

A novel surfactant-assisted dilute phosphoric acid plus steam explosion pretreatment of poplar wood for fermentable sugar production

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Abstract

Pretreatment is an indispensable process in lignocellulosic bioethanol production. In this work, a surfactant agent JFC was introduced into the dilute phosphoric acid plus steam explosion pretreatment scheme for fermentable sugar production using poplar as substrate. Four crucial factors (phosphoric acid concentration, surfactant concentration, pressure, and residence time) affecting the pretreatment efficiency were optimized using the single factor tests. The optimal parameters obtained were as follows: 1:2.5 solid/liquid rate, 2 h pre-soaking time, 1.5 % (v/v) JFC-M + 2.0 wt% phosphoric acid, 2.0 MPa pressure, and 120 s residence time, resulting in a maximum cellulose recovery rate of 86.33 % and enzymatic saccharification rate of 84.62 %, which was 38.97 % higher than that of control. The morphological and structural characteristics of samples before and after pretreatment, were characterized by the scanning electron microscopy (SEM), X-ray diffraction (XRD), and Fourier transform infrared (FTIR) method. The addition of JFC-M was of a notable influence in overcoming biomass recalcitrance and boosting cellulose digestion, showing great application potentials in biomass conversion process.

1. Introduction

Lignocellulosic biomass (LCB) is the most abundant and renewable resource on the planet, which can act a low-cost and promising feedstock for production biofuels, biomaterials, and biochemicals, etc., attracting worldwide interests and attentions (Alonso et al. 2017). However, owing to the high biomass recalcitrance, a suitable pretreatment process is required to remove lignin and hemicellulose, depolymerize of cellulose of LCB, and release fermentable sugars for further bioconversion such as bioethanol production (Qi et al. 2018; Tu and Hallett 2019).

To date, several LCB pretreatment techniques have been developed, which are mainly categorized into physical, chemical, physicochemical, biological techniques, and some cocktail of these strategies (Haldar and Purkait 2020). Among them, steam explosion (SE) method is of advantages such as cost-effective and environmentally -friendly supermolecular deconstruction, which is widely applied in LCB especially hardwood and agricultural residual pretreatment (Jacquet et al. 2015). Meanwhile, to improve the efficiency and reduce the severity of SE (e.g., lowering the temperature and pressure, shortening the residence time), some chemical additives, mainly mineral acids or alkalis (such as SO_2 , H_2SO_4 , H_3PO_4 , and NH_4), are introduced into SE process (Zhang et al. 2020).

Dilute phosphoric acid plus steam explosion (DPASE) is a promising alternative LCB pretreatment method (Fockink et al. 2018; Geddes et al. 2011; Oliva et al. 2020), holding the advantages as following: (i) acting as an exogenous weak acid catalyst, dilute H_3PO_4 results in low sugar loss and yield of toxins simultaneous with SE (Pitarelo et al. 2016; Zeng et al. 2014); (ii) H_3PO_4 can not only serve as an additional source of nutrients for microbe, but can also be partly recovered and reused as a fertilizer (Geddes et al. 2011); and (iii) H_3PO_4 is less corrosive to the SE apparatus, which is beneficial for the equipment and efforts at scaling up (Haykiri-Acma and Yaman 2019; Koradiya et al. 2016). However,

regarding the distinct components and microstructures of multisourced LCB feedstocks (e.g., dedicated crops, agricultural residues, and short-rotation energy coppices), more researches are needed to expand the application of DPASE.

It has been reported that surfactants, particularly nonionic surfactants (Tween, Triton, and polyethylene glycol, etc.) in the pretreatment process can promote the wettability of LCB, decrease the crystallinity of cellulose, give higher cellulose conversion, improve the delignification rate, maximize the enzymatic convertibility, and enhance the ethanol yields, etc. (Cao and Aita 2013; Jørgensen et al. 2007; Nasirpour et al. 2014; Zheng et al. 2020). A fatty alcohol polyoxyethylene ether based nonionic surfactant agent JFC ($R-O(CH_2CH_2O)_5-H$, wherein $R = C_{7-9}$), holds the properties of reducing interfacial tension, enhancing the wettability of materials, improving the capillary effect inside fibers, etc., which has been introduced in textile modification (Gao et al. 2017), coal mining (Shi et al. 2019), porous geopolymers preparation (Yan et al. 2021), mineral flotation (Chen et al. 2018), etc. Considering its other advantages such as strong acid, alkali, and high temperature resistance and no bio-toxicity, the surfactant agent JFC holds potential of assisting the LCB steam explosion pretreatment.

In this work, a novel surfactant JFC-assisted dilute phosphoric acid plus steam explosion pretreatment of poplar wood craft was developed. Four crucial factors (phosphoric acid concentration, surfactant concentration, pressure, and residence time) affecting the pretreatment efficiency were optimized using the single factor tests. The morphological and structural characteristics of samples were characterized for analyzing the mechanism of pretreatment and further optimizing of the process.

2. Materials And Methods

2.1 Materials

The dried European black poplar (*Populus nigra L.*) chips were pulverized by a plant shredder to sawdust (1–2 mm diameter, 2–15 mm length) as feedstocks. The surfactant JFC-M (industrial grade) was purchased from Shandong Yousuo Chemical Technology Co., Ltd. (Zhou et al. 1995). The cellulase was purchased from Qingdao Vland Biotech INC.(Qingdao, China), holding a filter paper activity (FPA) of 191 IU/mL. The chemical agents like phosphoric acid, sulfuric acid, and calcium carbonate, etc., were analytical grade and could be used without further purification.

2.2 Experimental method

2.2.1 DPASE

The poplar sawdust was presoaked in a JFC-M and dilute H_3PO_4 solution at 1:2.5 solid/liquid rate for 2 h, and then pretreated in a SE apparatus QBS-80B (Tsing Gentle Eco-technology (Suzhou) Co., Ltd.) equipped a 400 mL cylinder. The exploded slurry was collected for further study. For detection the removal of cellulose and hemicellulose after DPASE, the exploded slurry was separated by a centrifuge at 1000 r/min and the filtrate was collected for further analysis.

2.2.2 Single factor experiment

The single factor experiment was conducted using four critical factors influencing the pretreatment efficiency such as surfactant JFC-M and H_3PO_4 concentration, pressure of explosion, and residence time.

2.2.3 Enzymatic saccharification

The exploded slurry was dried in a drying oven (Tianjin Taisite Instrument Co., Ltd.) at $60^\circ C$ for 48h and then added into 100 mL Erlenmeyer flasks containing 50 mL citric acid buffer and with 1:50 solid/liquid rate. The enzymatic saccharification was carried out with parameters as following: 30 IU/g dry exploded slurry cellulase loading, 180 rpm/min stirring speed, initial solution pH 4.8, $50^\circ C$ reaction temperature for 72 h.

The saccharification rate is calculated as follows (Kim 2011):

$$\text{Saccharification Rate (\%)} = \frac{C_0 \times V \times 0.9}{(C_1 + C_2) \times M} \times 100\%$$

Wherein, C_0 is the concentration of reducing sugar (RS) in the hydrolysate. V is the volume of hydrolysate. C_1 and C_2 are the cellulose and hemicellulose contents in the substrate after pretreatment. M is the dry mass of the substrate, and "0.9" means that 1.0 g RS is equivalent to the mass of 0.9 g cellulose and hemicellulose.

2.2.4 Composition analysis

The cellulose, hemicellulose and lignin of samples were analyzed according the national renewable energy laboratory (NREL) standard method (Sluiter et al. 2008).

The solid and cellulose recovery rate (or retention rate) is calculated as follows:

$$\text{Solid or cellulose recovery (\%)} = \frac{m_2}{m_1} \times 100\%$$

Wherein, m_1 and m_2 are the masses of poplar substrate or cellulose before and after pretreatment, respectively.

The hemicellulose removal rate is calculated as follows:

$$\text{Hemicellulose recovery (\%)} = \left(1 - \frac{m_2}{m_1}\right) \times 100\%$$

Wherein, m_1 and m_2 are the masses of hemicellulose before and after pretreatment, respectively.

The concentration of RS was determined by a high-performance liquid chromatography (HPLC, Thermofisher U3000, Thermo Fisher Scientific) with a chromatographic column Aminex HPX-87H according the NREL standard method (Sluiter et al. 2008). The analysis conditions were as follows: 5 mmol/L H₂SO₄ mobile phase, 45°C column temperature, 5 mL/min flow rate, 30°C differential refractive index temperature, and 20 µl sample volume. All tested samples were filtered by a 0.22 µm filter membrane. The standard sugar gradient solution was prepared with cellobiose, glucose, xylose, and arabinose, and the standard curve was drawn by the external standard method.

2.2.5 Structure characterization

The samples before and after pretreatment were dried in a constant-temperature drying oven at 105°C to a constant weight for further structure characterization. The SEM was carried out by a Zeiss Sigma 300 scanning electronic microscope. The cellulose crystallinity of samples were analyzed by an X-ray diffraction analyzer (X'Pert PRO, PANalytical, Netherlands), with the following parameters: 0.15406 nm wavelength, 45 kV voltage, 40 mA current, 5°–50° scanning angle 2θ range, and 5°/min scanning rate.

The crystallinity is calculated as follows (Segal et al. 1959):

$$CI (\%) = \frac{I_{002} - I_{am}}{I_{002}} \times 100\%$$

Wherein, I_{002} represents the maximum intensity of the crystalline part at 22°, and I_{am} represents the minimum intensity of the non-crystalline part at 18°.

The Fourier transform infrared (FTIR) analysis was conducted by an IRAffinity-1S spectrometer (Shimadzu Corp., Kyoto, Japan) with following parameters: 400–4000 cm⁻¹ wave number, 32 scans per second analysis speed, and 4 cm⁻¹ resolution ratio, respectively.

3. Results And Discussion

3.1 Effect of phosphoric acid on pretreatment

Generally, the content of glucose and xylose in exploded filtrate reflects the removal of cellulose and hemicellulose, respectively. As shown in Fig. 1a, the content of glucose in exploded filtrate increased when the H₃PO₄ was less than 2.0 wt%, and then decreased slightly with the increase of H₃PO₄, holding a maximum value of 3.61 g/L, which was 3.4 times that in the blank group. The content of xylose in exploded filtrate increased with the increase of H₃PO₄, holding a maximum value of 8.79 g/L at 2.5 wt% H₃PO₄, which was 2.5 times that in the blank group. The higher titter of xylose yielded compared with that of glucose could be attributed to two reasons: (i) xylan is more labile than glucan in LCB substrate (Chen et al. 2007); (ii) the exogenous H₃PO₄ catalyst presents higher capacity of solubilizing hemicellulose of LCB (Oliva et al. 2020). According to Fig. 1b, the saccharification rates of samples were positively

correlated with the H_3PO_4 concentrations, holding a maximum value of 78.52 % at 2.5 wt% H_3PO_4 , which was 27.95 % higher than that in the blank group. The removal of hemicellulose increased the internal porosity of the fiber and provided more enzyme contact sites, boosting the enzymatic saccharification of substrate (Leu and Zhu 2013). The addition of surfactant JFC-M helps to remove hemicellulose and increase the enzymatic saccharification rate. Considering the cost-effectiveness of pretreatment, 2.0 wt% H_3PO_4 was used for further study.

3.2 Effect of surfactant JFC-M on pretreatment

As shown in Fig. 2a, the content of glucose in exploded filtrate increased when the JFC-M concentration was less than 1.0 % (v/v), and then decreased slightly with the increase of JFC-M, holding a maximum value of 3.28 g/L, which was 3.3 times that in the blank group. The content of xylose in exploded filtrate increased when the JFC-M concentration was less than 1.5 % (v/v), and then decreased with the increase of JFC-M, holding a maximum value of 8.56 g/L, which was 2.5 times that in the blank group. When the JFC-M concentration was less than 2.0 % (v/v), the saccharification rate of samples increased with the increase of JFC-M, and then decreased, holding a maximum value of 80.91 % at 2.0 % (v/v) JFC-M, which was 20.02 % higher than that in the blank group (Fig. 2b). The decreased of saccharification rate with the increase of JFC-M might be caused by the negative effect of surfactant and inhibitors generated in the pretreatment process, which should be done further research. The reasons for this may be as follows (Kim 2011; Leu and Zhu 2013; Li et al. 2014): (i) the addition of surfactant JFC-M increased the surface area and porosity of poplar fiber; (ii) alleviation the adhesion of pseudo-lignin to the fiber surface after explosion, and reduced the ineffective adsorption of cellulase on the surface of lignin; (iii) improvement the stability of cellulase activity; and (iv) changes of the substrate structures to further promote enzymatic hydrolysis. Considering the cost-effectiveness of pretreatment, 1.5 % (v/v) surfactant JFC-M was used for further study.

3.3 Effect of pressure on pretreatment

The contents of glucose and xylose increased with the increase of SE pressure, holding maximum values of 3.48 g/L and 8.73 g/L, respectively (Fig. 3a). The saccharification rates of samples increased with the increase of SE pressure, holding a maximum value of 82.96 % at 2.4 MPa SE pressure, which was 22.07 % higher than that in the blank group (Fig. 3b). Pressure is correlated to temperature and has impacts on hemicellulose degradation to soluble mono- and oligosaccharides, and cleavage of lignin carbohydrate complex (Chen and Qiu 2010). The recently study revealed that with the increase of pressure, the inhibitors generated in SE process using poplar as a feedstock basically maintained at the same level (Wang et al. 2020). Considering the increase of pressure would increase the energy consumption, 2.0 MPa SE pressure was used for further study.

3.4 Effect of residence time on pretreatment

The content of glucose in exploded filtrate increased when the residence time was less than 180 s, and then decreased slightly, holding a maximum value of 3.03 g/L, which was 2.8 times that in the blank group (Fig. 4a). The content of xylose in exploded filtrate increased with residence time, holding a

maximum value of 8.70 g/L at residence time of 300 s, which was 2.5 times that in the blank group. As shown in Fig. 4b, when the residence time was less than 120 s, the saccharification rates of samples increased with the increase of JFC-M, and then decreased, holding a maximum value of 84.62 % at residence time of 120 s, which was 23.73 % higher than that in the blank group. The hydrolysis of hemicellulose is closely correlated to the residence time of SE, and a high residence time allows the complete hydrolysis of hemicellulose and decrease the solid recovery (Jacquet et al. 2015). The drops of saccharification rate, when the residence time was over 120 s, might be the reason that part of the xylose or glucose are converted into enzymolysis inhibitors, which adversely inhibits the enzymatic hydrolysis (Junhua Zhang et al. 2012). Considering the saccharification rate of poplar, 120 s residence time was selected. In summary, a maximum saccharification rate was obtained under the DPASE parameters as follows: 1:2.5 solid/liquid rate, 2 h pre-soaking time, 1.5 (v/v)% JFC-M + 2.0 wt% H₃PO₄, 2.0 MPa SE pressure, and 120 s residence time.

3.5 Components change of samples

The components of samples before and after pretreatment are shown in Table 1.

The contents of cellulose and lignin in DPASE (55.61 % and 31.05 %) and JFC-M + DPASE (64.80 % and 33.10 %) group were greater than that of the raw material (44.58 % and 28.32 %), respectively. The content of hemicellulose in the two pretreatment groups (6.05 % and 2.97 %) decreased dramatically compared with that of the raw material (12.05 %). As the assistant removal of hemicellulose and lignin using a surfactant (Qing et al. 2010), the solid recovery rate in JFC-M + DPASE (55.97 %) group was lower than the one in DPASE group (73.12 %). The hemicellulose removal rate in JFC-M + DPASE (83.50 %) group was higher than the one in DPASE group (60.47 %). Comparing the results reported in Table 2, the addition of surfactant JFC-M increased the removal of hemicellulose, leading to a relatively high cellulose recovery rate obtained. Considering the SE conditions, sugars resolved in exploded filtrate, recovery rates, and saccharification rate, the surfactant-assisted DPASE pretreatment method performs advantages over other SE method

Table 1
Compositions (%), recovery and removal rates (%) of samples*.

Component (%)	Raw	DPASE	JFC-M + DPASE
Carbohydrate	56.63	55.61	64.80
Cellulose	44.58	49.56	61.83
Hemicellulose	12.05	6.05	2.97
Lignin	28.32	31.05	33.10
Acid-soluble lignin	1.54	1.36	1.29
Acid-insoluble lignin	26.78	29.69	31.81
Solid recovery rate	/	73.12	55.97
Cellulose recovery rate	/	88.85	86.33
Hemicellulose removal rate	/	60.47	83.50
* Note: (1) the alcohol extract and ash of raw poplar feedstock is 1.68 % and 0.84 %, respectively. (2) All the samples were analyzed according the NREL standard method.			

Table 2
Compositions (%) and recovery rates (%) of samples in literature*.

SE Conditions	Composition (%)	Sugars in filtrate(%)	Recovery (%)	Reference
1–2 × 2–15 mm particle size, 1:2.5 solid/liquid rate, 2 h pre-soaking time, 1.5 (v/v)% JFC-M + 2.0 wt% H ₃ PO ₄ , 2.0 MPa pressure, 120 s residence time.	44.58 % C, 12.05 % H, 28.32 % L.	1.56–3.41 % glucose, 2.15–8.79 % Xylose.	86.33–88.85 % C, 55.97–73.12 % SR.	this work
30×30×5 mm particle size, N ₂ added in SE process, 209 ± 0.5°C, 7 min residence time.	44.1 % C, 19.4 % H, 27.8 % L.	1.35–1.39 % glucose, 7.36–8.14 % xylose.	55.1–56.2 % C, 4.9–5.6 % H, 19.5–20.8 % L, 59.5–65.0 SR.	Wang et al. (2020)
10(± 2) mm particle size, 200 or 220°C, 5 min residence time.	35.5–40.4 % C, 18.1–22.9 % H, 30.1–38.2 % L.	1.3–1.9 % glucose, 6.4–9.8 % xylose.	32.0–40.0 acid-insoluble lignin, 62.8–76.5 SR.	Martín-Davison et al. (2015)
30×15×5 mm size, 190–220°C, 7–11 min residence time.	44.3 % C, 16.2 % H, 26.9 % L.	/	62.8–76.5 SR.	Castro et al. (2014)
2.5×2.5 cm and 5.0×5.0 cm size, Alkaline-oxygen impregnated SE: 12 wt % Na ₂ CO ₃ 100–110°C, 100 psig O ₂ , 135°C, 3 min; SE: 210°C, 10 min residence time.	45.7 % C, 17.4 % H, 34.9 % L.	/	94–97 % C, 54–71 % H.	Chu et al. (2017)
* Note:(1) C, H, L, and SR represents cellulose, hemicellulose, lignin, and solid recovery, respectively. (2) All the samples were analyzed according the NREL standard method.				

4. Characterization Of Samples

4.1 SEM analysis

The morphological changes of samples were characterized by the SEM method, and the results were shown in Fig. 5. The raw poplar is of tightly packed and dense structure with the smooth surface morphology (Fig. 5a,b). In DPASE group (Fig. 5c,d), some slight damages occurred at the surface and interior of poplar fiber. The uneven spherical precipitates were formed on the surface of fiber, which is probably the result of the formation of lignin microspheres or pseudo-lignin microspheres at high temperatures (Cheng et al. 2018). In JFC-M + DPASE group (Fig. 5e,f), severe damages were occurred, performing densely wrapped and broken structures of fiber, which greatly increased the specific surface area and porosity of the feedstock and was beneficial for subsequent enzymatic saccharification. The surfactant JFC-M may have a unique mode of action in assisting of the DPASE process by the means of following. Firstly, JFC-M promotes the wetting of the surface of poplar fiber by H_3PO_4 solution and permeation into the interior of the fibers, so as to improve the acid-catalyzed deconstruction of poplar fiber. The results were in line with literature reported that the addition of surfactants quite better option for acid pretreatment (Singh et al. 2015).

4.2 XRD analysis

Crystallinity of LCB is a crucial factor that influences the enzymatic hydrolysis. The crystallinity index (Crl) of samples are determined by the XRD analyse and the results are shown in Fig. 6. The relative crystallinities of DPASE (62.09 %) and JFC-M + DPASE (63.42 %) pretreated samples were greater than that of the unpretreated poplar(53.59 %), which was consist with the results of previous studies (Singh et al. 2015). The increase of crystallinity was mainly caused by the deconstruction of crystalline area of cellulose, removal of hemicellulose and delignification (Jacquet et al. 2015). Once the crystalline areas become small and amorphous, the accessibility of enzyme to the cellulose substrate could be improved. The higher crystallinity of JFC-M + DPASE pretreated sample than that of the DPASE is in line with the results of the single factor test and changes of components. After enzymatic hydrolysis, the Crl (46.72 %) of sample decreased, which might be caused by some destroy of the crystalline area of cellulose during the enzymatic hydrolysis process, resulting in the loose and irregularly arranged area becoming into the amorphous area.

4.3 FTIR analysis

The structural modifications of samples were identified by a FTIR analysis and the results were shown in Fig. 7. The enhancements of C-H and -OH stretching vibrations ($3423\text{ cm}^{-1} - 2924\text{ cm}^{-1}$) of cellulose after pretreatment (Wang et al. 2020), indicates more loose cellulose are obtained for the subsequent enzymatic hydrolysis process, especially in the JFC-M added group, which is consistent with the results of SEM. The characteristic absorption peak of hemicellulose xylan (1740 cm^{-1}) (Bu et al. 2011), decreases after pretreatment, especially in the JFC-M + DPASE group, indicating that the addition of surfactant is more conducive to removing hemicellulose. Here, 1510 cm^{-1} is the C = C stretching vibration of the pure lignin aromatic ring in LCB (Anderson et al. 1991). The C-H and C-O stretching vibration of the syringyl benzene ring in the lignin is noted at 1120 cm^{-1} (Schwanninger et al. 2004). All samples after pretreatment show absorption at 1120 cm^{-1} , indicating the existing of syringyl lignin in

poplar wood. The stretching vibration at 898 cm^{-1} is the characteristic peak of β -glycosidic bond (Ibrahim et al. 2010), the intensity of which dropped slightly after pretreatment, indicating that a small amount of glucan is degraded during the pretreatment process. This is consistent with the glucose content in exploded filtrate after pretreatment.

5. Conclusions

In this study, a surfactant JFC-M assisted dilute phosphoric acid plus steam explosion pretreatment of poplar wood was developed. Four crucial factors such as phosphoric acid concentration, surfactant concentration, pressure, and residence time were optimized using the single factor tests. The results showed under the optimized conditions: 1:2.5 solid/liquid rate, 2 h pre-soaking time, 1.5 (v/v)% JFC-M + 2.0 wt% phosphoric acid, 2.0 MPa pressure, and 120 s residence time, a 86.33 % cellulose recovery rate and 84.62 % enzymatic saccharification rate were obtained. The surfactant JFC-M increased the removal of hemicellulose and a high cellulose recovery rate, which in turn booted the enzymatic saccharification of substrate. The morphological and structural characteristics of samples were analyzed for revealing the mechanism of pretreatment and further optimizing of the process. The novel pretreatment process shows great application potentials in biomass conversion process.

Declarations

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interest statement

There are no conflicts of interest to declare.

Data availability statement

The datasets generated during and/or analysed during the current study are available from the corresponding author on reasonable request.

References

1. Alonso DM, Hakim SH, Zhou S, Won W, Hosseinaei O, Tao J, Garcia-Negron V, Motagamwala AH, Mellmer MA, Huang K (2017) Increasing the revenue from lignocellulosic biomass: Maximizing feedstock utilization. *Sci Adv* 3(5):e1603301
2. Anderson EL, Pawlak Z, Owen NL, Feist WC (1991) Infrared studies of wood weathering. Part I: Softwoods. *Appl Spectrosc* 45(4):641–647

3. Bu L, Tang Y, Gao Y, Jian H, Jiang J (2011) Comparative characterization of milled wood lignin from furfural residues and corncob. *Chem Eng J* 175:176–184
4. Cao S, Aita GM (2013) Enzymatic hydrolysis and ethanol yields of combined surfactant and dilute ammonia treated sugarcane bagasse. *Bioresour Technol* 131:357–364
5. Castro E, Nieves IU, Mullinnix MT, Sagues WJ, Hoffman RW, Fernández-Sandoval MT, Tian Z, Rockwood DL, Tamang B, Ingram LO (2014) Optimization of dilute-phosphoric-acid steam pretreatment of *Eucalyptus benthamii* for biofuel production. *Appl Energ* 125:76–83
6. Chen C, Zhu H, Qin W, Chai L, Jia W (2018) Improving collecting performance of sodium oleate using a polyoxyethylene ether in scheelite flotation. *J Cent South Univ* 25(12):2971–2978
7. Chen H, Qiu W (2010) Key technologies for bioethanol production from lignocellulose. *Biotechnol Adv* 28(5):556–562
8. Chen Y, Sharma-Shivappa RR, Keshwani D, Chen C (2007) Potential of agricultural residues and hay for bioethanol production. *Appl Biochem Biotechnol* 142(3):276–290
9. Cheng B, Wang X, Lin Q, Zhang X, Meng L, Sun R-C, Xin F, Ren J (2018) New understandings of the relationship and initial formation mechanism for pseudo-lignin, humins, and acid-induced hydrothermal carbon. *J Agric Food Chem* 66(45):11981–11989
10. Chu Q, Chandra RP, Kim C-S, Saddler JN (2017) Alkali-oxygen impregnation prior to steam pretreating poplar wood chips enhances selective lignin modification and removal while maximizing carbohydrate recovery, cellulose accessibility, and enzymatic hydrolysis. *ACS Sustain Chem Eng* 5(5):4011–4017
11. Fockink DH, Sánchez JH, Ramos LP (2018) Comprehensive analysis of sugarcane bagasse steam explosion using autocatalysis and dilute acid hydrolysis (H₃PO₄ and H₂SO₄) at equivalent combined severity factors. *Ind Crops Prod* 123:563–572
12. Gao A, Shen H, Zhang H, Feng G, Xie K (2017) Hydrophilic modification of polyester fabric by synergetic effect of biological enzymolysis and non-ionic surfactant, and applications in cleaner production. *J Clea Prod* 164:277–287
13. Geddes CC, Mullinnix M, Nieves I, Peterson J, Hoffman R, York S, Yomano L, Miller E, Shanmugam K, Ingram L (2011) Simplified process for ethanol production from sugarcane bagasse using hydrolysate-resistant *Escherichia coli* strain MM160. *Bioresour Technol* 102(3):2702–2711
14. Haldar D, Purkait MK (2020) A review on the environment-friendly emerging techniques for pretreatment of lignocellulosic biomass: Mechanistic insight and advancements. *Chemosphere* 264:128523
15. Haykiri-Acma H, Yaman S (2019) Effects of dilute phosphoric acid treatment on structure and burning characteristics of lignocellulosic biomass. *J Energ Resour -ASME* 141(8):082203
16. Ibrahim MM, Agblevor FA, El-Zawawy WK (2010) Isolation and characterization of cellulose and lignin from steam-exploded lignocellulosic biomass. *BioResources* 5(1):397–418
17. Jacquet N, Maniet G, Vanderghem C, Delvigne F, Richel A (2015) Application of steam explosion as pretreatment on lignocellulosic material: a review. *Ind Eng Chem Res* 54(10):2593–2598

18. Jørgensen H, Kristensen JB, Felby C (2007) Enzymatic conversion of lignocellulose into fermentable sugars: challenges and opportunities. *Biofuel Bioprod Bior* 1(2):119–134
19. Kim TH (2011) Sequential hydrolysis of hemicellulose and lignin in lignocellulosic biomass by two-stage percolation process using dilute sulfuric acid and ammonium hydroxide. *Korean J Chem Eng* 28(11):2156–2162
20. Koradiya M, Duggirala S, Tipre D, Dave S (2016) Pretreatment optimization of Sorghum pioneer biomass for bioethanol production and its scale-up. *Bioresour Technol* 199:142–147
21. Leu SY, Zhu J (2013) Substrate-related factors affecting enzymatic saccharification of lignocelluloses: our recent understanding. *Bioenerg Res* 6(2):405–415
22. Li H, Pu Y, Kumar R, Ragauskas AJ, Wyman CE (2014) Investigation of lignin deposition on cellulose during hydrothermal pretreatment, its effect on cellulose hydrolysis, and underlying mechanisms. *Biotechnol Bioeng* 111(3):485–492
23. Martín-Davison JS, Ballesteros M, Manzanares P, Sepúlveda XP-B, Vergara-Fernández A (2015) Effects of temperature on steam explosion pretreatment of poplar hybrids with different lignin contents in bioethanol production. *Int J Green Energy* 12(8):832–842
24. Nasirpour N, Mousavi S, Shojaosadati S (2014) A novel surfactant-assisted ionic liquid pretreatment of sugarcane bagasse for enhanced enzymatic hydrolysis. *Bioresour Technol* 169:33–37
25. Oliva JM, Negro MJ, Álvarez C, Manzanares P, Moreno AD (2020) Fermentation strategies for the efficient use of olive tree pruning biomass from a flexible biorefinery approach. *Fuel* 277:118171
26. Pitarelo AP, Fonseca CSd, Chiarello LM, Gírio FM, Ramos LP (2016) Ethanol production from sugarcane bagasse using phosphoric acid-catalyzed steam explosion. *J Braz Chem Soc* 27:1889–1898
27. Qi W, He C, Wang Q, Liu S, Yu Q, Wang W, Leksawasdi N, Wang C, Yuan Z (2018) Carbon-based solid acid pretreatment in corncob saccharification: specific xylose production and efficient enzymatic hydrolysis. *ACS Sustain Chem Eng* 6(3):3640–3648
28. Qing Q, Yang B, Wyman CE (2010) Impact of surfactants on pretreatment of corn stover. *Bioresour Technol* 101(15):5941–5951
29. Schwanninger M, Rodrigues J, Pereira H, Hinterstoisser B (2004) Effects of short-time vibratory ball milling on the shape of FT-IR spectra of wood and cellulose. *Vib Spectrosc* 36(1):23–40
30. Segal L, Creely JJ, Martin A Jr, Conrad C (1959) An empirical method for estimating the degree of crystallinity of native cellulose using the X-ray diffractometer. *Text Res J* 29(10):786–794
31. Shi GQ, Han C, Wang Y, Wang H-T (2019) Experimental study on synergistic wetting of a coal dust with dust suppressant compounded with noncationic surfactants and its mechanism analysis. *Powder Technol* 356:1077–1086
32. Singh J, Suhag M, Dhaka A (2015) Augmented digestion of lignocellulose by steam explosion, acid and alkaline pretreatment methods: a review. *Carbohydr Polym* 117:624–631

33. Sluiter A, Hames B, Ruiz R, Scarlata C, Sluiter J, Templeton D, Crocker D (2008) Determination of structural carbohydrates and lignin in biomass. *Lab Anal Proced* 1617(1):1–16
34. Tu WC, Hallett JP (2019) Recent advances in the pretreatment of lignocellulosic biomass. *Curr Opin Green Sust* 20:11–17
35. Wang H, Liu Z, Zheng X, Pan X, Hui L, Li J, Zhang H (2020) Assessment on temperature-pressure severally controlled explosion pretreatment of poplar. *Carbohydr Polym* 230:115622
36. Yan J, Nie W, Xiu Z, Yuan M, Zhou W, Bao Q, Peng H, Niu W, Yu F (2021) Development and characterization of a dust suppression spray agent based on an adhesive NaAlg-gln-poly/polysaccharide polymer. *Sci Total Environ* 785:147192
37. Zeng J, Tong Z, Wang L, Zhu J, Ingram L (2014) Isolation and structural characterization of sugarcane bagasse lignin after dilute phosphoric acid plus steam explosion pretreatment and its effect on cellulose hydrolysis. *Bioresour Technol* 154:274–281
38. Zhang J, Tang M, Viikari L (2012) Xylans inhibit enzymatic hydrolysis of lignocellulosic materials by cellulases. *Bioresour Technol* 121:8–12
39. Zhang J, Zhou H, Liu D, Zhao X (2020) Pretreatment of lignocellulosic biomass for efficient enzymatic saccharification of cellulose. In P D Yousuf A, Sannino F (Ed) *Lignocellulosic Biomass to Liquid Biofuels*. Amsterdam, pp 17–65
40. Zheng T, Jiang J, Yao J (2020) Surfactant-promoted hydrolysis of lignocellulose for ethanol production. *Fuel Process Technol* 213:106660
41. Zhou H, Wang J, Han C, Yang B (1995) Preparation and application of penetrant JFC(M type). *Jiangsu Silk* 01:26 (In Chinese)

Figures

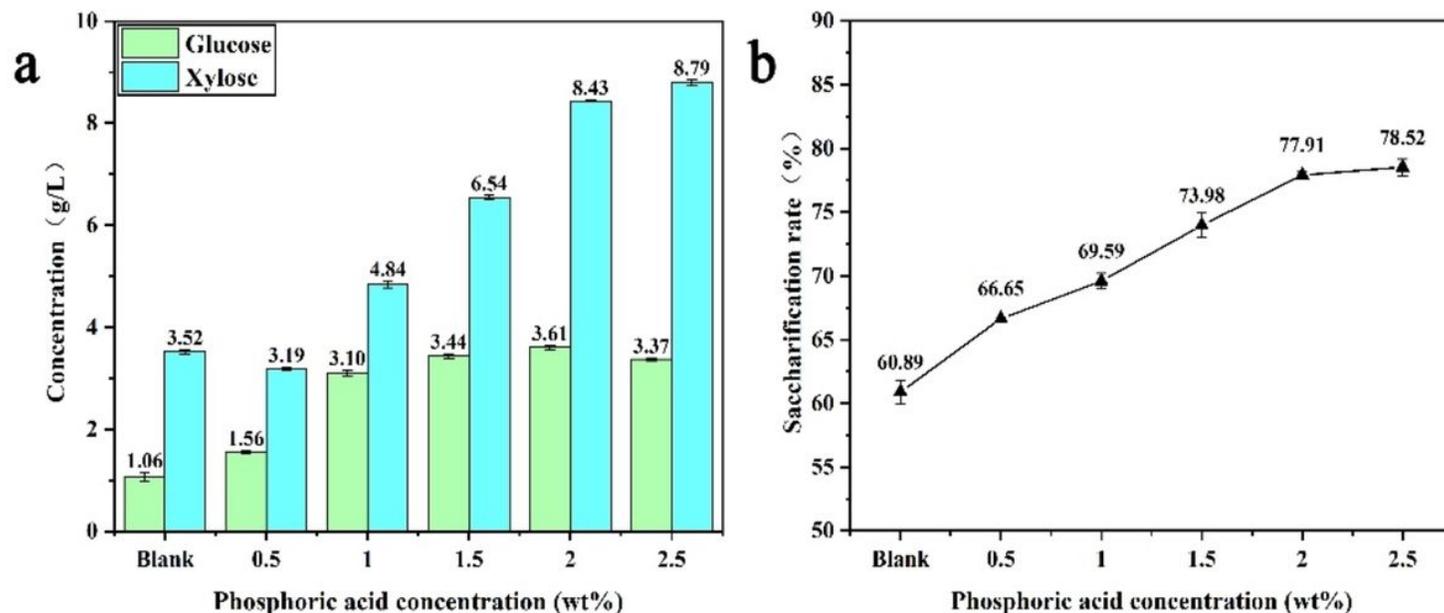


Figure 1

Effect of phosphoric acid on pretreatment. a: Yield of glucose and xylose in exploded filtrate; b: Saccharification rate. Note: (1) Blank group: 2.0 wt% H₃PO₄ + purified water; 0.5-2.5 wt% H₃PO₄ groups: adding 2 % (v/v) JFC-M. (2) SE parameters: 1:2.5 solid/liquid rate, 2 h pre-soaking time, 2.2 MPa SE pressure, 180 s SE residence time. (3) The contents of cellobiose and arabinose were below the detection limits.

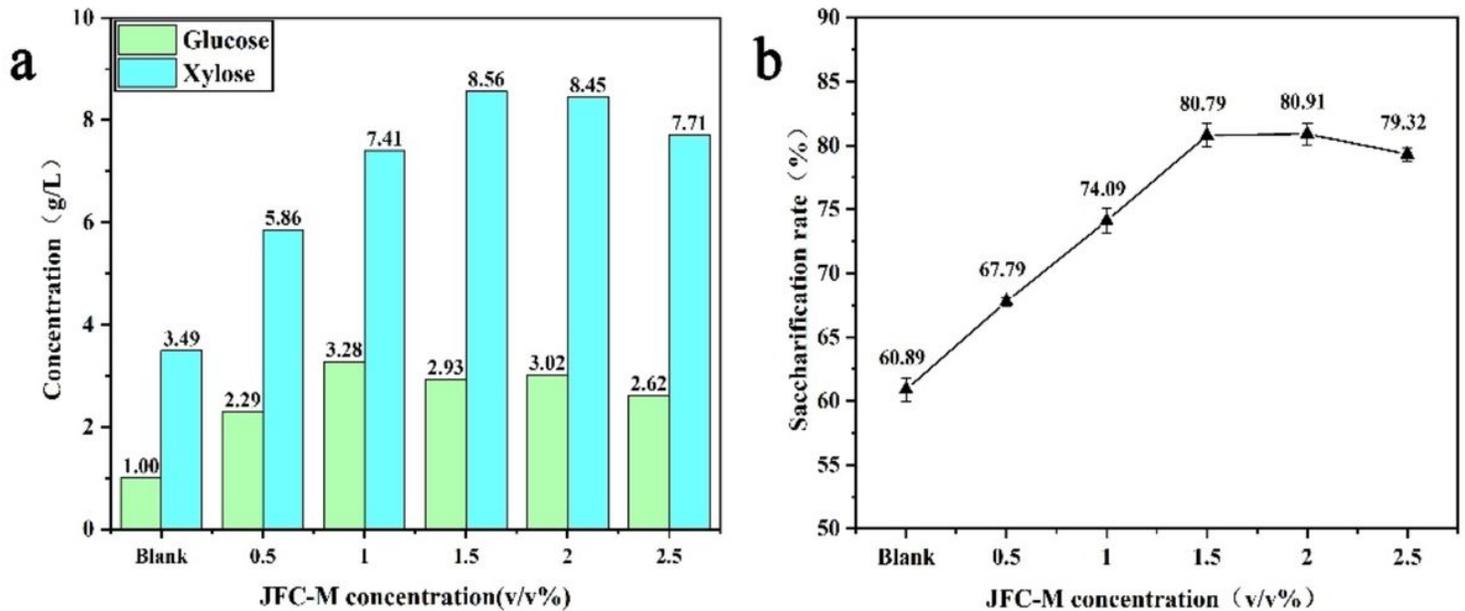


Figure 2

Effect of surfactant JFC-M on pretreatment.

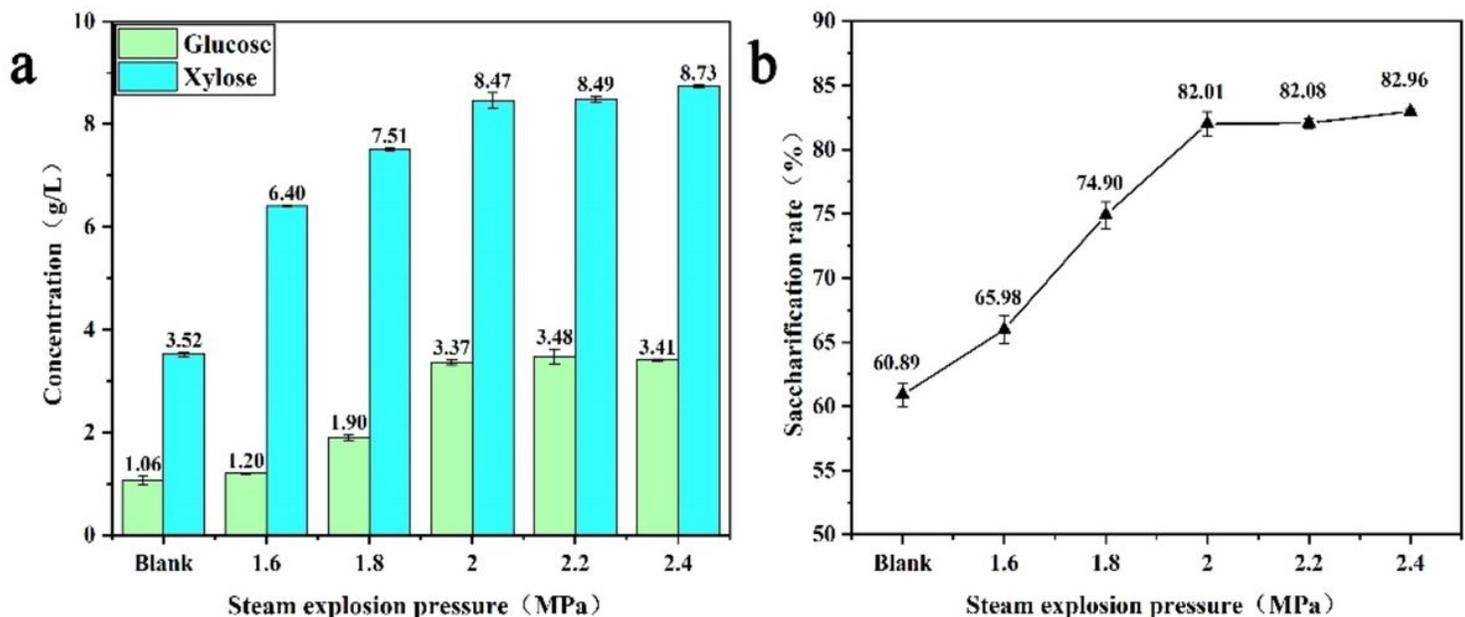


Figure 3

Effect of pressure on pretreatment.

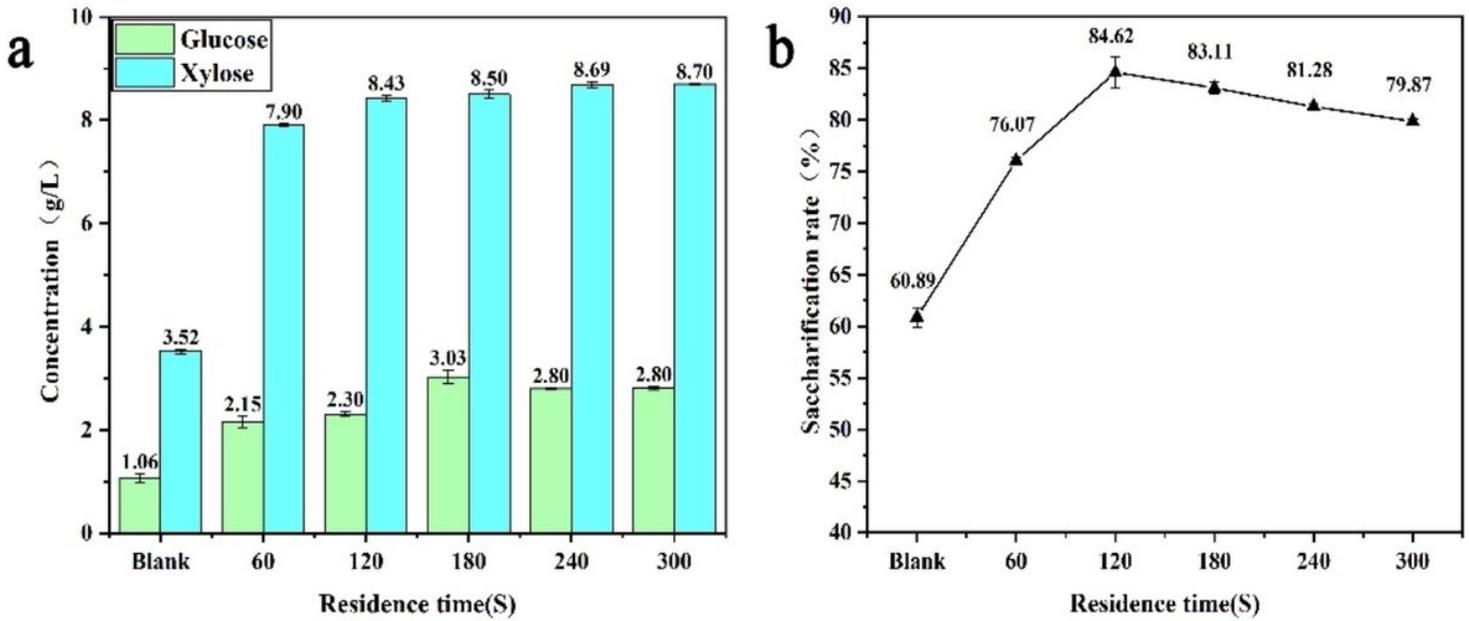


Figure 4

Effect of residence time on pretreatment. a: Yield of glucose and xylose in exploded filtrate; b: Saccharification rate. Note:(1) Blank group: 2.0 wt% H₃PO₄ + purified water;60-300 s residence time groups: adding 1.5 (v/v)% JFC-M + 2.0 wt% H₃PO₄. (2) SE parameters: 1:2.5 solid/liquid rate, 2 h pre-soaking time, 2.0 MPa SE pressure.

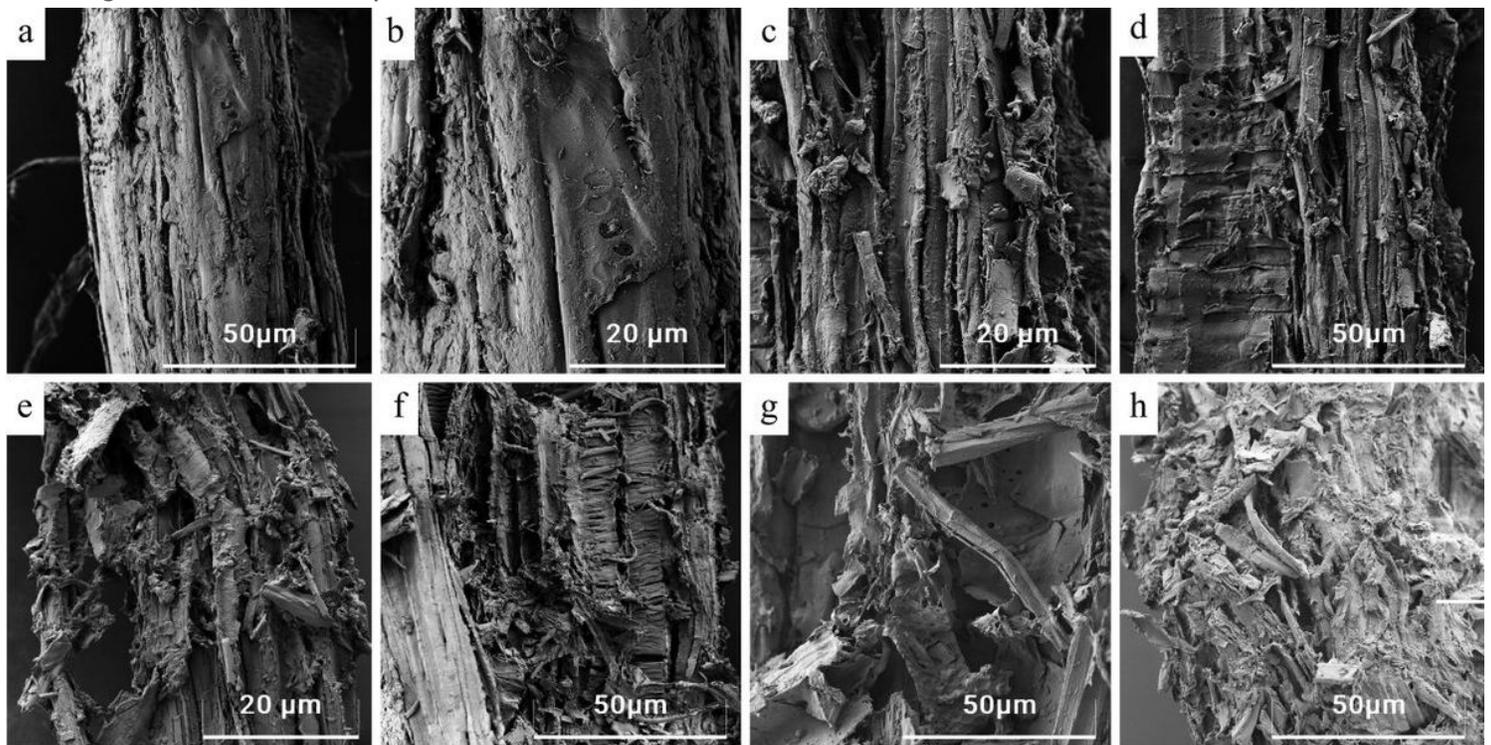


Figure 5

SEM images of samples before and after pretreatment.

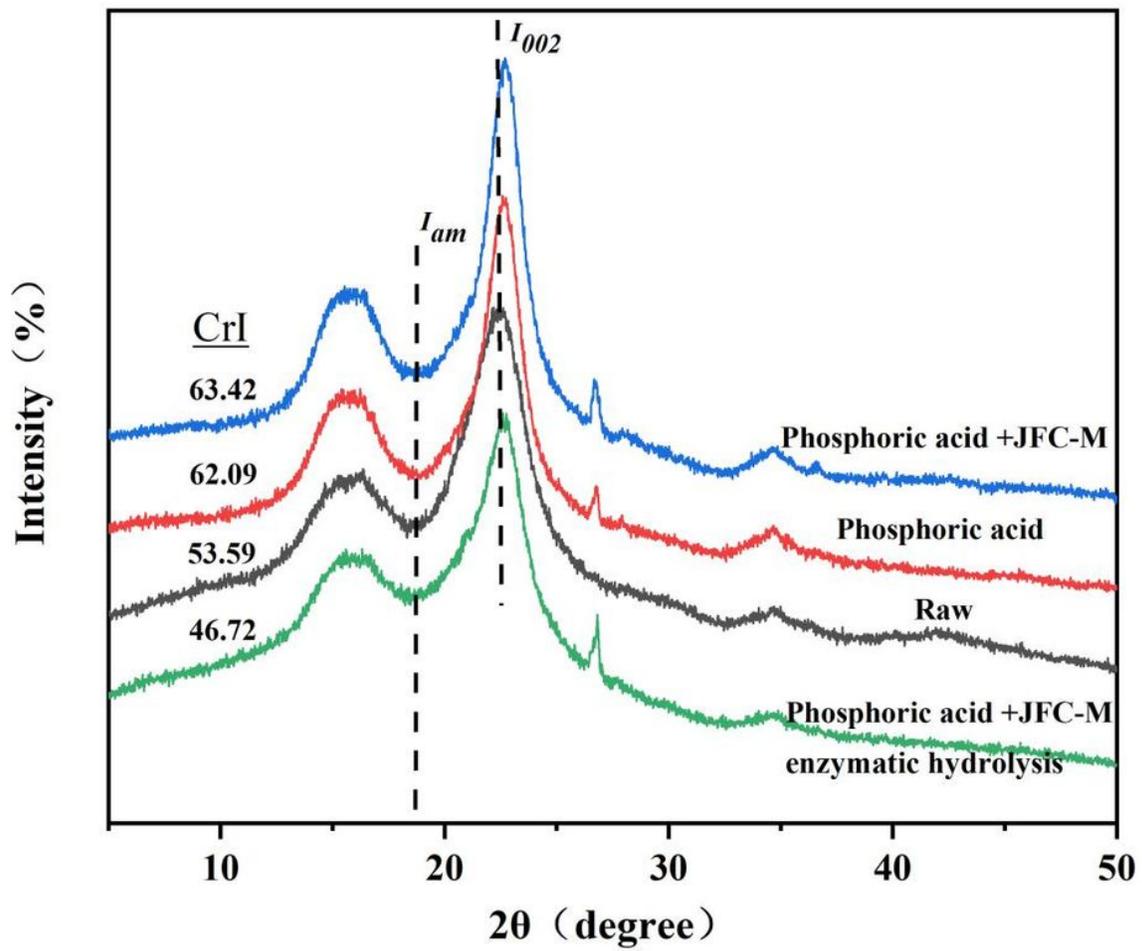


Figure 6

XRD patterns of samples.

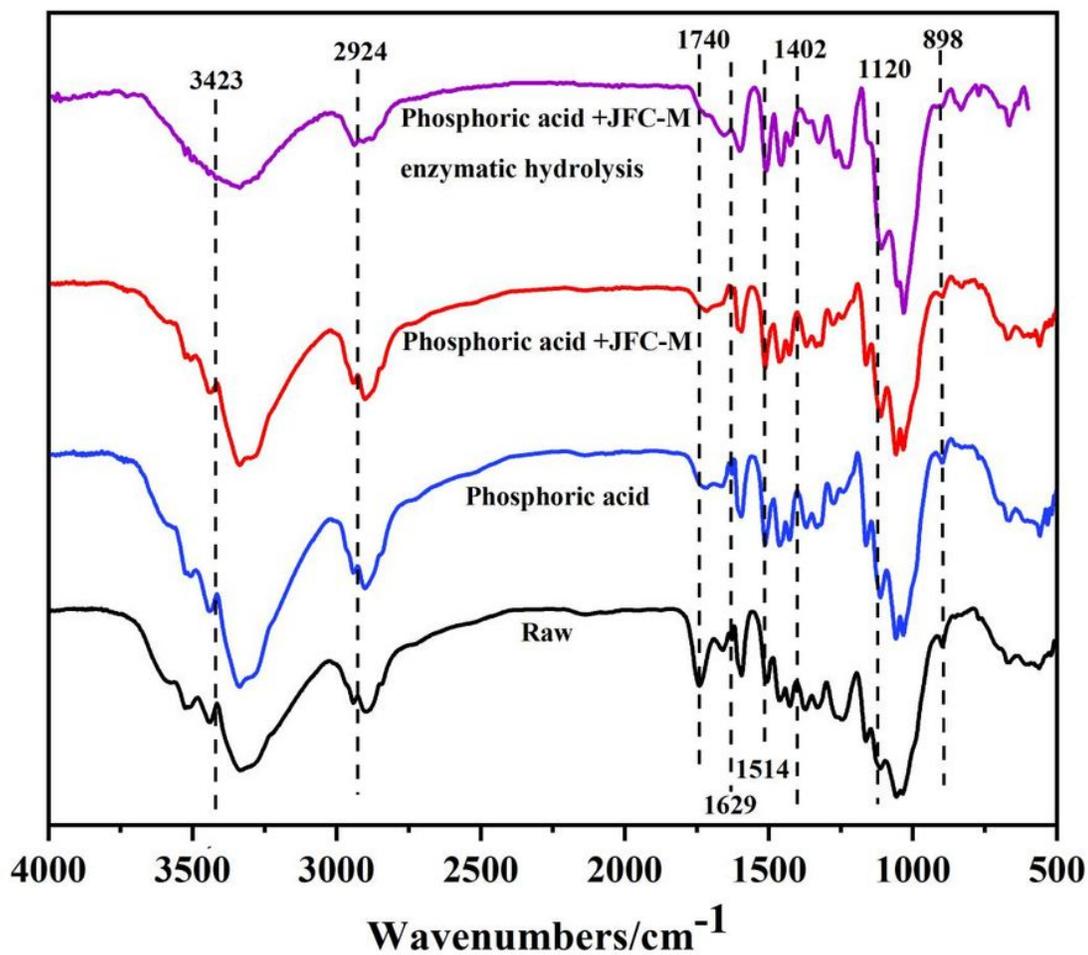


Figure 7

FTIR spectra of samples.

Supplementary Files

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