The price of potash fertilizer containing 62% of potassium chloride refers to the market price (\$0.371/kg), and the equivalent potassium price is calculated by \$0.371/62% × 74.5/39 = \$1.143/kg. The price of nitrogen fertilizer containing 46.3% of urea refers to the market price (\$0.445/kg), and the equivalent nitrogen price is calculated by 0.445/46.3% = \$0.961/kg. The price of phosphate fertilizer containing 55% of ammonium dihydrogen phosphate refers to the market price (\$0.355/kg), and the equivalent phosphorus price is calculated by (0.355/55%-0.961 × 14/115) × 115/31 = \$1.959/kg after deducting the nitrogen fertilizer among ammonium dihydrogen phosphate. Therefore, the selling price of the corn stover combustion ash is calculated by 1.143 × 44 × 31 × 2/142 + 1.959 × 23 × 2/94 = \$59.35 per metric ton. Similarly, the prices of combustion ash from wheat straw and rice straw are calculated as \$60.87/metric ton and \$78.61/metric ton, respectively. The numbers of 74.5, 39, 115, 14, 31, 142 and 94 are the molar mass of KCl, K, $NH_4H_2PO_4$, N, P, P_2O_5 and K_2O, respectively. The prices of potassium chloride, urea and ammonium dihydrogen phosphate quoted from Alibaba (www.1688.com).

respectively. The reduction on MESP was 3.78, 5.42, 7.36 cents/gal ethanol, equivalent to 2.1–3.3% reduction of the total MESP values.

The potential of phosphorus and potassium recycling from lignin combustion ash was improved greatly by dry biorefining of lignocellulose. Actually the combustion ash from corn stover, wheat straw and rice straw contents more than half insoluble sold composition, including 49.95–74.94% of SiO_2 and 3.75–14.73% of CaO [29]. The composition of lignin combustion ash from cellulosic ethanol production is basically same with that of combustion ash from crop straws, with about half is SiO_2 from soil [26,29]. The combustion ash is used as the fertilizer to farm land in the same way to the straw combustion ash in the conventional agricultural operation. However, calcium hydroxide was added into pretreated acid biomass to adjust the pH before biodetoxification operation. Insoluble calcium sulfate was generated and finally stayed in the lignin combustion ash. The existence of calcium sulfate may limit the directly application of lignin combustion ash on the farmland. The method of water extracting has been widely applied to recovery potassium from plant ash, and the recovery rate could be reached to 95% [30]. In this study, the assay of elemental content was done in aqueous solution of lignin combustion ash. Thus the solid calcium sulfate can be removed by water extraction according to the solubility. The phosphate and potash fertilizers are reserved for supplemented into farmland at the same time [30]. The solid residues can be used as paving or building materials [31]. Besides, the acid neutralization should be replaced with ammonium hydroxide from lime in future experiments and industrial applications. Although the price of ammonia is higher than that of calcium hydroxide, residual ammonium can be used as nutrient for microbial growth in the subsequent bioconversion process, and the produced lignin combustion ash can be directly applied into soil [32].

4. Conclusions

The dry acid pretreatment and biodetoxification (DryPB) biorefining processing maximized recycling of water-soluble phosphorus and potassium with extreme low wastewater generation. The lignin combustion ash obtained from biorefining and power generation of lignocellulose can be applied as compound fertilizer to reduce the world average usage of chemical phosphate and potash fertilizers by 78–90% and 100% (with surplus), respectively. Even for the need of farmland in China with the heavily overused phosphate and potash fertilizers, the lignin combustion replaced 17–19% of phosphate fertilizer and 30–70% of potash fertilizer. And the MESP can be also decreased by 2.1–3.3% by the selling of lignin combustion ash. The utilization of lignin combustion ash can significantly reduce the usage of chemical fertilizers, and improve the over economy of biorefining process.

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Appendix A. Supplementary data

Supplementary material related to this article can be found, in the online version, at doi:https://doi.org/10.1016/j.bej.2019.01.011.

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