Revalorization of Sunflower Stalk Pitch As Feedstock for the Coproduction of Pectin and Glucose Using a Two-Step Dilute Acid Pretreatment Process

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Research

Keywords: Sunflower stalk pith, Two-step acid treatment, Pectin, Glucose, Enzymatic hydrolysis

Posted Date: May 19th, 2021

DOI: https://doi.org/10.21203/rs.3.rs-514113/v1

Abstract

Background: Sunflower stalk pith, residue from the processing of sunflower, is rich in pectin and cellulose, thereby acting as an economic raw material for the acquisition of these compounds. In order to increase the commercial value of sunflower processing industry, a two-step sequential dilute sulfuric acid treatment combined with subsequent enzymatic hydrolysis was conducted on spent sunflower stalk pith to obtain the value-added products, pectin and glucose.

Results: In this study, pectin was firstly extracted with a mild condition to avoid pectin degradation, which conducted at 95℃ with a pH of 2.0 for 2 h, and approximately 0.12 g/g of pectin could be recovered. Then the remaining solids followed by extracted pectin were subjected to the reinforced treatment process with 0.75% H_2SO_4 at 150 ^oC for 30 min to further improving enzymatic hydrolysis efficiency. Moreover, a fed-batch enzymatic hydrolysis was successfully performed with a solid 16% content, the glucose titer reached 103.1 g/L with a yield of 83.6 %.

Conclusion: Finally, approximately 140 g pectin and 260 g glucose were produced from 1 kg of raw sunflower stalk pith using the integrated biorefinery process. This work put forward a two-step dilute acid pretreatment combined with enzymatic hydrolysis method to produce pectin and glucose from sunflower spent waste.

Background

The food processing industry generates abundant wastes, which as renewable precursor sources, are attracting increasing amounts of attention for their implementation in the development of production of green materials, fuels, and chemicals [1]. These environmentally friendly resources are gaining traction as a complementary energy feedstock to avoid competition with the growth of crops for human food or fuels [2]. Sunflower is planted on more than 26 million hectares worldwide and is the third largest source in worldwide vegetable oil, which generated abundant lignocellulosic residues, including sunflower heads, leaves and stalks; in most cases they are poorly utilized and even burned directly in the fields, which cause a waste of biomass resources and an increase in environmental pollution [3, 4].

Sunflower stalk pith (SSP), as the primary byproducts of sunflower processing industry, are composed of a high content of pectin (15–20%) and cellulose (35–45%) and a low content of hemicellulose (5–10%) and lignin (3–5%) [5, 6]. Pectin is a structural hetero-polysaccharide contained in the primary cell walls of terrestrial plants, and it has been widely used in the food and pharmaceutical industries as a commercial hydrocolloid [7, 8]. The extraction of high value-added pectin products from SSP can potentially increase the commercial value of sunflower processing industry [7]. In general, most commercial pectin is recovered using acidic hot water with mineral acids, such as HCl and H_2SO_4, at a mild temperature range of 60–90 ^oC and a low pH [9, 10]. After the pectin has been extracted, a large amount of glucan, which is a polymer composed of repeating units of glucose, is still retained in the extracted solids. Subsequently,

glucan can be hydrolyzed enzymatically to glucose, which is easily transformed to various high value-added chemicals [2, 11, 12].

However, the processing of mildly acidic extraction of pectin cannot render cellulose accessible by subsequent enzymatic activities, it is also essential to develop an integrative and efficient process to disrupt the lignocellulosic matrix of biomass and render cellulose more accessible to further enzymatic attack [13]. Dilute acid hydrolysis is the most frequently studied process for agriculture biomass, and it has been regarded as an suitable and the most feasible technology for bioethanol production on an industrial scale [14, 15]; Taken together, a two-step biorefinery concept was introduced via multiple product formation by (i) a slightly acidic extraction process using dilute H_2SO_4 as a solvent to break the SPP cell walls to release pectin by offering disruptive shear forces; and (ii) a reinforced acidic treatment process for pectin-extracted solids using dilute H_2SO_4, which can boost the efficiency of saccharification.

Materials And Methods

Materials

The raw and dry SSP was ground to a particle size of 60-100 meshes. The chemical composition of SSP by percentage of weight was: 32.25% cellulose, 10.36% hemicellulose, 2.87% lignin, and 19.31% ash, and composition content all were based on an oven dry weight basis.

Slightly dilute sulfuric acid pre-hydrolysis for pectin extraction

First, the SSP powder was mixed with water at a solid-to-liquid ratio of 1:25 (w/v) in a beaker while magnetically stirred. H_2SO_4 was simultaneously added to adjust the pH to 2.0. The extraction assays were then performed in a 95 ^oC water bath for 0.5-4.0 h while stirred with an agitator at 160 rpm. After the pectin was extracted, the solid and liquid fractions were separated by vacuum filtration, the liquid fraction was used to collect pectin, and the solid fraction was air-dried and stored for further treatment. The liquid fraction was added with the same volume of absolute ethanol, and the pH was adjusted to 3.5 with KOH. This is the pH value at which pectin is minimally soluble. The pectin was precipitated after 12 hours of sedimentation and then harvested by centrifugation at 6000 × *g* for 15 minutes; the precipitated pectin was also washed with 70% ethanol, centrifuged again (6000 × g for 15 min at 4 ^oC), and dried at room temperature for 24 h [18].

Reinforced dilute sulfuric acid pre-treatment

The pectin-extracted SSP (PE-SSP), as solid fraction, was firstly mixed with 0.75 % (w/w) H_2SO_4 (solid/liquid: 1:10 [w/v]) in a stainless steel vessel reactor. Then the sealed stainless reactor was immersed in an glycerol bath and performed at 150 °C for 30 minutes [33]. Once the pretreatment finished, the stainless steel vessel reactor was cooled to the room temperature by immersing in cold water bath. Finally, the solid after reinforced pretreatment of PE-SSP (RP-PE-SSP) and liquid fractions were separately collected by vacuum filtration.

Enzymatic hydrolysis assays

The enzymatic hydrolysis assays were all performed in a stirred reactor with a 0.5 L round-bottom flask (Proculture® glass spinner flask, Corning®, Corning, NY, USA) warmed by a 50^{o}C hot plate and stirred for 72 h at 150 rpm using a two blade propeller. Three different substrates (solid fractions of raw SSP, PE-SSP and RP-PE-SSP) were subjected with 4% solids dosage. Four batch enzymatic hydrolysis assays with different solids loading (4, 8, 12 and 16%) and a fed batch enzymatic hydrolysis assay of 4%-4%-4%-4% (every 12 h) solids dosage (total load 16%) were conducted under the same conditions to perform the enzymatic hydrolysis at high RP-PE-SSP solids loading [31]. All the hydrolysis experiments were performed with cellulase (C2730, Celluclast® 1.5 L, Novozymes, Sigma Co., Shanghai, China) 20 FPIU/g glucan and 0.05 mol/L citrate buffer for maintaining pH at 4.8. In addition, the solid residue was rinsed off with water until attained neutral pH and then subjected to enzymatic hydrolysis process.

Analytical methods

The contents of the raw materials and pretreatment and enzymatic residues were analyzed according to standard two-step acid hydrolysis method, which provided by the U.S. National Renewable Energy Laboratory (NREL) [34]. Glucose from the enzymatic hydrolysate and galacturonic acid were determined by high performance liquid chromatography (Agilent1260, USA) equipped with an Aminex Bio-Rad HPX-87H column. The weight average molecular weight (*Mw*) of the extracted pectin were analyzed by gel permeation chromatography (GPC) (1260 Infinity, Agilent Technologies.) equipped with an Aminex Bio-Rad 42A column and the molecular weight (Mw) was estimated using a calibration curve of standard dextrans.

Fourier transform infrared (FTIR) spectroscopy (Tensor 27-IR, Bruker, Billerica, MA, USA) was performed on the extracted pectin and treated and untreated SPP solids. Microscopic structure of the non-pretreated and pretreated SSP were observed by scanning electron microscope (SEM) (FEI Quanta 400, Hitachi, Tokyo, Japan) [35]. The specific surface area, pore volume and pore size of the non-pretreated and pretreated SSP were determined using the Brunauer Emmett Teller (BET) based on nitrogen gas adsorption at 77 K [28]. The crystal size of cellulose and crystallinity index (CrI) of the non-pretreated and pretreated SSP were analyzed using X-Ray diffraction (XRD) [36].

The yields of pectin and glucose enzymatic hydrolysis were calculated as follows:

$$\textbf{Pectin yield (\%)}=\frac{\text{Extracted pectin (g)}}{\text{Raw material (g)}}\times 100\%$$

$$\textbf{Enzymatic hydrolysis yield (\%)}=\frac{\text{Glucose in enzymatic hydrolysate (g)}}{\text{Glucan in raw material (g)}}\times 100\%$$

Results And Discussion

Slight acid pre-hydrolysis for pectin extraction

H_2SO_4, as the most commonly used inorganic acid, was used for pectin extraction owing to its low cost and ability to highly efficiently extract pectin [16]. In this study, pectin is extracted by sulfuric acid at pH 2.0 with conventional heating techniques (95 °C) for 0.5-4.0 hours [17, 18]; the pectin solids was then collected by ethanol precipitation and washing process. The results showed that pectin with a wide range of yield of 6.5-14.8% could be obtained from the dry powder of SSP. Figure 1 indicates that the yield of pectin increased linearly with the increase in duration of extraction over the range of 0.5-2.0 h. However, the yield of pectin did not increase when it was extracted for more than 2 h, and it even decreased when the duration of extraction was excessive. A similar phenomenon was obtained by Yapo et al. (2007) that the overlong extraction time for pectin from sugar beet resulted in large molecules were almost completely degraded into smaller sized ones [19]. It was deduced that the pectin obtained in the extraction medium had been destroyed and disintegrated [20]. Overall, the extraction time was fixed at 2 h for the pectin extraction experiments to minimize the cost.

Since the presence of high content galacturonic acids, therefore, pectic materials all have a characteristic fingerprint region in FTIR spectroscopy [21], the distinctive peaks shown for galacturonic acids in Fig. 2 are at 1150, 1100 and 1020 cm^{-1}, which confirmed that its identities was pectin [5, 16]. Moreover, the HPLC analysis showed that the galacturonic acid content was approximately 67% from the SSP, indicating that the extracted solids were pectic materials. The average molecular weight calculated from the GPC analysis was 33.2 kDa. Although the average *Mw* of SSP-pectin was lower than that the values of commercial pectin from apple or citrus, it was still consistent with previous studies that show that the average *Mw* of pectic materials from various sources, which typically in the range of 10-100 kDa, and can be used as value-added food additives [22].

Reinforced acid pretreatment for improving the efficiency of enzymatic hydrolysis

After the pectin had been extracted with H_2SO_4 at pH 2.0, approximately 95.8% of the glucan was retained in PE-SSP residues, and the relative content increased from 32.3% to 51.5%. The retained glucan in pretreated solids can be enzymatically hydrolyzed into glucose; subsequently, glucose is easily bioconverted into ethanol or other biochemicals. It is well known that pectin is the major component of the primary cell walls and the main of pectin is hydration and adhesion of wall cell. Thus, the presence of pectin can influence the porosity of cell wall and morphogenesis of plant. To a certain extent, pectin, as a physical barrier, restrict the access of enzymes to the cellulosic part of cell wall [23], [24]. Namely, pectin can affect the accessibility of cellulases. Thus, to verify that the removal of pectin improves the saccharification of cellulose, the raw SSP and solids after pectin-extracted SSP (PE-SSP) were all offered to enzymatic hydrolysis process that 4% (w/v) of the solids were loaded.

It can be observed that in Fig. 3, the glucose yield from SSP was 44.2% at 72 h when 20 FPU/g of cellulose was loaded, indicating that the raw SSP was poorly digested by the enzymes. Correspondingly, a glucose yield of 68.1% was obtained by enzymatic hydrolysis of the 4% PE-SSP solids, indicating that the slight acid pretreatment to remove the pectin with H_2SO_4 could improve the efficiency of saccharification. While the pectin-extracted strategy improved the yield of enzymatic hydrolysis, the

results have not been entirely satisfactory for generating the maximum quantity of profit-generating products.

The crystallinity of cellulose is known to significantly affect the digestibility of cellulases. The XRD analysis (Fig. 4) showed that the characteristic peak of the SSP and the PE-SSP were at 2θ=22.1°, which was cellulose I. The CrI of the PE-SSP solids sample (46.3%) increased merely 10% compared with that of the raw SSP material (36.7%). The results suggested that the PE-SSP required additional pretreatment to increase the CrI and improve the enzymatic accessibility for cellulose. Dilute acid pretreatment is the most frequently studied process for agricultural biomass and has been considered to be a suitable technology for bioethanol production at an industrial scale [13]. Thus, a second step, aimed at improving the efficiency of enzymatic hydrolysis, was performed with 0.75% (w/v) H_2SO_4 at 150 ℃ for 30 min [25].

An analysis of the reinforced pretreated PE-SSP (RP-PE-SSP) showed that the relative content of glucan increased significantly from 51.5 % to 71.1 % with a yield of 94.1%, and the xylan content decreased from 7.2% to 3.1%. Moreover, the peak of step-2 pretreatment was sharper, and the CrI increased substantially to 62.4%. In addition, the physical structure of the SSP raw material and RP-PE-SSP residues were studied using SEM. As shown in Fig. 5a, the surface morphologies of SSP were smooth and highly ordered. Observation of Fig. 5b showed that the surface morphologies of PE-SSP changed slightly, and only a small part of the cellulose was exposed, while RP-PE-SSP (Fig. 5c) depicted a highly unstructured rough surface with a substantial amount of cracks.

Large specific surface area and total pore volume can offer high adsorption capacity, therefore, the specific surface area and pore volume of lignocellulosic materials are considered to be key factors impacting the enzymatic hydrolysis. The specific surface area of raw SSP, PE-SSP and RP-PE-SSP were 1.529, 2.803, and 3.591 m^2/g, respectively, and the corresponding total pore volume was 0.0026, 0.0051, and 0.0064 cm^3/g, respectively. The result depicted that the specific surface area and total pore volume significantly increased by enhancement of pretreatment intensity. Such an increase can be attributed to the partial breakdown of the raw material microstructure, resulting in the formation of more cracks and larger pore size, as evident from the micrographs of SEM [26]. Enzymatic hydrolysis is known to be strongly affected by porosity, including specific surface area and total pore volume, since the cellulase can directly contact the cellulose structure through the pores. Overall, incremented specific surface area and pore volume percentage allow better access of the cellulase to inner portion of lignocellulosic solids. Visual observations show that reinforced pretreatment efficiently dissected the physical structure after reinforced pretreatment with 0.75% H_2SO_4 at 150 °C for 0.5 h, which resulted in an enzymatic hydrolysis yield of up to 92.3% at 72 h with a loading dosage of 4% RP-PE-SSP solids. Obviously, reinforced pretreated can enhance interfacial interactions between the solids and cellulases, resulting in improvement of saccharification efficiency [27, 28].

High-solids enzymatic hydrolysis with batch and fed-batch modes

The fermentable sugars should be at levels as high as possible in the industrial scale utilization of lignocellulosic materials due to high final sugars is benefit to improve utilization rate of equipment, and low the consumption of water and energy. Using bioethanol generation as an example, obtaining fermentation broths of at least 4% (w/v) of ethanol is essential for economical large-scale production owing to the costs of ethanol purification, which dramatically increase when the ethanol titer is lower [29]. Thus, in this framework, at least more than 80 g/L of total reducing sugars obtained from the biomass is required. Correspondingly, enzymatic hydrolysis must be conducted with a pretreated solid loading of more than 10~20% (w/v) on the basis of the cellulose proportion. Moreover, high-solids enzymatic hydrolysis is preferred for economic reasons for biorefinery processes on an industrial scale. Thus, enzymatic hydrolysis assays with batch mode were first performed with 4, 8, 12, and 16% RP-PE-SSP solids loading to produce a high titer of glucose.

Figure 6 shows that a glucose titer of 28.9, 51.6, 63.5, and 54.4 g/L final glucose accumulated with a solid loading of 4, 8, 12, and 16%, respectively. It was apparent that 4% solid in the system could be liquefied within 8 h. However, the mixing became fouled with a batch operation of more than 8% (w/v) solids loading, and in the case of 12% (w/v) RP-PE-SSP solids loading, the reaction medium was not mixed at the beginning of the experiments, even with a significant increase in the rate of agitation. In addition, the system with high solids loading had difficulty becoming liquefied even when treated for more than 24 h, which speculate that this due to the reduction of crystallinity and the limited catalytic sites for cellulases [30, 31]. It is apparent that a high consistency of hydrolysis may result in difficulties mass transfer owing to high viscosity as a result of high solids loading and the lack of free water in the enzymatic system. Thus, glucose yields from the loading of 8, 12, and 16% solids declined linearly. The enzymatic hydrolysis yield from loading of 16% solids was only 43.4%.

Although increasing the solids loading was the simplest and most direct way to enable a high concentration of sugars, this technique resulted in high viscosity, poor mixing, heat transfer and enzyme distribution problems, which reduced the efficiency of enzymatic hydrolysis [30, 32]. The fed-batch process is regarded as an effective way to minimize these negative effects. Thus, we conducted a fed-batch enzymatic hydrolysis of RP-PE-SSP material to produce glucose. A fed-batch operation of 4%-4%-4%-4% (every 12 h) solids dosage was conducted for 72 h for enzymatic hydrolysis. When the enzymatic hydrolysis was successfully performed with a solid content as high as 16% (w/v), the glucose titer obtained and yield reached 103.1 g/L and 83.6 %, respectively. Overall, the fed-batch strategy enables an easy dynamical load and the production of a solution with a high glucose titer. The mass balance of the whole process for releasing pectin and glucose showed that a total of 120 g pectin and 260 g glucose were recovered from 1,000 g oven-dried raw material using a two-step pretreatment technology and the fed-batch enzymatic hydrolysis mode. The entirety of the results clearly suggests that the two-step acid-treatment with H_2SO_4 is a profitable option for the further exploitation of sunflower residue.

Conclusions

The extraction of pectin and glucose from SSP using a two-step dilute acid pretreatment combined with enzymatic hydrolysis was presented. As a first step, slightly acidic hydrolysis is an efficient technology for extracting pectin. The results indicated that the tight and complex structure of the SSP was deconstructed by the reinforced pretreatment, which benefitted the subsequent enzymatic hydrolysis of the residue from the first step. Overall, this study provides a two-step pretreatment strategy that achieves economical and efficient utilization for SSP.

Abbreviations

SSP: sunflower stalk pith; PE-SSP: pectin-extracted sunflower stalk pith; RP-PE-SSP: reinforced pretreated pectin-extracted sunflower stalk pith; Mw: molecular weight; BET: brunauer emmett teller; FTIR: fourier transform infrared; CrI: crystallinity index; SEM: scanning electron microscope; XRD: X-Ray diffraction; HPLC: high performance liquid chromatography; GPC: gel permeation chromatography.

Declarations

Ethics approval and consent to participate

Not applicable.

Consent for publication

Not applicable.

Availability of data and materials

All data generated or analyzed during this study are included in this published article.

Competing interests

The authors declare that they have no competing interests.

Funding

This study was funded by the National Key R&D Program of China (2017YFD0601001), the National Natural Science Foundation of China (31901270), and the Scientific Research Start-up Funds of Nanjing Forestry University, China (163030127).

Authors' contributions

QZ and XM performed the research, data analysis and prepared manuscript. XZ and YX developed the idea for the study, and revised the manuscript. All authors read and approved the final manuscript.

Acknowledgements

We sincerely thank the support of the Advanced Analysis and Testing Center of Nanjing Forestry University.

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Figures

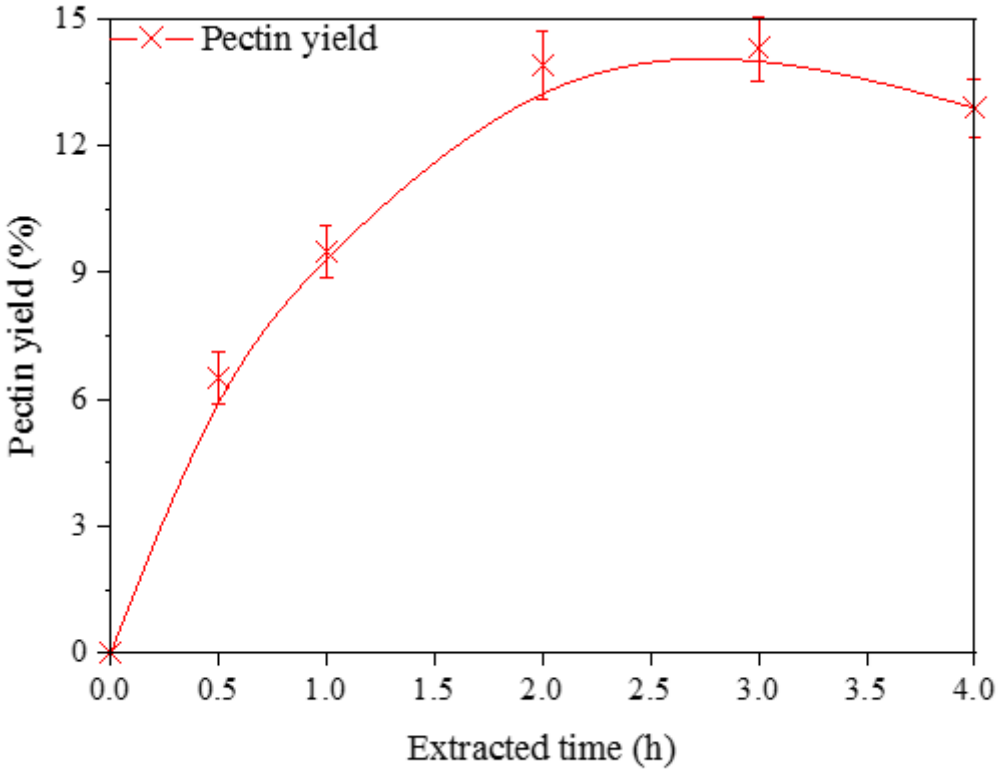

Figure 1

Yield of pectin from SSP with different durations of extraction

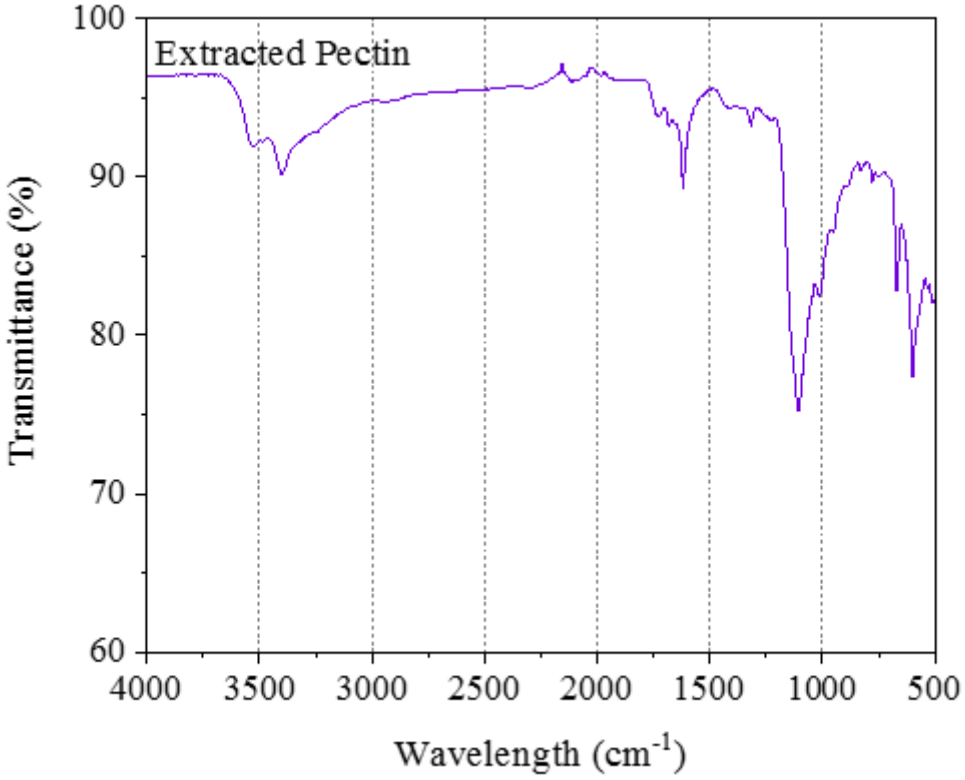

Figure 2

FTIR spectra of sunflower pith pectin (obtained at 95 oC, 120 min, pH 2.0)

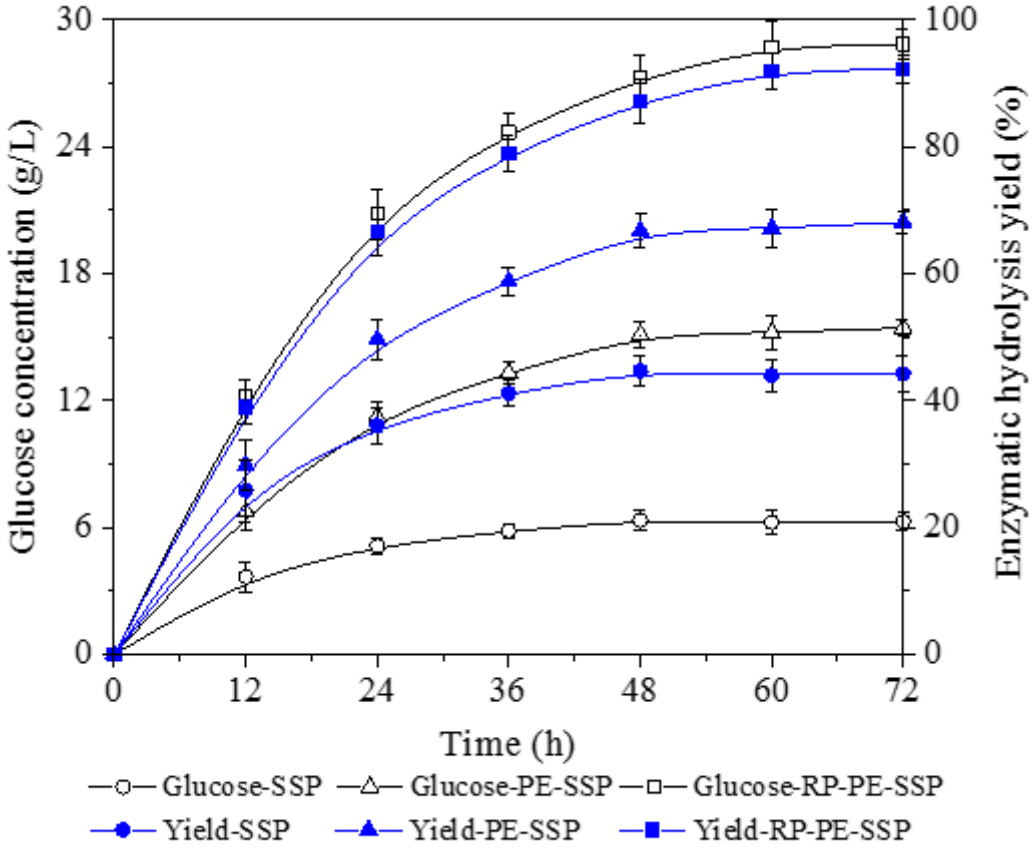

Figure 3

Time course of enzymatic hydrolysis with 4% solids dosage of SSP, PE-SSP and RP-PE-SSP

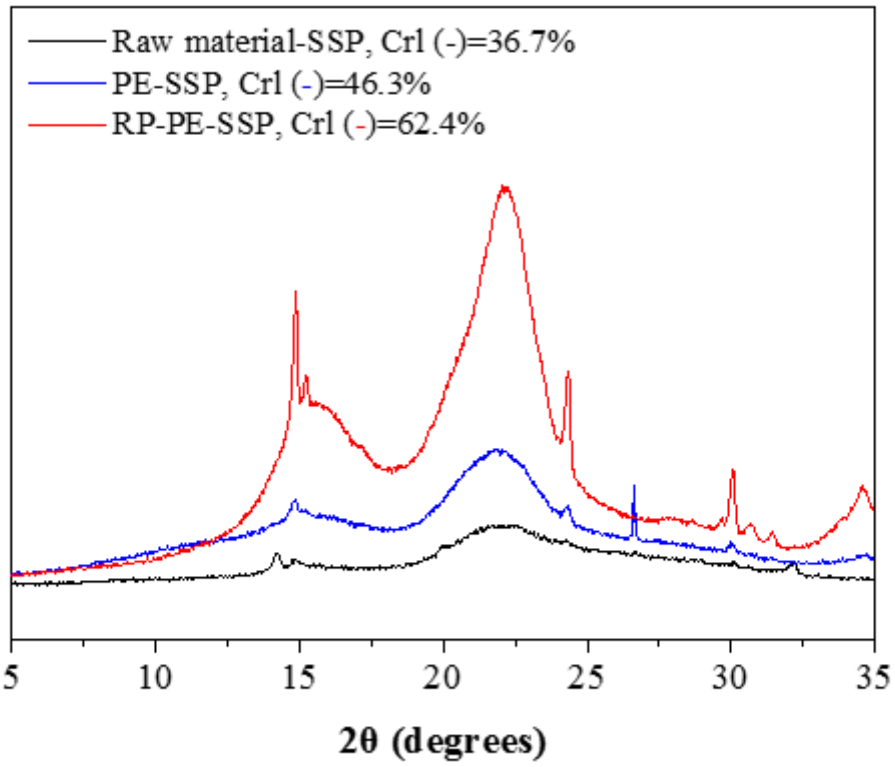

Figure 4

XRD patterns obtained from the SSP, PE-SSP and RP-PE-SSP

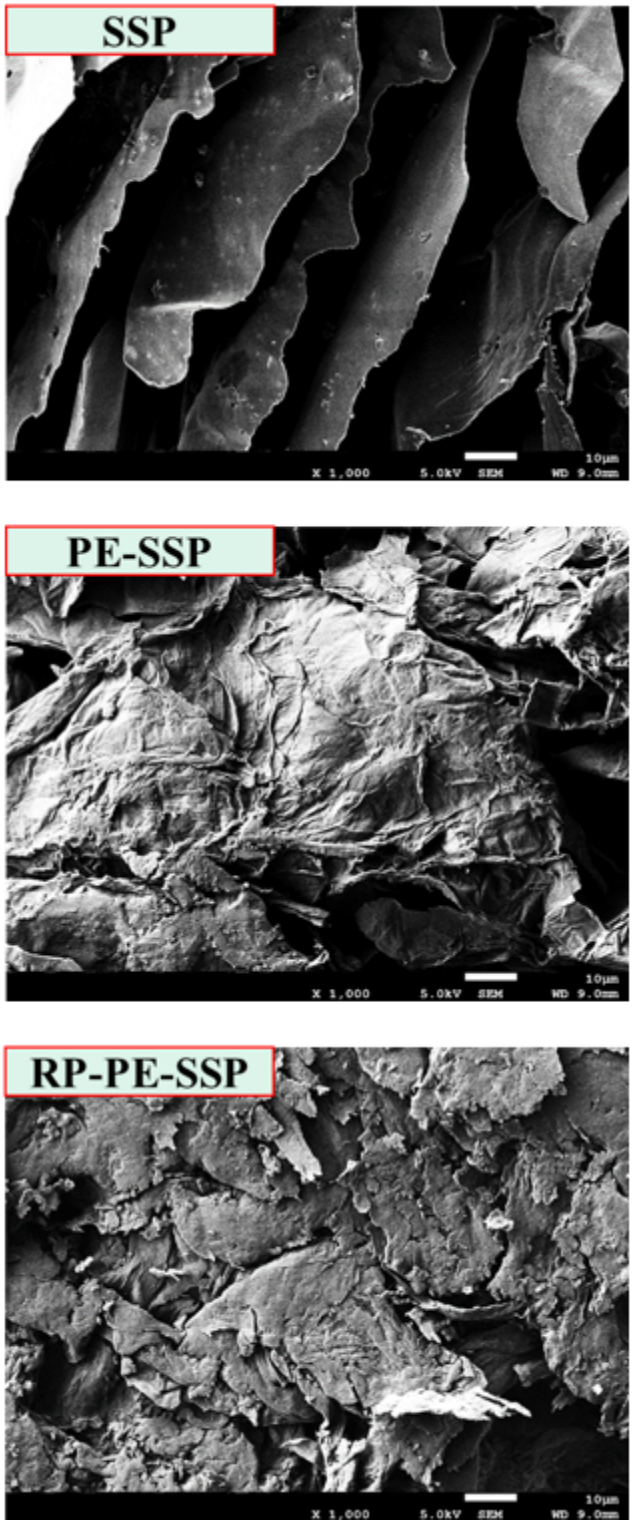

Figure 5

Scanning electron micrographs of SSP, PE-SSP, and RP-PE-SSP materials.

Fig. 6.

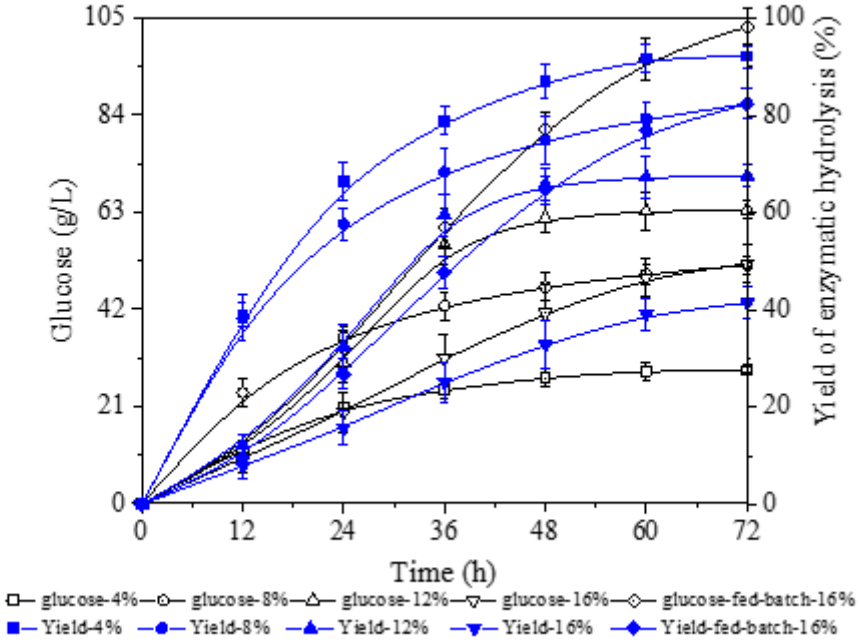

Figure 6

Time course of batch enzymatic variety at different solid loadings (4–16%) and a fed-batch enzymatic hydrolysis with total 16% solids dosage of RP-PE-SSP

Supplementary Files

This is a list of supplementary files associated with this preprint. Click to download.

- 4Graphicabstract.docx