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Effect of Steam Flash Explosion Conditions on Preparation of Reed Pulp

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Abstract

The effects of different steam explosion conditions on preparation of reed pulp by steam flash explosion were analyzed. Because the main factors affecting steam explosion are steam pressure, retention time and liquid-to-reed ratio, so that the effects of these conditions on the steam explosion pretreated reed straw, and constituent of reed pulp were analyzed. Reed straw samples, 2~3cm in length, were loaded into a 5L stainless steel reactor and treated by saturated steam for various retention time, respectively. The analysis parameters were yield (steam explosion, washing and digestion yield), composition *(alpha-cellulose content, holocellulose content, hot water extract, content of pentosan, degree of delignification), reducing sugar content and total sugar content, and all experiments were performed in duplicate, and the average results are shown. FTIR and SEM were used in this study to comprehensively investigate the characteristics of the steam exploded reed straw. The untreated and steam explosion pretreated reed straw samples were subjected to an FTIR analysis, and the changes in chemical structure were investigated. FTIR spectra for both the pretreated and untreated reed straw samples indicate that no new functional groups and chemical bonds were produced in the reed straw after the steam explosion. SEM photos of reed straw treated by steam explosion under different pressures show that steam explosion method is a very effective method in modifying the properties of reed cellulose. The results of the research presented in this paper are considered to be a very important basis for future application of the reed.*

Keywords: Steam explosion, reed straw, SEM, FTIR, modification

INTRODUCTION

The lignin carbohydrate complex (LCC) or lignin-carbohydrate polymer, which is the primary structure of biomass, can be broken down using the effective, cost-effective, and environmentally benign steam explosion pretreatment method. [1–3] Steam explosion is one of the most effective pretreatment technologies for breaking structural recalcitrance of lignocelluloses to promote further feedstock conversion in subsequent enzymatic hydrolysis and fermentation in commercial production.

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Several research have documented comparisons between steam-explosion pretreatment and other pretreatments. Additionally, the pretreatment of steam explosion alters the chemical composition of biomass, including the amount of cellulose, hemicellulose, lignin, and other particular components. Steaming (also known as steam loading) and explosion are the two primary processes in this process.

The lignocellulose material is exposed to high pressure saturated steam during the steaming process, which is based on the hydrothermal pretreatment method with high pressure and short retention time. After then, it abruptly depressurized to create the effect of an explosion due to the pressure differential between the steaming chamber's pressure and the explosion chamber's regular pressure. [4] The depolymerization, dissolution, and removal of lignin and hemicellulose into lower molecular weight products are aided by the explosion stage.

The crystalline structure of cellulose is thought to become disordered after steam explosion pretreatment, increasing the surface area and improving enzyme accessibility, which results in more efficient hydrolysis. The ideal temperature-pressure and retention time parameters for the steam explosion process vary depending on the kind of feedstock since a rise in steam pressure and residence time, in particular, promotes the disruption of the cellulose structure. [5–13]

Natural cellulose fibers made from reed straws have important qualities, and their industrial potential is currently being thoroughly reexamined. Reed is a promising and advantageous renewable resource that may be used to create cellulose at a cheap cost with acceptable features and biodegradability The main factors affecting steam explosion process are steam explosion pressure, steam explosion retention time and liquid-to-reed ratio.

Therefore, this paper aims to investigate the effects of these conditions on the steam explosion pretreated reed straw and constituent of reed pulp.

MATERIALS AND METHODS

Materials And Preparation

The reeds differ from wood in several characters such as chemical composition and morphological structure.

For industrial uses, reed's physicochemical characteristics must be carefully taken into account The thickness of the primary cell membrane in reed cells is 0.2~0.8 ㎛, the thickness of the outer layer of the secondary cell membrane is 0.7~0.8 ㎛, the thickness of the middle layer is 3.5~5.7 ㎛, and the thickness of the inner layer is 0.3~0.4 ㎛.

The thick cell membrane of reed cells may be an important reason that reed pulp has the poor reactivity and beating degree.

Therefore, the industrial application of reed requires focus on the effective removal of reed cell membranes and to create pulping conditions that would severely disrupt the morphological structure of reed cells. The helical twist of fibrils can affect the swelling of the fiber.

In reed fibers, the cellulose fibers twisted in a helical shape with a large angle of 42° about the fiber axis. Of course, there are other reasons, but it can be explained that the reed fibers are not well swelled due to the large twist angle. Thus, when making pulp for chemical fibers from reeds, each process must be treated in a stronger condition than the conventional wood material.

An appropriate chemical composition of reed is 16.3~16.9% of lignin, 39.1~39.9% cellulose, 27.3~28.4% hemicelluloses, 3.86~4.2 of ash and 12.1~15.3% of extractives.

The Silicone content in the ash of reed is relatively high and this makes it one of the most difficult problems in application of reeds as raw materials for rayon pulp production.

However, in leaves, joints, bark, etc. ash of reed is abundant and content of cellulose is low, so these parts must be removed. Therefore, the stem portion of reeds (leaf and bark was removed) was cut into pieces 2~3 cm in length at an automatic cutter (Fig. 1) and used as raw material The moisture content of the sample was 10.34%.

Figure 1. Automatic cutter.

Liquid-to-reed ratio means that moisture content of the sample to dry weight basis of raw reed, so the water content of the sample is 33.33% in the case that Liquid-to-reed ratio is 0.5.

Steam Explosion Pretreatment and Alkali Pulping Process

The apparatus for the steam explosion (Fig. 2) consisted of boiler (a steam generator), steam explosion equipment (high-pressure reactor), receiver, and computer, etc.

The steam pretreatment of reed straw was carried out in a 5L stainless steel reactor.

The reactor was charged with 250g (dry weight basis) of feedstock per batch and quickly heated to the desired temperature (the heating time was less than 10 s) and treated by saturated steam.

Once the desired residence time and temperature in a constant-pressure had been reached, then an electromagnetic ball valve with a diameter of 55mm triggered the explosion, and the depressurization happened in 0.01 seconds.

In the single-factor experiments, a single selected factor varied, while all other factors were fixed simultaneously in the optimal conditions obtained in every step.

The average outcomes of each experiment, which was carried out in duplicate, are displayed Some of the pretreated material was taken as the corresponding index analysis sample and the remaining sample was washed, and then performed alkali pulping under the same conditions.

The alkali pulping technique was used to the steam-exploded reed straw.

At 170 °C for 90 minutes, the sample was reacted with 20% (w/w) NaOH in a 4:1 liquid to solid ratio. Analytical-grade chemicals were all utilized just as supplied, requiring no additional purification.

Analytical Methods

Measurement of yield

The dry weight of the solid left over after explosion treatment was compared to the dry weight of reed straw added to the reactor in order to compute the steam explosion yield.

Following the steam explosion, samples were rinsed with clean water through a sieve (mesh size 80) until the water ran clear, oven-dried at 60°C, and weighed to determine the proportion of solid material that remained (100 g of the steam-exploded reed straw). Washing yield is the definition of this % figure Digestion yield was calculated as the dry weight of the solid remaining after digestion treatment referred to the dry weight of reed straw pretreated with a steam explosion.

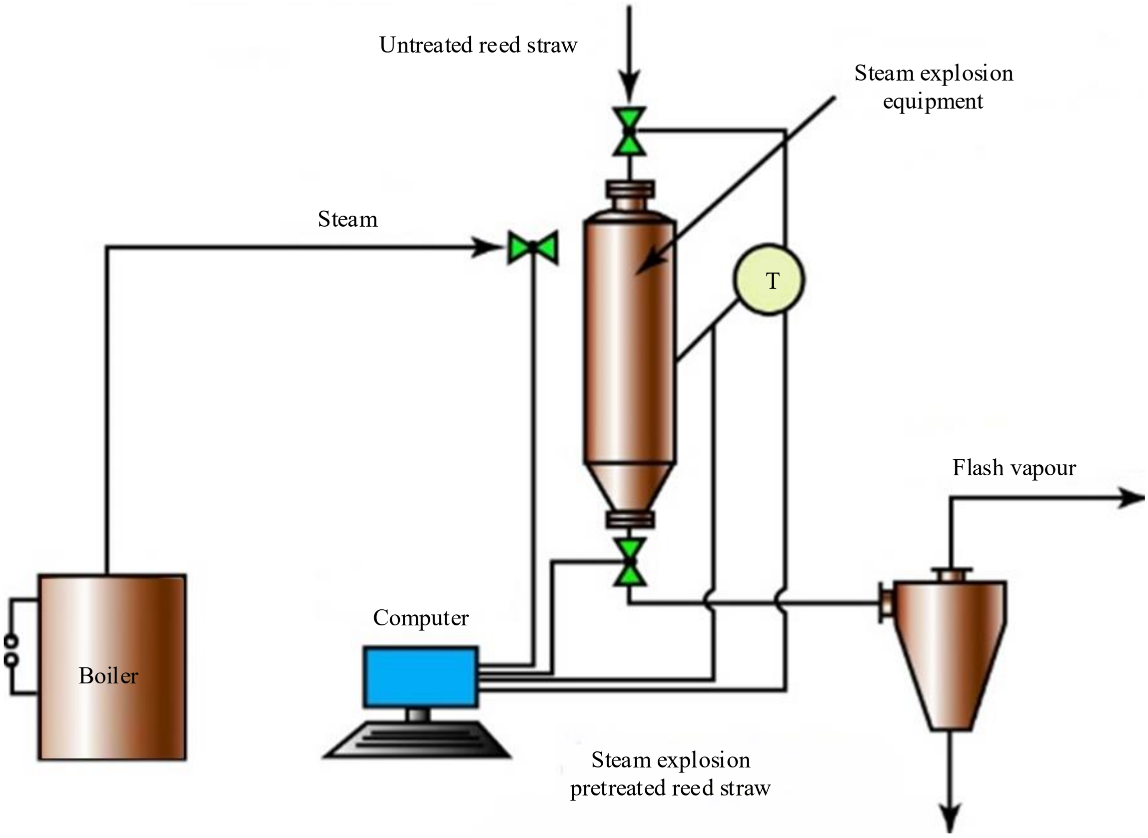

Figure 2. Schematic of a batch steam explosion equipment.

Composition analysis

Alpha-cellulose content was tested according to TAPPI-T203-om-93, holocellulose content according to GB/T2677.8, hot water extract according to GB/T2677.4, content of pentosan (or pentosan removal yield)according to GB/T2677.9, degree of polymerization (DP) according to TAPPI-T230-om-94, lignin content (or degree of delignification) according to TAPPI-T222-om-98.

Measurement* of the reducing sugar *content

Each sample of steam explosion pretreated reed straw was transferred to a 200-mL flask with Milli-Q water. The diluted sample was centrifuged at 6 000 rpm for 10min, and the supernatant was passed through filter paper. The 3, 5-dinitrosalicylic acid method (DNS method) was ultimately used to determine the reducing sugar. The average findings of the two analytical determinations of the reducing sugar are displayed.

***Measurement* of the total sugar content**

The total saccharide content (saccharide compounds: arabinose, galactose, glucose, xylose, mannose and cellobiose) were determined by HPAEC (high- performance anion-exchange chromatography). The detection experiment on the saccharide compounds was performed by using a Dionex ICS-3000 ion chromatograph system.Each sample of steam explosion pretreated reed straw was transferred to a 200mL flask with Milli-Q water. And these samples were further diluted prior to the analysis to bring the sample concentration into the linear range of the pulsed amperometric detector and the diluted sample was centrifuged at 6 000 rpm for 10 min. Then, the supernatant was filtered through a nylon membrane with a pore size of 0.22㎛ and the samples were finally injected into the column, respectively.

The average outcomes of the analytical calculations, which were carried out in duplicate, are displayed.Ion chromatography of sample was carried out using a CarboPac PA20 (150mm×3 mm i.d., Dionex) anion-exchange column for saccharide separation, with an AS40 autosampler, pulsed amperometric detector, Au electrode, Ag-AgCl reference electrode and linked personal computer loaded with the Chromeleon 6.7 software to record and process the data.

Scanning electron microscopy (SEM)

The morphological property of steam exploded reed cellulose was carried out using a scanning electron microscopy (SEM), JEOL JSM-6380 LV scanning electron microscope at an applied voltage of 15 kV.

Prior to SEM measurements the samples of steam exploded reed cellulose were mounted on aluminum stubs and coated with a thin layer of gold by a SPI sputter coater.

Fourier-transformed infrared (FTIR) spectroscopy

FTIR spectroscopy is a non-destructive method used to obtain important information about the chemical structure of the steam exploded reed straw. The structure of the steam-exploded reed straw was monitored by using a PerkinElmer "Spectrum BX" spectrometer.

Spectra were recorded from KBr pellet samples in the wavenumber range 4 000 to 400cm^{-1}, with a resolution of 0.4 cm^{-1}. The sample dried under vacuum for 24 h before the FTIR measurement.

RESULTS AND DISCUSSION

Effects of The Steam Explosion Pressure

Effects of the steam explosion pressure on the steam explosion pretreated reed straw

Steam explosion shall be carried out under as safe conditions as possible, i.e. in the temperature range from the softening temperature of lignin and hemicellulose (160~200 °C) to the softening temperature of cellulose (231~253 °C). Therefore, steam explosion treatment was carried out by varying the steam explosion pressure from 1.0 to 2.0 MPa. Figures 3 and 4 display the digital and SEM micrographs of untreated reed straws and pretreated reed straws by steam flash explosion at various steam explosion pressures. According to the digital micrographs, the pretreated reed straws' color clearly turned dark brown as the steam explosion pressure increased from 1.0 to 2.0 MPa. Higher steam explosion pressures can split reed straws into finer fibers, as illustrated in Figure 4. These fibers have more longitudinal grooves and cleaner surfaces.

The steam explosion isolated single fibers from fiber bundles and increased the surface area exposed to chemicals. Therefore, the reaction was carried out effectively.

As shown in Figure 5, the steam explosion yield decreases from 98.70% to 90.0% and the washing yield decreases rapidly from 78.51% to 70.3% with increasing steam explosion pressure.

Steam explosion yield was higher than that of prehydrolysis (85%), but the yield after washing with cold water was lower than that of prehydrolysis.

The content of pentosan of the steam-exploded reed straw also decreases gradually as shown in Figure 6 with the intensification of the steam explosion, from about 15% to 6.5% in the steam-exploded reed straw, from 12.06% to 5.44% after washing, especially decreases rapidly at more than 1.6 MPa.

Pentosan removal yield can be removed by about 65% in the case of steam exploded reed straw and 80% after washing under same experimental condition. When the steam exploded reed straw is washed with water, the yield is significantly reduced by about 20%, because the wood components converted to water soluble form during the steam explosion are still contained in the steam explosion pretreated reed straws.

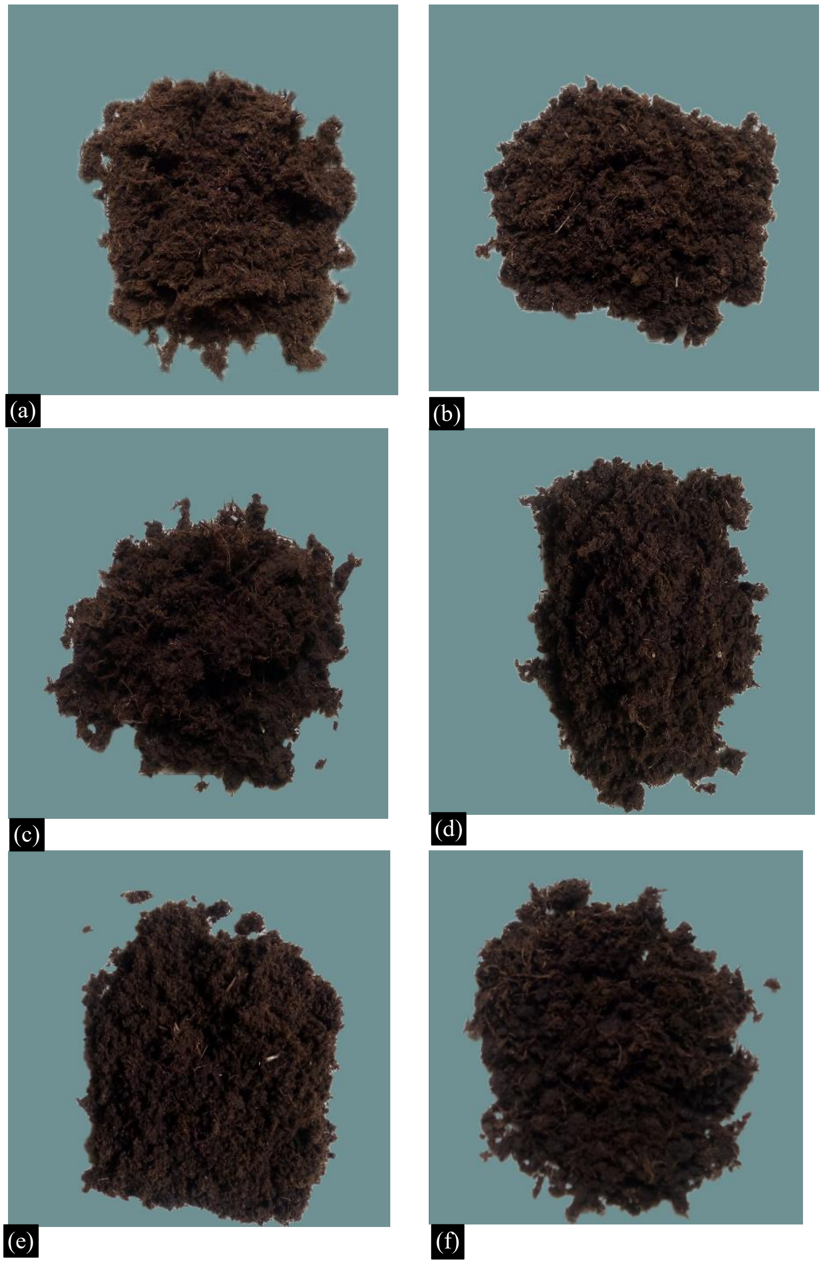

Figure 3. Digital images for steam explosion pretreated reed straws by steam flash-explosion under the steam pressure of (a) 1.0 MPa, (b) 1.2 MPa, (c) 1.4 MPa, (d) 1.6 MPa, (e) 1.8 MPa, and (f) 2.0MPa for 3 min retention time.

Before washing of steam-exploded reed straw

After washing of steam-exploded reed straw

The results of the hot water extract are shown in Figure 7, the amount of components extracted into water ranges from 17% to 21.5% with increasing steam explosion pressure.

The analysis results about effect of the steam explosion pressure are shown in Figure 5.

These extracts contain a variety of monosaccharide and oligosaccharide produced as a result of hydrolysis during steam explosion. It contains up to high of 16% as total reducing sugar, and above 1.8 MPa it tends to decrease. This is probably due to the heavy degradation of the produced saccharides.

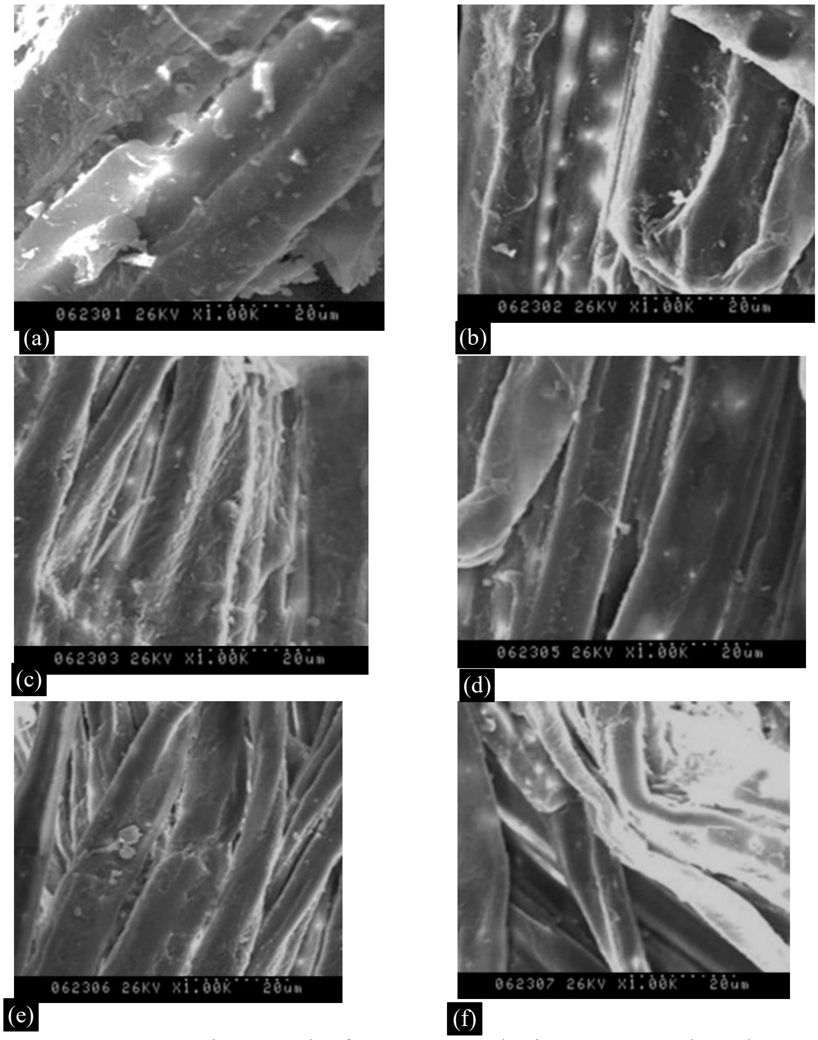

Figure 4. SEM micrographs for steam explosion pretreated reed straws by steam flash explosion under the steam pressure of (a) 1.0 MPa, (b) 1.2 MPa, (c) 1.4 MPa, (d) 1.6 MPa, (e) 1.8 MPa, and (f) 2.0MPa for 3 min.

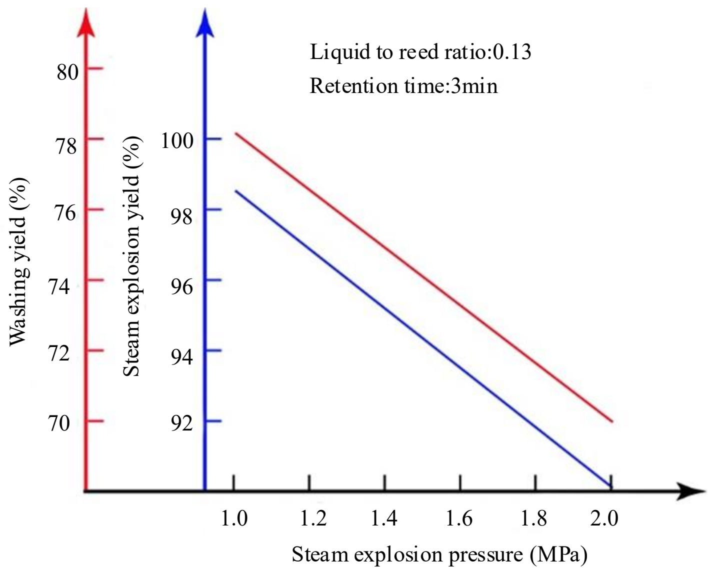

Figure 5. Effects of the steam explosion pressure.

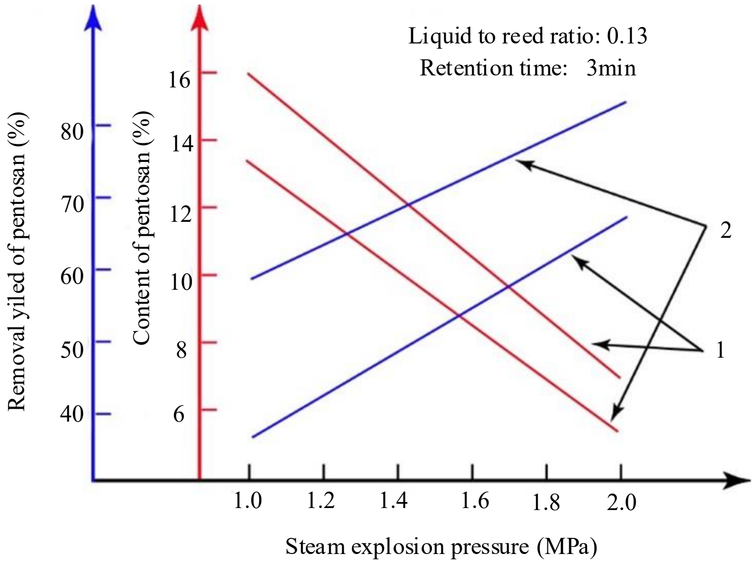

Figure 6. Curve of change of content of pentosan with steam explosion pressure.

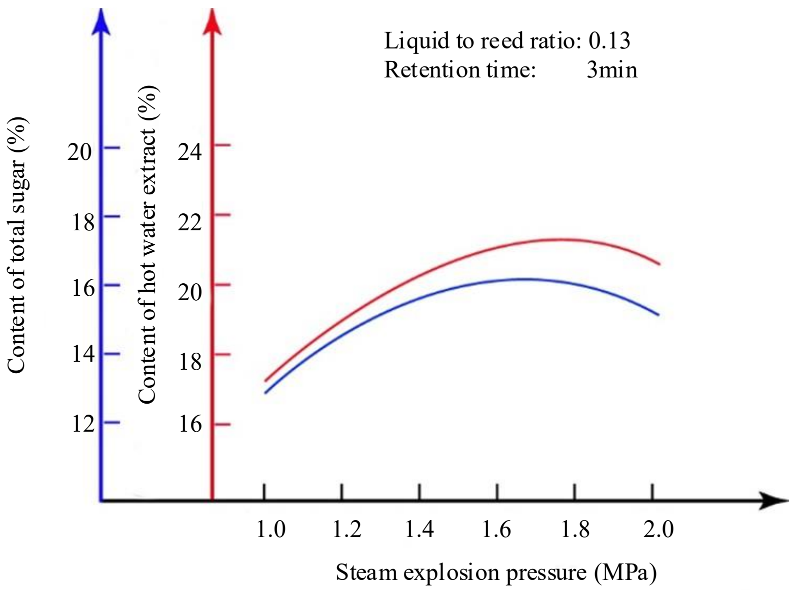

Figure 7. Content of hot water extract and content of total saccharides with steam explosion pressure variation.

Effect of steam explosion pressure on constituent of reed pulp

The character changes of unbleached pulp obtained by soda digestion of steam-exploded reed straw under different steam explosion pressure conditions are shown in Table 1.

Table. 1. Effect of steam explosion pressure on constituent of reed pulp.

Condition of steam explosion		**Steam explosion yield (%)**	**Degree of delignification**	**Pentosan**		**Content of celluloses (%)**		**Degree of polymerization**
Pressure (MPa)	***Time (min)***			***Content (%)***	***Removal yield (%)***	***Holo-cellulose***	***α-cellulose***	
1.4	3	40.76	51	6.59	88.35	97.23	88.29	1180
1.6	3	38.19	47	5.11	91.55	97.79	90.25	1110
1.8	3	34.55	37	3.80	94.32	98.09	91.89	1055
2.0	3	30.16	35	3.46	95.67	97.07	89.64	816

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The holocellulose and alpha cellulose contents increased with increasing steam explosion pressure, unlike the steam explosion yield, and then decreased again at above 1.8 MPa. The decrease in yield from above 1.8 MPa was attributed to the destruction of fibers by high pressure, and the gradual decrease in the degree of polymerization with increasing steam explosion pressure. The pentosan removal efficiency during soda digestion is very high, and there are some differences in the digested samples as well as the pentosan of steam explosion pretreated reed straws, but samples under other conditions except 1.4 MPa are reaching the pentosan index of rayon pulp. From the above experimental results, it is expected that the content of pentosan can be well below 4% that is lower than the prehydrolysis method. Considering the various conditions, it is most reasonable to have a steam explosion pressure of 1.8 MPa.

Effects of Retention Time

Effects of retention time on the steam explosion pretreated reed straw

The infrared spectra of the untreated and steam explosion pretreated reed straw samples are shown in Fig.8. All curves exhibit similar absorption bands at 3348 cm^{-1} (O-H stretching, H-bonded), 2918 cm^{-1} (C-H stretching vibrations), 1704 cm^{-1} (hemicellulosic sub-fractions), 1650 cm^{-1} (C=C aromatic skeletal vibration), 1428(aromatic C-H vibrations), 1057 cm^{-1} (arabinosylside chains or C-O-C stretching for glucose ring), 900 cm^{-1} (β-glycosidic linkages) and 835 cm^{-1} (α-anomers in side chains).

This indicates that the main chemical structure of the reed straw had not been significantly changed, and that no new functional groups and chemical bonds were produced in the reed straw after the steam explosion.

A, untreated; B, pretreated for 1min; C, pretreated for 2min; D, pretreated for 3min; E, pretreated for 4min

The change in the quality of the steam explosion pretreated reed straws with time when the steam explosion retention time is changed from 1 to 4 min is shown in Figure 9.

With the increase of the steam explosion retention time, hydrolysis of wood is more strengthened by the increasing the organic acid production. As a result, the steam explosion yield decreases linearly to 90% after steam explosion and total yield after washing decrease to about 70%.

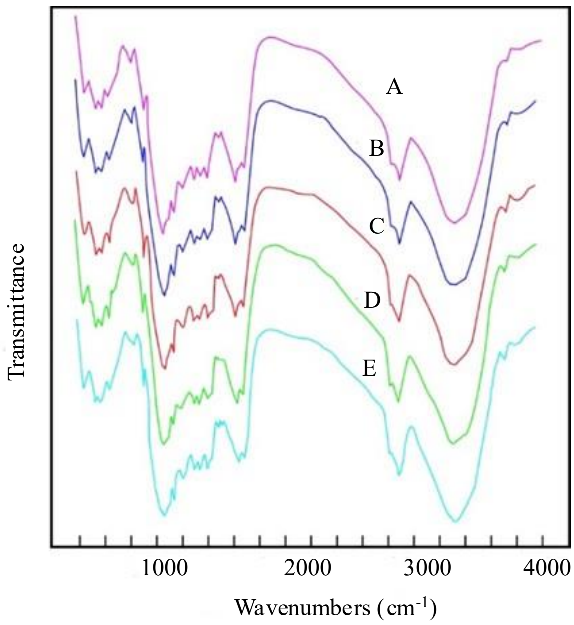

Figure 8. FTIR Spectra of untreated and steam explosion pretreated reed straw.

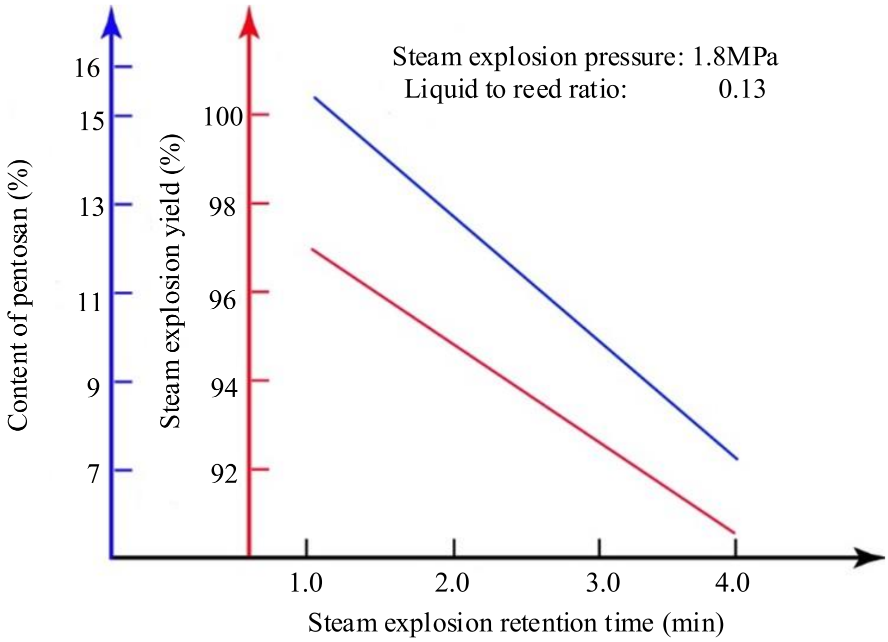

Figure 9. Steam explosion yield and content of pentosan with the change of steam explosion retention time.

With increasing steam explosion retention time, the content of pentosan is severely lowered, which decrease to about 8~9% lower within the experimental condition range.

The content of pentosan decreases gradually in the whole range, but it was more severe especially above 3 min. The content of pentosan reaches 7.16% and 5.51% for steam-exploded samples and washed samples, respectively. This index is lower than content of pentosan of the prehydrolysed reed samples.

As a result, it can remove up to 83% of the initial content of pentosan in steam explosion process, which is considered to be of higher level than the case of prehydrolysis (60%).

As the steam explosion retention time increases, the oligosaccharide is converted into monosaccharide, so that up to a certain time, the higher the monosaccharide content.

But the longer the steam explosion retention time, the lower the total saccharide content and the lower the water extract content, as glycolysis occurs.

Thus, the hot water extract content of steam explosion pretreated reed straws and the changing behavior of water-soluble saccharides are ultimately related to the change of content of pentosan.

With increasing steam explosion retention time, the water extract content of steam explosion pretreated reed straws changes from about 17 to 21%, which increases up to 3 min and then decreases again.

This tends to equal change of the water-soluble saccharide content of the steam explosion pretreated reed straws. As shown in Figure 10, when steam explosion pretreated reed straws were extracted with water, the reducing sugar content increased from 5.89% to 9.45% and then decreased to 6.45% at 4 min.

However, reducing sugar content gradually decreases from about 10% to about 8% with increasing time.

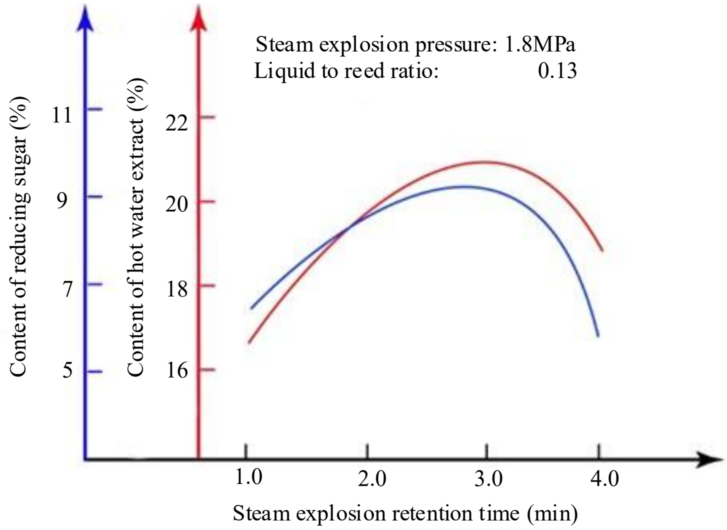

Figure 10. Content of hot water extract and content of reducing sugar with steam explosion retention time.

Effects of retention time on constituent of reed pulp

As shown in Figure 11, delignification of the pulp is more strengthened with increasing steam explosion retention time under the same digestion conditions, and the degree of delignification shows a severe difference of about 30 units from 65 to 34. The degree of delignification was reduced by about 10 units, i.e. 1/2, between 3 and 4 minutes, compared to a 21-unit reduction in the steam explosion time from 1 to 2 minutes. The content of pentosan is relatively rapidly decreasing until a certain time, but after 3 min the rate is slow and is reached below 4%. Both digestion yield and degree of polymerization of reed pulp trend to decrease with increasing retention time, it indicates that the pulp purity was not high at 1 min and 2 min of retention time. Because some cellulose was harmed during the steam flash explosion during steam explosion retention duration greater than three minutes, the adverse effects might have happened.

Therefore, there is no need to set the steam explosion retention time more than 3 min.

Effects of Liquid-To-Reed Ratio

Effects of liquid-to-reed ratio on the steam explosion pretreated reed straw

The liquid-to-reed ratio was varied from 0.13 to 3.0 to characterize the quality of the steam-exploded samples and unbleached pulp. The liquid-to-reed ratio has a certain effect on the quality of steam explosion pretreated reed straws and unbleached pulp. As shown in Figure 12, when liquid-to-reed ratio is changed from 0.13 to 3.0, the steam explosion yield decreases to liquid-to-reed ratio 0.5 and then gradually increases again.

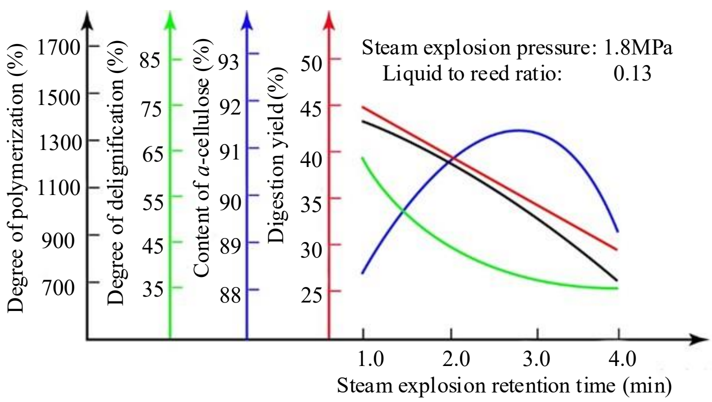

Figure 11. Change of pulp quality with steam explosion retention time.

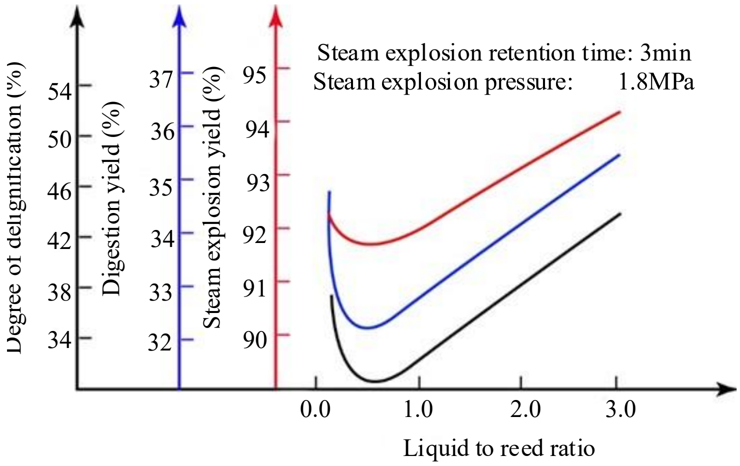

Figure 12. Steam explosion yield and degree of delignification with liquid-to-reed ratio.

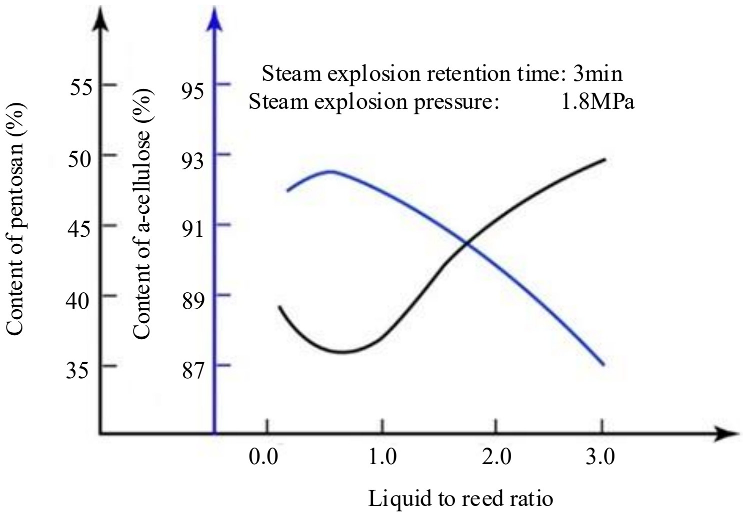

Figure 13. Content of α-cellulose and content of pentosan with liquid-to-reed ratio.

Effects of liquid-to-reed ratio on constituent of reed pulp

As shown in Figure 13, other quality parameters, including pentosan and cellulose content, also show the same trend.

That is, if the liquid-to-reed ratio is higher than a certain limit, the steam explosion will be inadequate. Therefore, in steam explosion process, the liquid-to-reed ratio should not exceed 0.5, and it is reasonable to perform below it when considering operational convenience and economic aspects.

CONCLUSION

The amorphous and non-cellulosic components of reed straw can be extracted by a steam flash explosion, which can also break down the tight lignocellulose structures Reed straw was treated by steam flash explosion at different steam explosion conditions in a 5L stainless steel reactor.

Effects of the steam explosion pressure, retention time and liquid-to-reed ratio on the steam explosion pretreated reed straw and constituent of reed pulp were analyzed.

Excessive steam explosion pressures or prolonged retention periods will break down cellulose's crystal structures and potentially cause it to breakdown; also, the liquid-to-reed ratio shouldn't be greater than 0.5.

Scanning electron microscopy (SEM) photos showed surface morphology of reed straw were damaged after the steam explosion and small fibers were produced by fibrillation.

Fourier-transformed infrared (FTIR) spectroscopy for both the pretreated and untreated reed straw samples indicate that the main chemical structure of the reed straw had not been significantly changed after the steam explosion.

The results show that the steam explosion pretreatment had such effects on the organizational structure and morphological properties of the reed straw as the removal and destruction of hemicellulose and lignin.

A rapid, efficient, chemical-free, and practical method for creating reed pulp with desired qualities is moderate steam flash explosion.

Credit Authorship Contribution Statement

Ryu-Gyong Kim: Conceptualization, Investigation, Visualization, Writing-original draft, Writing - review & editing.
Yong-Chol Jang: Conceptualization, Methodology, Investigation, Writing - original draft, Writing - review & editing, Visualization, Supervision, Project administration.
Jin-Guk Pak: Investigation, Visualization, Writing - review & editing, Supervision.
Il-Hak Kim: Conceptualization, Validation, Writing – review & editing
Tan-Gyol Yun: Conceptualization, Validation, Writing - review & editing, Supervision.
Su-Il Kim: Conceptualization, Investigation, Validation, Writing - review & editing, Supervision.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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