Synchronous Silicon Removal and Viscosity Reduction in the Soda-Oxygen Pulping of Wheat Straw

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Synchronous silicon removal and viscosity reduction in the soda-oxygen pulping of wheat straw

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Abstract

The black liquor (BL) obtained by straw pulping can hardly be applied to conventional alkali recovery systems because of its high concentration of silicon and viscosity. Soda-oxygen pulping can synchronously deposit silica on the surface of the cellulose to reduce the silicon content and viscosity of BL remarkably. In this paper, the BLs of wheat straw soda-oxygen puling obtained at different end points (pH < 10, 11.5 < pH < 12) and conventional soda-anthraquinone (soda-AQ) were obtained. The extent of silicon removal and viscosity reduction before and after centrifugation or membrane filtration as well as the thermodynamic properties of the BLs were investigated. Compared with that achieved by soda-AQ, over 45% silicon was removed from BL after soda-oxygen cooking at a similar delignification level. The total solid (TS) concentration of the soda-oxygen BL was easily concentrated by up to approximately 50%. SiO_2 can be further removed by simple centrifugation and membrane filtration, and its TS could be increased to 60% at 300 mp·s. With cooking end point further decreased pH <10, the centrifugated BL had the lowest silica content, the highest volumetric isothermal expansivity (VIE) value, and the lowest pyrolysis temperature.

Keywords Wheat straw · Silicon removal · Viscosity reduction · Soda-oxygen pulping

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Introduction

Wheat straw is a low-cost, valuable, and abundant bioresource that can be used in many fields, such as feed and compost. Massive quantities of this bioresource, the annual output could reach 100 million tons, cannot be proper treated and utilized in a timely manner (Jiang et al. 2009; Pang et al. 2012). While modern treatment methods have been intensively developed to expand its range of applications, wheat straw features a low effective utilization rate of <60%. The unused straw is usually burned without treatment, which causes serious environmental pollution and biomass waste. Therefore, effective methods to enhance the utilization of this bioresource are highly desirable (Zhang et al. 2018).

Pulping is an effective method to consume large biomass resources. As the feedstock for pulping, wheat straw may play an important role in various raw materials because of its low cost, short growth period, and wide availability (Yang et al. 2017). However, its high ash and silicon contents could give rise to the "silicon problem" in the BL derived from wheat straw pulping. BL with high silicon contents and viscosity can hardly be recycled by traditional alkali recovery systems, which directly results in its poor market share (Cardoso et al. 2009; Li et al. 2015). Today, only 4% straw produced from feedstock is used for pulping in China.

Researchers have been developed many methods to address the inherent deficiency of wheat straw for pulping. Wet and dry stock preparation could decrease the silica and ash contents of wheat straw, and vertical digesters for straw pulping could reduce the cost of heat while increasing production efficiency (Yue et al. 2016). Recycling of BL during the cooking process could increase the solid contents in the BL. However, while these strategies can decrease the silica in the BL by more than 70%, there are still some silicon remained in the BL (Belov et al. 2012; Mohan et al. 2006). Moreover, alkali recovery in the BL is fairly challenging.

Soda-oxygen pulping (soda-O_2) is performed under low temperatures (120 °C) over short times (30 min) and can increase the yield of pulp with the effective removal of lignin (Yang et al. 2016). Because of the synergistic effect between oxygen and alkali, the pH the BL would decreased pH < 10, which can synchronously retained the silicon on the pulp fibers during the pulping process (Demirbas et al. 2006). The BL obtained from soda-O_2 of wheat straw has an acceptable silica content and low viscosity which similar to the BL obtained from wood pulping (Brown et al. 2013; Oba et al. 2006). Thus, the latter is superior to the former in terms of BL features. However, the lack of research on BL characteristics following treatment in a recovery furnace still confused the application on the alkali and energy recovery (Jafari et al. 2014; Zhang et al. 2017). Investigating the properties of the BL obtained from wheat straw pulping is an important endeavor. This paper focuses on the viscosity, silica content, and thermodynamic properties of BL obtained from soda-O_2 for wheat straw.

2.Experimental Section

2.1Chemical composition of wheat straw

Table 1. Chemical composition of wheat straw (wt%)

Ash	SiO_2	Pentosan	Benzene-ethanol extractives	Holocellulose	Klason lignin	Acid-soluble lignin
8.89	1.16	20.05	2.21	65.75	22.48	2.83

Wheat straw was sampled from Jining, Shandong Province, and its chemical composition is listed in Table 1 (Danielewicz et al. 2017). The standard methods of the Technical Association of the Pulp and Paper Industry (TAPPI, Atlanta, GA) were used to determine the chemical composition of the materials, including acid-insoluble lignin (T 222 om-26), ash (T 244 cm-99), pentosane (T 233 cm-01), SiO_2 (T 245 cm-98), and cellulose content based on the nitric acid-ethanol method (T 203 om-93). Its chemical composition is listed in Table 1.

2.2Preparation of the BL

The cooking trial conditions are shown in Table 2. After cooking, centrifugation was used to separate BL from the pulp and then washed BL with hot water three times. The detail information was act in accordance with our previous work (Zhang et al. 2017).

Table 2. Cooking trials

	Alkali charge/(wt%)	Assistant amount/g	Temperature/(°C)	Heating-up time/min	Holding time/min	Supplementary water/L	Solid-to-liquor ratio	MgSO4 dosage/(wt%)
Soda-AQ	19	11.25	155	120	120	5.772	1:5	\
Soda-oxygen 1	24	7.5	120	120	30	2.594	1:5	0.5
Soda-oxygen 2	26	7.5	120	120	30	2.594	1:5	0.5

2.3 Determination of the viscosity and volumetric isothermal expansivity (VIE) of the BL

VIE, which reflects the extent of carbonization and degree of dehydration, has a significant influence on the combustion performance. This parameter was determined as follows. (a) The BL was concentrated to 40%–60% solid content in a crucible, the volume of which was considered Vo (mL). (b) Approximately 2–3 g of the oven-dried sample was weighed as M (g), burned in a muffle furnace at 300 °C for 60 min, and then weighed once more after cooling as G1. (c) silica sand ρ (mL/g), considered as G2. VIE is defined as:

$$\mathrm{VIE} = \frac{[\mathrm{V_0} - (\mathrm{G2} - \mathrm{G1}) / \rho]}{\mathrm{M}} (\mathrm{mL} / \mathrm{g})$$

The viscosity of each BL sample was determined using an SNB-1 viscometer (Hengping Company, China).

2.4 Component analysis of the BL

The ash obtained from the combustion of the BL was analyzed to obtain the proportion of inorganic matter to organic matter following the standard protocol for "Analysis of Soda and Sulfate BL" (TAPPI T625 cm-14). Silicon content was determined using TAPPI T632-11 "Analysis of sodium silicate." for which inorganic was calcined at 1000°C after treatment with concentrated sulfuric acid and nitric acid.

2.5 TG-DTG Method

Experiments were conducted on a Jupiter thermogravimetric analyzer (STA 449C; Netzsch, Germany) at a heating rate of 20 °C/min within the temperature range of 50–800°C. High-purity nitrogen was used as the carrier gas at a flow rate of 20 mL/min. Approximately 30 mg of BL was placed in the ceramic crucible for each analysis.

2.6 Scanning electron microscopy (SEM) Method

Imaging with a scanning electron microscope (VEGA-3SBH, TESCAN) was performed. Samples were air-dried prior to imaging and mounted on aluminum stubs using conductive carbon tape. The stubs were then sputter-coated with approximately 3 nm of gold-sputter coater (SBC-12, KYKY Technology Co., Ltd., Beijing, China). Imaging was performed with a beam acceleration voltage of 10 kV.

3.Results and Discussion

3.1 Analysis of pulp properties

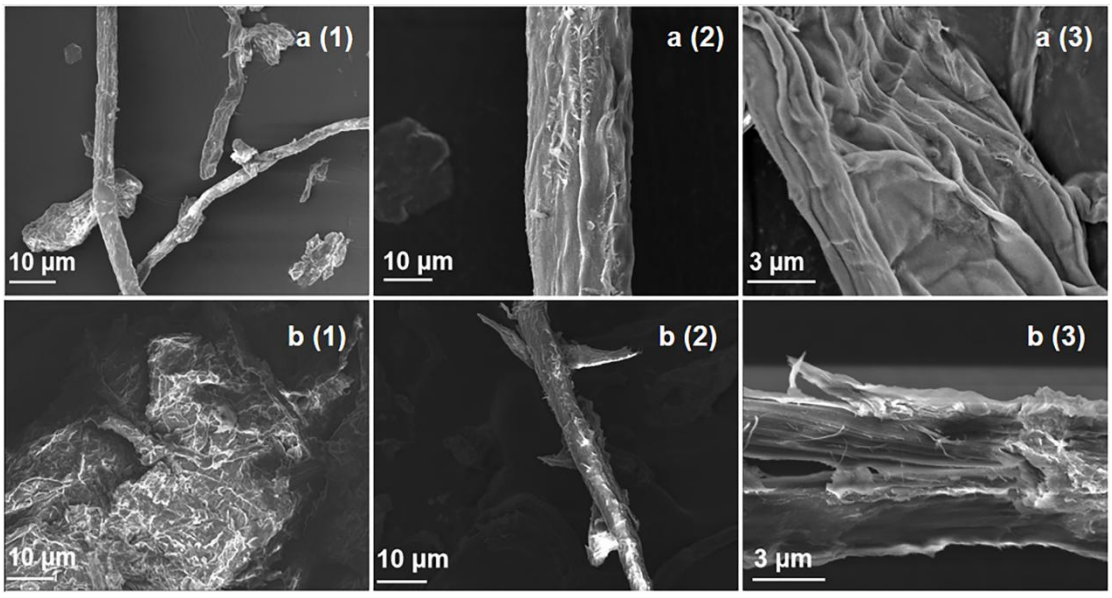

Fig. 1 SEM images of a(1)–a(3) soda-AQ pulp and b(1)–b(3) soda-oxygen pulp.

As shown in Fig. 1a, the cellulose surface of the pulp obtained from soda-AQ retained its the

tubular structure. Compared with that obtained from soda-AQ, the wheat straw cellulose obtained from soda-O_2 swelled and cracked, as shown in Fig. 1b. Lignin acts as an adhesive that can glue cellulose and hemicellulose together. The synergistic effect between oxygen and alkali could loosen the original cellulose structure and expose the adjacent micro-fibers on the surface, as shown in Fig. 1 b(2) and b(3).

Because oxygen is added to the pulping process, synergistic effects between oxygen and the alkali could remove large amounts of lignin in the pulp. Compared with soda-AQ, soda-O_2 shows distinct advantages in terms of pulp brightness and Kappa numbers. Moreover, more polysaccharides in the pulp could be depolymerized to simple sugars, which subsequently dissolve into the BL (Jiang et al. 2018; Sun et al. 2021). Therefore, the pulp obtained from soda-O2has higher brightness and a lower pulp yield than the pulp obtained from soda-AQ. However, the rejection yield of soda-oxygen was higher than that of soda-AQ. It may be result from the tight structure of the straw stalk. Tightly bundled fibers in stalk can not sufficiently dispersed by the low temperature in soda-oxygen cooking(cooking temperature is 120 ^{o}C). Thus, the oxygen and alkali may be unable to access to this part of the straw, resulting in higher rejection yields compared with that in soda-AQ.

Table 3. Pulp properties

	Pulp yield (%/dry material)	Reject yield (%/dry material)	Brightness	Viscosity (mL/g)	Kappa number
Soda-AQ	50.67	0.35	38.8	693	13.40
Soda-oxygen 1	45.49	3.3	43.5	520	12.34
Soda-oxygen 2	43.88	3.3	42.3	598	12.60

3.2 Effect of total solid content on the viscosity of the BL

The viscosity of BL is one of the major factors of alkali recovery. It can guide the maximum total solid content (TS) of BL injected into the recovery furnace. Generally, the viscosity of BL keep at 300 to 500 mPa·s at approximately 105℃ (Adams and Frederick 1987) to make sure the jet spraying fluently. So, the TS of BL should be concentrated to increase the heat efficiency of the furnace under reasonable viscosity range. The viscosity of BL at 90 °C was selected because this temperature is similar to the export temperature from evaporation concentrator which can reflects real industrial conditions (Wang et al. 2020). As shown in Fig. 2, the viscosity of the BL clearly increased with increasing solid content. As the level of soluble salt decreased to a critical value, the inorganic salt began to precipitate, and the viscosity of the BL uniformly increased (Lu et al. 2020). However, when the viscosity of the BL increased to a certain extent, the solubility of inorganic salt (such as sodium silicate) increased because the precipitation of inorganic salt was hindered.

The curves shown in Figs. 2-4 have an obvious turning point as the viscosity increased. This point is referred to as the critical concentration point. The solid content of the BL gradually increased over 0-45%TS, and the viscosity of the BL did not exceed 100 mPa·s. When the solid content exceeded 45%, the viscosity of the BL increased rapidly with a small increase in solid content.

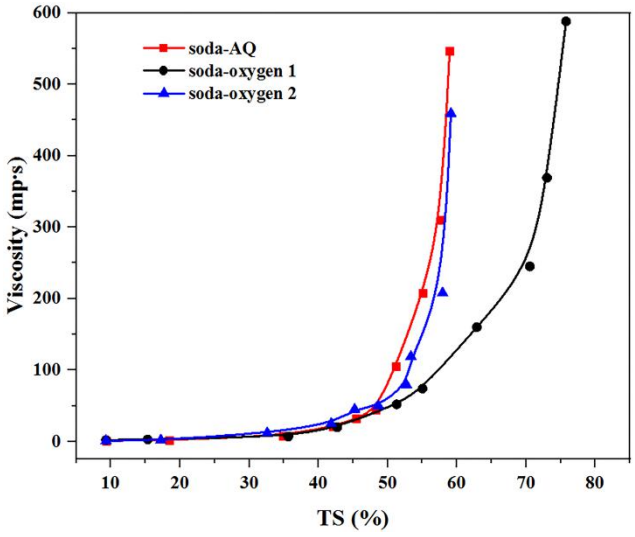

Fig. 2 Viscosity of the original black liquor with various total solid concentrations (TS) at 90 °C.

The turning point of the BL obtained from soda-O_2 appeared at a higher solid content compared with that obtained from soda-AQ pulping. The critical solid concentration of soda-oxygen 2 was approximately 55% while that of soda-oxygen 1 was approximately 65%.

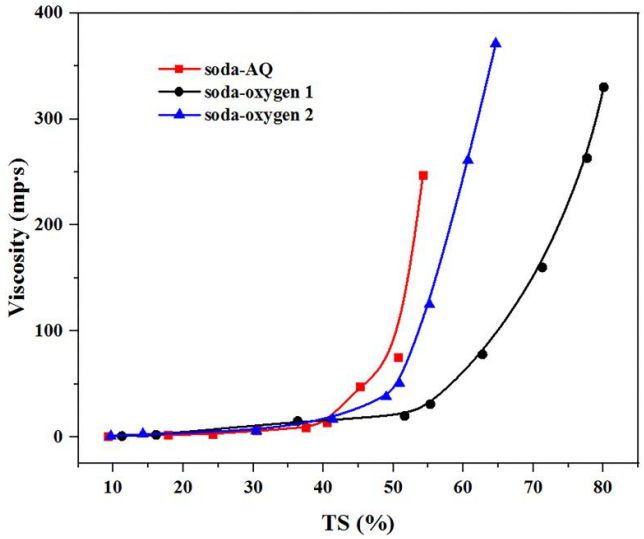

Fig. 3 Viscosity of the black liquor with various total solid concentrations (TS) at 90 °C after centrifugation.

Centrifugation could remove some suspended solids in the BL. After centrifugation, the turning point of the BL appeared at a higher solid content compared with that of the original BL. Interestingly, the solid content of the BL obtained from soda-oxygen 1 pulping exceeded 65%, which indicates that the

solid content of the BL has a significant influence on its viscosity. Compared with the original BL, centrifugation could remove suspended solids and increase the solid concentration of the BL by approximately 5% points.

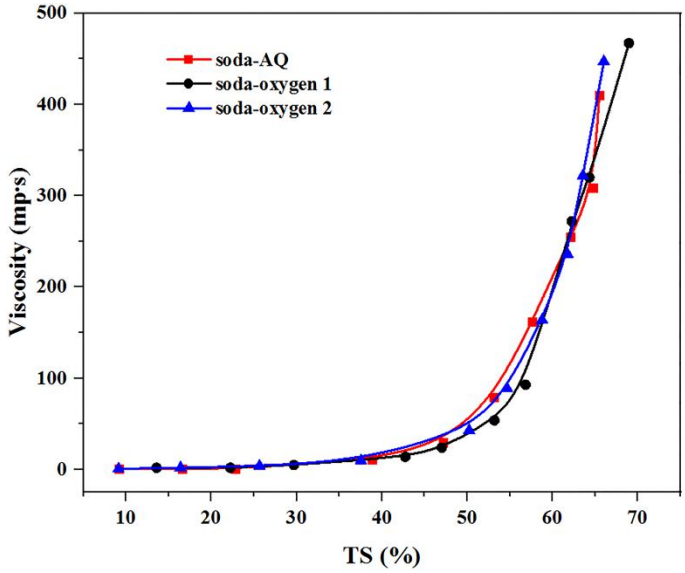

Fig. 4 Viscosity of the black liquor with various total solid concentrations (TS) at 90 °C after filtration.

Similar to centrifugal separation, filtration is also an effective method to remove suspended solids from BL. However, the thickening effect shown in Fig. 4 is not as good as that shown in Fig. 3. This finding demonstrates that the efficacy of filtration in removing suspended solids is poorer than that of centrifugal separation. Therefore, centrifugal separation is the better method to remove suspended solids from BL.

3.3 Effect of solid content on the value of VIE of the BL

As shown in Table 4, the silica content of the BL obtained from soda-O_2 was much less than that produced by soda-AQ pulping. The silica content of the BL obtained from soda-oxygen 1 pulping was approximately 6.2%, which is about 3% lower than that obtained from soda-AQ pulping. The content of silicon increased with increasing alkali charge during the soda-oxygen pulping (Cardoso et al. 2009). When the pH of the BL obtained from soda-oxygen 2 pulping was higher than 11.5, the silicon content of the BL was 9.42%. This finding shows that the pH of the BL has a decisive effect on the dissolution of silica in the liquid. Centrifugation and filtration have different effects on silica removal in environments of different pH (Xu et al. 2015). Specifically, the effects of higher pH (>15) on silica removal were not obvious, but over half of the silicon in the BL could be removed at pH less than 11.5.

Changes in the silica content and viscosity of the samples indicate that silica is one of the most important factors affecting the viscosity of the BL.Centrifugation and filtration could reduce the silicon content and the viscosity of back liquor. In this case, the silica dissolved in the original solution could

be converted into $HSiO_3^-$ in an alkaline environment. When the colloidal system is subjected to shear force, additional energy is needed to overcome the interaction between the glue core and the adsorption and diffusion layers, which leads to an increase in the viscosity of the BL (Guo et al. 2014).

Table 4. Components and VIE values of BL

	inorganic content [% (g/g of black liquor)]	organic content [% (g/g of black liquor)]	organic / inorganic	silica / in organic	values of VIE
soda-AQ pulping (PH>13)					
original solution	42.39	57.61	1.36	11.44	2.56
centrifugal solution	42.14	57.86	1.37	9.88	3.19
filtrate	41.62	58.38	1.40	9.17	3.57
soda-O21 (PH<10)					
original solution	48.80	51.20	1.05	6.20	1.63
centrifugal solution	47.29	52.71	1.11	3.27	3.95
filtrate	46.82	53.18	1.14	2.23	7.92
soda-O22 (11.5<PH<12)					
original solution	43.08	56.92	1.32	9.42	0.67
centrifugal solution	42.42	57.58	1.36	5.65	2.83
filtrate	41.88	58.22	1.39	5.49	4.82

After centrifugation and filtration, the VIEs of the BL decreased gradually and the thermal expansion properties of the BL improved remarkably (Liu et al. 2017). The silica content of the BL produced by soda-oxygen 1 pulping decreased rapidly under low pH environment. The VIE increased from 1.63 mL/g in the original solution to 3.95 mL/g in the centrifugal solution to 7.92 mL/g after the filtration. These results show that the thermal expansion properties of the BL could be improved appreciably with the removal of silicon.

The results also showed that silicon content has a significant influence on the VIE of the BL. Silicon content did not have a significant effect on the ratio of inorganic matter to organic matter but affected VIE greatly. These findings reveal that silica plays a decisive role in the VIE of inorganic components.

3.4 DTG analysis

The pyrolysis process of wheat straw BL is usually divided into three stages. The evaporation of water and small-molecule organic matter mainly occurs in the first stage (<200 °C), while the pyrolysis of carbohydrates primarily occurs in the second stage (200–300 °C) (Leite et al. 2013). Compared with soda-oxygen pulping, soda-AQ is conducted under higher temperatures and longer reaction times. Thus, part of the cellulose in wheat straw is decomposed to sugars, which subsequently dissolve into the BL (Liu et al. 2011). As shown in Fig. 5, the weight loss rate of the BL obtained from soda-AQ is much higher than that obtained from soda-O_2 in the second stage of pyrolysis.

The cracking of lignin cracking mainly occurs in the third stage of pyrolysis (300–650 °C). Unlike in soda-AQ pulping, oxygen is used in soda-oxygen pulping. Thus, the latter has stronger deconstructive effects on lignin than the former at a similar delignification level. During soda-oxygen pulping, phenolic hydroxyl and *p*-hydroxyphenyl have occurred more selective structure changes, and they could be oxidized into carbonyl groups. Larger amounts of the β-O-4 structure are retained during soda-oxygen pulping (Wu et al. 2013). The BL obtained from soda-O2presents better pyrolysis characteristics and releases more volatile gases at lower temperatures compared with the BL obtained from soda-AQ pulping (Li et al. 2010; Li et al. 2015).

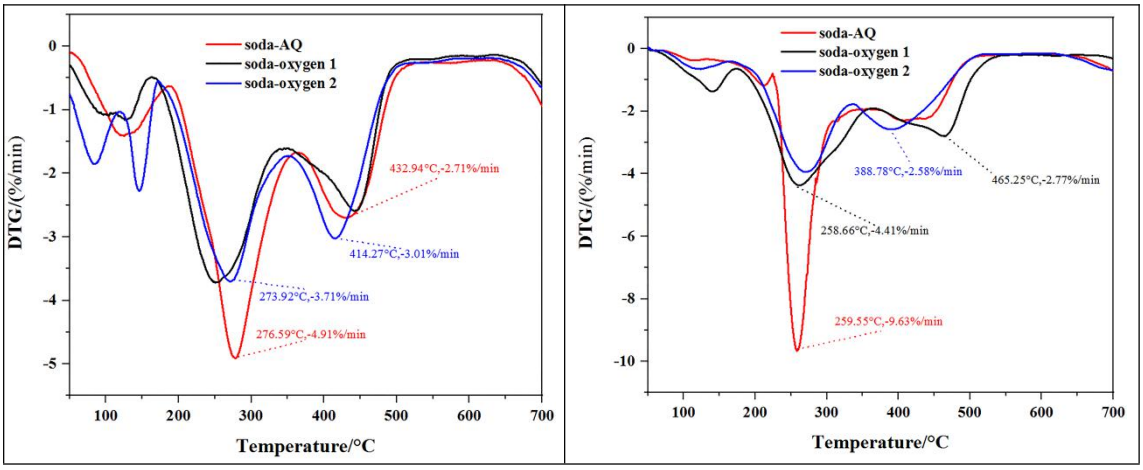

Fig. 5 DTG curves with derivative mass losses of black liquor (BL) samples at a heating rate of 10 K·min^{-1}: (a) Organic BL, (b) BL filtered by a micro-filtration membrane.

The pyrolysis of the BL obtained from soda-O2occurs at a lower temperature compared with that of the BL obtained from soda-AQ pulping. As the alkali charge increases during the pulping process, carbonyl groups obtained from phenolic hydroxyl and *p*-hydroxyphenyl concurrently increase. Thus, the BL obtained from soda-oxygen 2 is easier to pyrolyze than that obtained from soda-oxygen 1.

After filtration, macromolecular carbohydrates, macromolecular lignin, and SiO_2-lignin-carbohydrate groups are blocked on the filter membrane. Thus, their content in the BL is obviously decreased and the thermal stability of the filtrate is less than that of the original solution (Liu et al. 2016). Therefore, the maximum degradation rate of the filtrate is higher than that in the original solution in the second (200–300 °C) and third (300–650 °C) stages of pyrolysis.

Conclusion

The silica removal, viscosity reduction, and thermal properties of the BLs derived from the soda-AQ and soda-O2of wheat straw were evaluated.

Firstly, soda-O2can deposit silica in the pulp, which largely reduces the silicon content and viscosity of the BL. Compared with that during soda-AQ, over 45% silicon is removed from the BL during soda-O2under similar delignification levels with lower cooking temperature. The pulp obtained from

soda-O2presents higher brightness, and lower Kappa numbers.

Secondly, the TS concentration of soda-oxygen BL was easily concentrated by up to 50% while the TS concentration achieved by soda-AQ was only 45%. Moreover the viscosity of the BL obtained from soda-O2was lower than that of the BL obtained from soda-AQ at the same solid concentration. Moreover, the viscosity of the pulp could decrease from 693 mg/L to 520 mg/L.

The last but not last, simple centrifugation and membrane filtration could further removed SiO_2 and the TS concentration of soda-oxygen BL could be increased to 55%–60% at 300 mp·s. Compared with membrane filtration, centrifugation has more effective SiO_2 removal.

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Declaration of interests

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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Figures

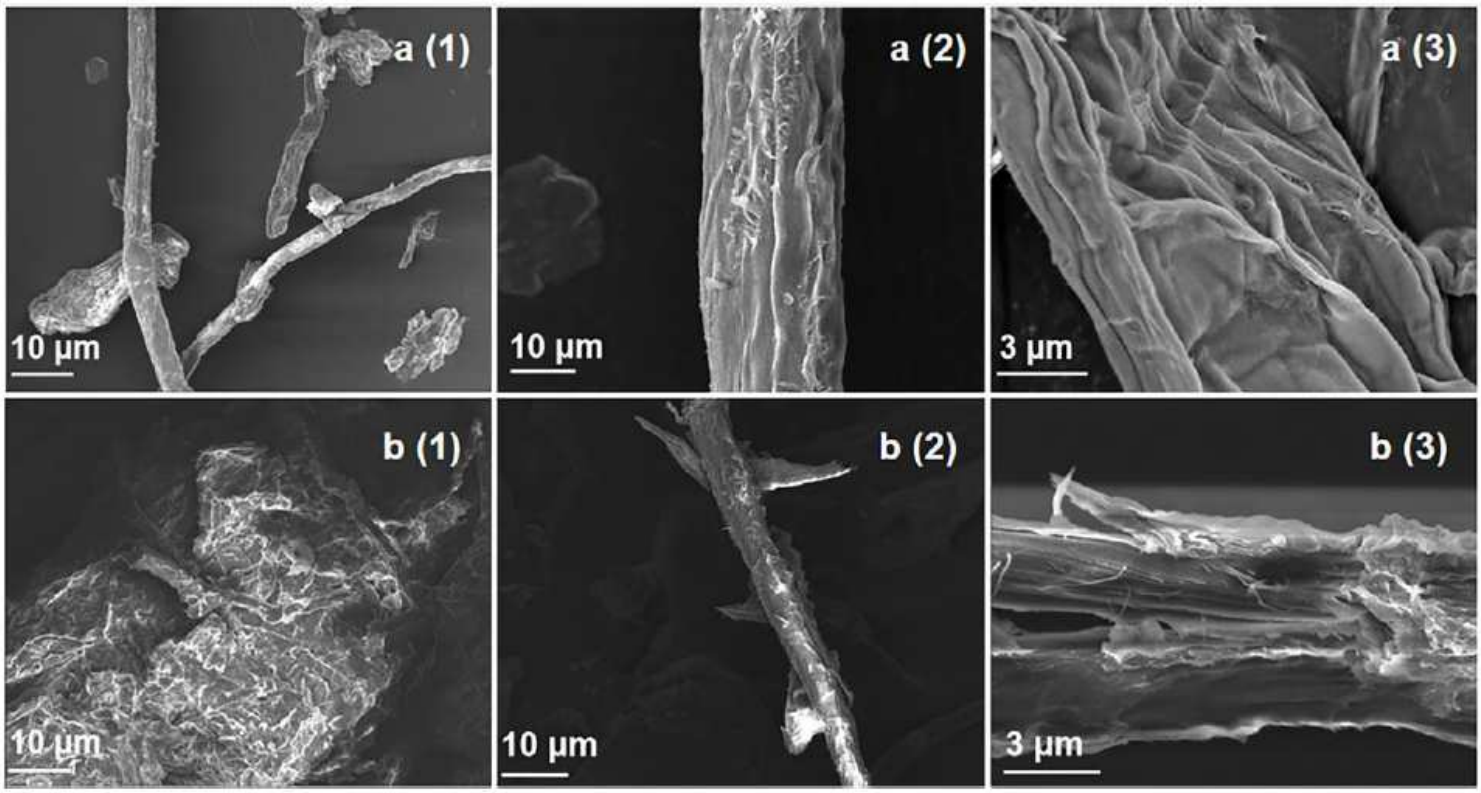

Figure 1

SEM images of a(1)–a(3) soda-AQ pulp and b(1)–b(3) soda-oxygen pulp.

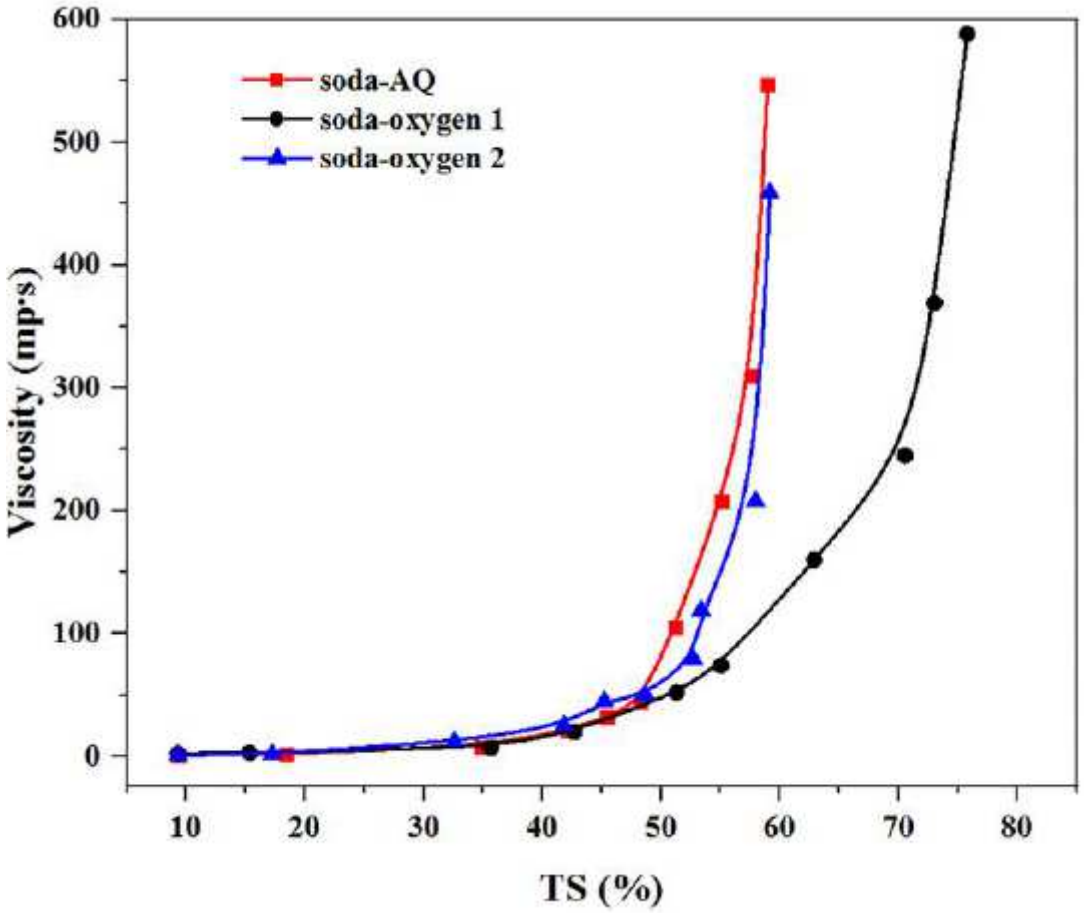

Figure 2

Viscosity of the original black liquor with various total solid concentrations (TS) at 90 °C.

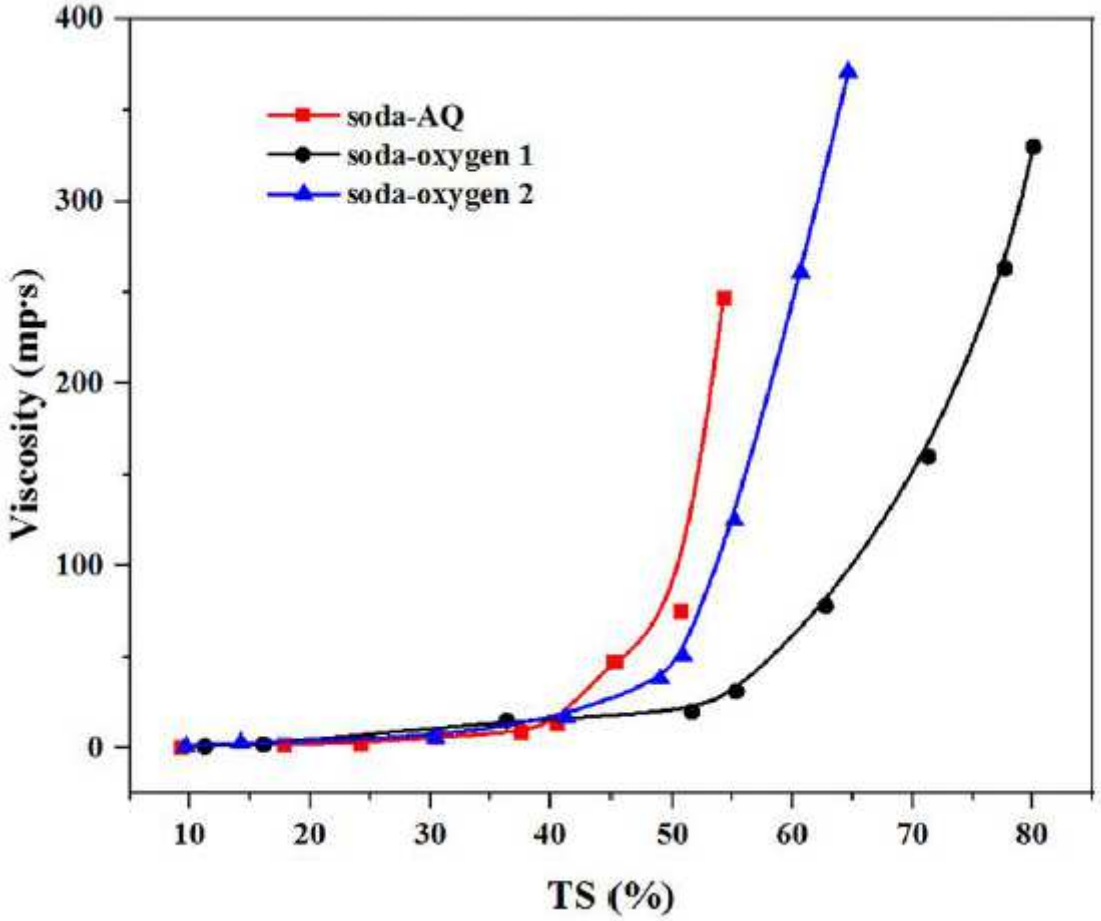

Figure 3

Viscosity of the black liquor with various total solid concentrations (TS) at 90 °C after centrifugation.

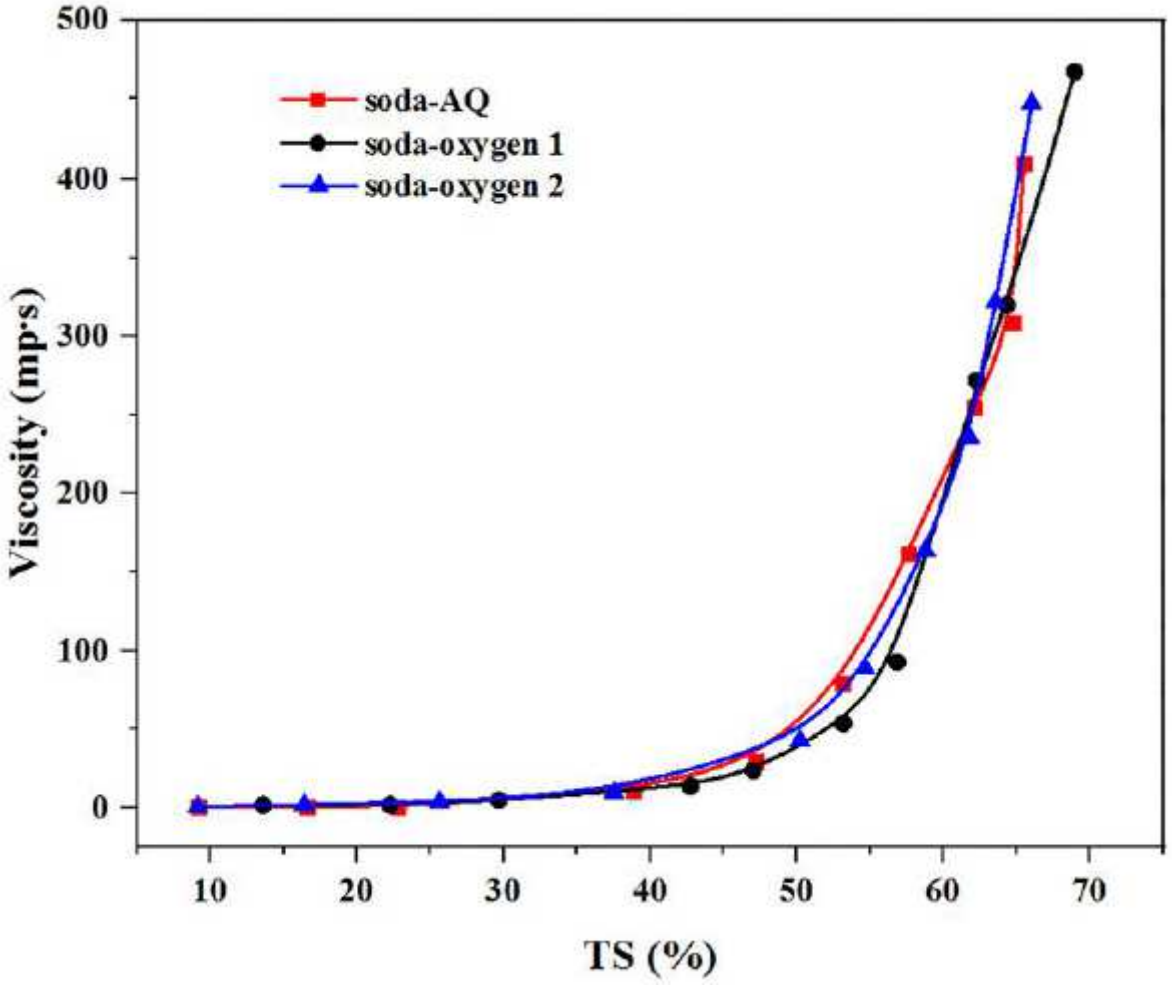

Figure 4

Viscosity of the black liquor with various total solid concentrations (TS) at 90 °C after filtration.

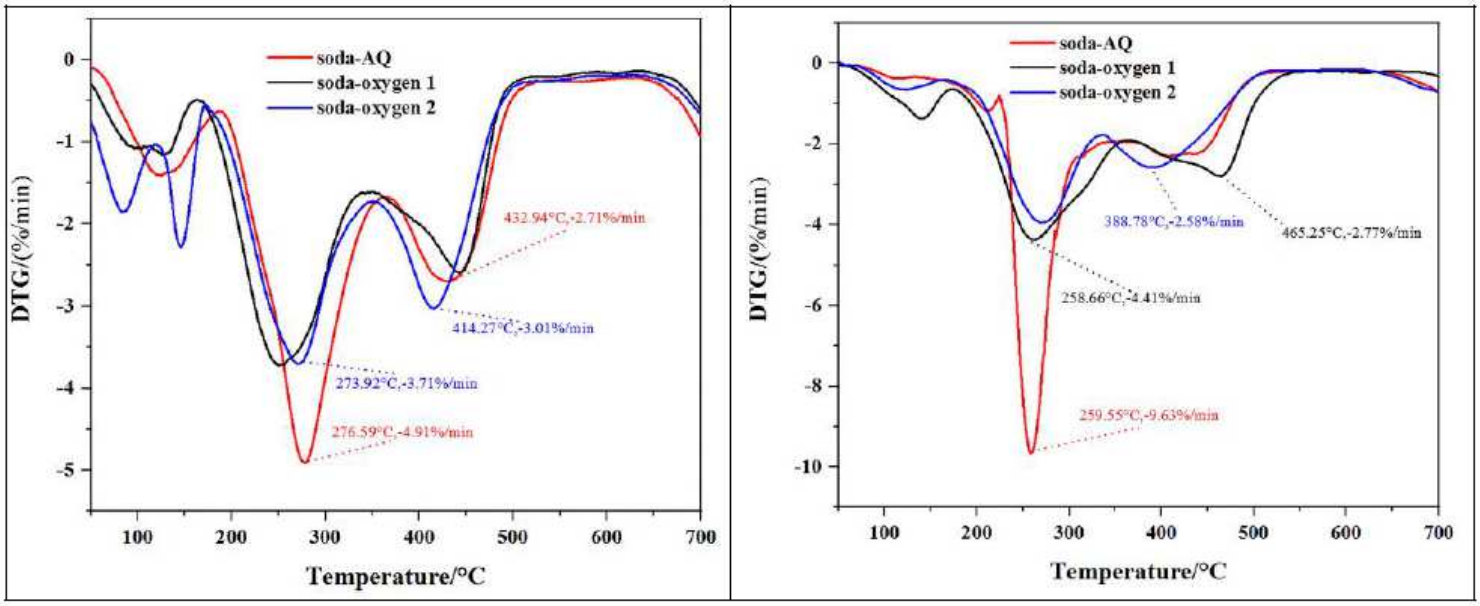

Figure 5

DTG curves with derivative mass losses of black liquor (BL) samples at a heating rate of 10 K□min−1: (a) Organic BL, (b) BL filtered by a micro-filtration membrane.