

Bleaching Flax Roving With Poly(Acrylic Acid) Magnesium Salt As Oxygen Bleaching Stabilizer For Hydrogen Peroxide

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Abstract

Alkali-oxygen one-bath scouring and bleaching process of the flax roving was studied by using a new type of synthesized non-silicon oxygen bleaching stabilizer Poly(acrylic acid) magnesium instead of sodium silicate. Based on the analysis of the effects of single factors such as sodium hydroxide concentration, hydrogen peroxide concentration, temperature, time and the amount of the synthesized non-silicon oxygen bleaching stabilizer poly(acrylic acid) magnesium salt on the performance of the bleached flax roving, including the whiteness, the breaking tenacity, the capillary effect and the weight loss ratio. The optimal process for the application of the stabilizer was determined by orthogonal test, namely, hydrogen peroxide concentration 8.5 g/L, sodium hydroxide concentration 5 g/L, sodium bisulfite 3 g/L, sodium carbonate 3 g/L, the synthesized non-silicon oxygen bleaching stabilizer poly(acrylic acid) magnesium 5.5 g/L, scoured and bleached at 90 °C for 60 min, and the bath ratio was 25:1. Compared with the traditional oxygen bleaching stabilizer sodium silicate, it not only has good ability to inhibit the rapid decomposition of hydrogen peroxide, but also has the advantages of higher whiteness, higher capillary effect, good feel and breaking tenacity, and can effectively solve the "silicon scale" problem and improve the quality of flax products.

Introduction

Flax fiber has the advantages of good moisture absorption, high tenacity, high resistance to alkali, antibacterial and mildew prevention, health care, breathable, smooth and generous, known as the queen of fibers (Perremans et al. 2018; Pandey 2016, Dudarev 2020). Flax fiber is mainly composed of cellulose. Besides cellulose, raw flax also contains 20–30% non-cellulose components including pectin, hemicellulose, lignin, waxes, nitrogen material and ash, which impair wettability and dressing performances of the end products (Huang and Yu 2019; Moawia et al. 2016; Dalbaşı and Kayseri 2019; Biljana 2018). The lignin content is very high, making barks and hards not easy to remove in dyeing and finishing process, resulting in low dye-uptake, harsh feel, poor wrinkle resistance and other shortcomings (Bernava and Reihmane 2018; Testoni et al. 2018). So treatments on flax roving to remove these natural impurities are important and necessary, determining quality of the flax products (Aina and Skaidrite 2018; Kaur et al. 2016). The conventional processing of removing the impurities rely on being scoured with a mixture of sodium hydroxide and sodium carbonate, and bleached with hydrogen peroxide (Chaishome and Rattanapaskorn 2017). Among them, hemicellulose and pectin substances are hydrolyzed into oligosaccharides and sodium salts of acids. Nitrogen-containing substances are hydrolyzed to sodium salt of amino acid; Lipid wax can be removed by saponification and emulsification. Lignin, which changes the least during scouring, cannot be directly decomposed into simple small molecule compounds, but produces other substances that are insoluble in alkali and deposited on the fiber, which need to be removed in bleaching (Ohmura, Yokoyama, and Matsumoto 2008; Heli, Anna, and Markku 2009). In actual production, in order to improve production efficiency, scouring and bleaching of the flax roving are usually combined, which is beneficial to reduce fiber damage and improve the environmental conditions of wet spinning. Alkali-oxygen one-bath scouring and bleaching process of flax roving is

usually carried out by hydrogen peroxide (H_2O_2) under alkaline condition (Abhishek et al. 2017; Lazic et al. 2021). H_2O_2 can produce perhydroxyl anions (HOO^-) under the strong alkaline condition, which is also responsible for swelling flax cellulose and removing lignin and other impurities. In addition, the hydroxyl free radical and perhydroxyl free radical produced by decomposition of H_2O_2 also play a role for it (Zeronian and Inglesby 1995; Fakin and Ojstrsek 2008). In the scouring and bleaching working solution of flax roving, the presence of heavy metal ions can accelerate the decomposition of H_2O_2 to produce the effective bleaching component, which not only reduces the bleaching effect and increases the consumption of H_2O_2 , but also causes serious degradation of cellulose under the condition of high temperature and strong alkali. Therefore, it is necessary to add a certain amount of oxygen bleaching stabilizer (OBS) in the process to inhibit excessive catalytic decomposition of H_2O_2 by heavy metal ions (Liu et al. 2018). So that H_2O_2 can maintain a high oxidation capacity in the total bleaching time, without wasting of the active components and damaging to the fiber excessively.

Sodium silicate (commonly known as water glass), is a traditional stabilizer for flax fiber bleaching, its structure is similar to that of a porous sponge, and it can absorb the Cu^{2+} and Fe^{3+} in the bleaching bath. Sodium silicate and magnesium ions form magnesium silicate, which rarely dissolves in water, and acts as a protective colloid. Under the influence of temperature and time, the protective film gradually decomposes, releasing HOO^- in a linear rather than geometric order, so as to achieve mild bleaching (Fakin et al. 2006; Laziz et al. 2020). But it can form insoluble salt with Ca^{2+} and Mg^{2+} during the production, that is so called silica scale. The silica scale deposited on the flax roving easily causes the rough handle, yellowing and other defects. The silica scale deposited on the equipment will bring difficulties to the cleaning (Sharnina, Vladimirtseva, and Odintsova 2019; Fu, Wu, and He 2008; Topalovic, Nierstrasz, and Warmoeskerken 2010). To solve the problem of silica scale, the research and application of non-silicon stabilizer is especially important.

Non-silicon OBS mainly include phosphonates and macromolecule compounds, such as inorganic phosphate, organic chelating agent, protein degradation products and organic polyphosphate, etc. (Chen 1997, 2011; Zhang 2008). Phosphonates OBS has not been recommended to continue development since it causes water quality deterioration.

In this paper, non-silicon OBS poly(acrylic acid) magnesium (PAA-Mg) was synthesized by solution polymerization, then it is applied to alkali-oxygen one-bath scouring and bleaching process of the flax roving as a stabilizer instead of sodium silicate. At the same time, the bleaching effect was compared with other stabilizers, including sodium silicate and OBS DM-1403. Experiment results proved that the non-silicon OBS PAA-Mg had little damage on flax roving, and PAA-Mg as the stabilizer has no pollution to environment and no affinity with the flax fiber, easy to wash out after bleaching. Therefore, the application of this synthesized non-silicon oxygen bleaching stabilizer PAA-Mg on flax roving bleaching process had vital significance. It can replace sodium silicate in the bleaching process, and solve the problem of silica scale.

Experimental

Materials

The flax roving was kindly provided by Qiqihar Jinya Group (China). Acrylic acid (chemically pure) as a monomer and potassium persulfate as an initiator were purchased from Tianjin Cameo Chemical Reagent Co. Ltd. (China). Hydrogen peroxide (30% w/w), magnesium sulfate, sodium hydroxide, potassium permanganate, sulfuric acid, sodium carbonate, sodium silicate and sodium bisulfite were purchased from Tianjin Kaitong Chemical Reagent Co. Ltd. (China). Unless otherwise stated, all other chemicals were of analytical grade.

Synthesis of non-silicon OBS PAA-Mg

This PAA-Mg stabilizer was synthesized with solution polymerization. Potassium persulfate (5% of the mass of acrylic monomer) and distilled water were added to a three-necked flask containing an electric stirrer, thermometer and reflux condensing unit. Stirring and heating until the initiator potassium persulfate was completely dissolved, acrylic acid monomer (cons. 25%) and distilled water were added into the initiator solution. Reacted at 65 °C for 4 h, the light yellow viscous product was obtained. Finally added 2 g magnesium sulfate based on 10 g acrylic acid, continue to stirring for 1 h, then stopped the reaction, and non-silicon OBS was obtained (Liu and Lv 2021).

Scouring and bleaching of flax roving

One bath method of scouring and bleaching was adopted. The flax roving (2 g) was firstly washed with 2.5 g/L sulfuric acid in 250-ml erlenmeyer flask in a water bath and performed at 30 °C for 10 min with a bath ratio of 25:1, then washed completely. The washed flax roving was immersed in 50 ml bleaching bath which contained [2, 4, 6, 8, 10, 12] g/L H₂O₂, 3 g/L Na₂CO₃, [3, 4, 5, 6, 7, 8] g/L NaOH, [0, 1, 2, 3, 4, 5] g/L NaHSO₃, JFC 1 g/L, [1, 2, 3, 4, 5, 6] g/L PAA-Mg, bleaching at [60, 70, 80, 90, 95, 100] °C for [15, 30, 45, 60, 75, 90] min with a bath ratio of 25:1. After scouring and bleaching, the samples were removed from the bleaching bath and then rinsed in hot and cool water, respectively, then oven dried under 60 °C for 30 min.

Whiteness measurement

The whiteness of the flax roving was determined on a spectrophotometer (YQ-Z-48A whiteness color tester, China) using a D₆₅ illuminant and 10° standard observer. Refer to Textile Fiber Whiteness Chroma Test Method GB/T 17644 - 2008 for determination. Each sample was about 80 mm long and was closely arranged on the pallet of the whiteness color tester. Each sample was rotated in four different directions for measurement, and the average value was calculated.

Determination of capillary effect

Refer to Textiles - Test method for capillary effect FZ/T 01071-2008 for determination, using YG(B)871 Capillary effect tester (China). Potassium dichromate solution was added to the tester, the upper end of a

certain length of yarns was fixed on the support of the tester, the lower end was clamped with a small clip, and the small clip was completely immersed in the liquid, and then started measuring, recorded the height of the liquid rising within 30 min. Tested 3 times and calculated the average value.

Breaking tenacity measurement

Breaking tenacity testing was carried out in accordance with GB/T 3923.1–2013, using an YG601 electronic single yarn strength tester (China), under standard laboratory conditions (20 ± 2 °C; $65 \pm 3\%$ relative humidity), with a gauge-length of 250 mm and a speed of 250 mm/min. Constant stretching, and the average value of 15 measurements was calculated.

X-ray diffraction measurements

Refer to Determination of crystal cells parameters of minerals – Powder X - ray diffraction method EJ/T 553–1991 for determination, using D8 X - ray diffractometer (BRUKER-AXS,Germany). The test conditions were: the tube voltage was 40 kV, the tube current was 30 mA, the scanning speed was $2^\circ/\text{min}$, and the 2θ scanning range was $5\text{--}45^\circ$.

Results And Discussion

Factors affecting the bleaching performance on flax roving

H_2O_2 concentration

In the one-bath scouring and bleaching of flax roving, the perhydroxyl anions produced by the decomposition of hydrogen peroxide can interact with some components of cellulose to achieve the purpose of discoloration and removal of impurities. If the concentration of hydrogen peroxide is too low, it can not reach the bleaching effect, can not meet the production requirements. If the concentration of hydrogen peroxide is too high, although it can achieve a very high whiteness, but because of its oxidation, the flax fiber will be seriously damaged. Therefore, strict control of the concentration of hydrogen peroxide is one of the key conditions in scouring and bleaching.

Figure 1 shows the effect of H_2O_2 concentration on whiteness, breaking tenacity, weight loss ratio and capillary effect of flax roving bleached using 2, 4, 6, 8, 10, 12 g/L H_2O_2 , 4 g/L NaOH, 4 g/L synthesized non-silicon OBS PAA-Mg $\times 3$ g/L Na_2CO_3 and 3 g/L NaHSO_3 under 90 °C for 60 min. Apparently, with the increase of hydrogen peroxide concentration, the whiteness of flax roving gradually improved and the breaking tenacity gradually decreased (Fig. 1a), the weight loss ratio increased gradually and the capillary effect fluctuated but not much (Fig. 1b). Such a relationship between bleaching properties and H_2O_2 concentration may be ascribed to increase in the amount of active oxidation species when hydrogen peroxide concentration increased (Yu, et al. 2018). However, it was also pointed out that the whiteness and weight loss ratio of the flax roving remained almost constant when the concentration of H_2O_2 was over 8 g/L, but the breaking tenacity decreased greatly. This may be associated with the excessive

decomposition of H_2O_2 , which contributed little to the bleaching result, on the contrary, the flax fiber was severely damaged. Therefore, the concentration of hydrogen peroxide should be selected about 8 g/L.

NaOH concentration

In one-bath scouring and bleaching process of flax roving, alkali is another important factor, which helps to the removal of impurities such as pectin, nitrogen containing and waxes. And the lignin attached to the surface of the fibers will be partially sulfonated to remove by sodium bisulfite under the presence of strong alkali. At the same time, the addition of alkali can ensure that the working liquid remains within a certain pH value range, promoting H_2O_2 decomposition to produce more active components, in order to remove impurities and improve the whiteness.

Figure 2 shows the effect of NaOH concentration on whiteness, breaking tenacity, weight loss ratio and capillary effect of flax roving bleached using 8 g/L H_2O_2 , 3, 4, 5, 6, 7, 8 g/L NaOH, 4 g/L PAA-Mg, 3 g/L Na_2CO_3 , and 3 g/L $NaHSO_3$ under 90 °C for 60 min. As can be seen from Fig. 2, with the increase of sodium hydroxide concentration, the two indexes of whiteness and breaking tenacity of the bleached flax roving showed opposite trends, the whiteness increased gradually while the breaking tenacity decreased gradually (Fig. 2a), the weight loss ratio and capillary effect of flax roving increased with the increase of sodium hydroxide concentration (Fig. 2b). The reason was that with the increase of concentration of sodium hydroxide, the impurities which influenced whiteness and absorption of flax were removed more thoroughly, fibrous tissue became uniform, hygroscopicity enhanced, so capillary effect and whiteness gradually improved. But when pH value increased to a certain degree, the decomposition of hydrogen peroxide was intense, and the interaction with the fiber was enhanced, caused the fiber damage, so led to the fiber strength decreased obviously. Therefore, considering the influence of alkali concentration on the whiteness and breaking tenacity, as well as the requirements of the later spinning process, the sodium hydroxide concentration should be controlled at about 5 g/L.

$NaHSO_3$ concentration

In the scouring and bleaching of flax roving, the addition of $NaHSO_3$ can prevent the cellulose from being excessively oxidized by hydrogen peroxide and the oxygen in the air, and can form lignosulfonate with lignin, increasing its solubility in alkali and promoting its shedding from flax fiber. Figure 3 shows the effect of $NaHSO_3$ concentration on whiteness, breaking tenacity, weight loss ratio and capillary effect of flax roving bleached using 8 g/L H_2O_2 , 5 g/L NaOH, 4 g/L PAA-Mg, 3 g/L Na_2CO_3 and 0, 1, 2, 3, 4, 5 g/L $NaHSO_3$ under 90 °C for 60 min.

As can be seen from Fig. 3, the whiteness of the bleached flax roving decreased with the increase of sodium bisulfite dosage, but it decreased not obviously after reaching a certain degree. After scouring and bleaching, the breaking tenacity was enhanced, but still maintained at a high level, indicating that the addition of sodium bisulfite had a certain effect on the protection of flax fiber. Considering the whiteness and breaking tenacity of the sample comprehensively, the appropriate amount of sodium bisulfite was 3g/L.

Bleaching time

Treatment time can affect bleaching effect of the flax roving, if the bleaching time is too short, the impurities will be not removed thoroughly, and if bleaching time is too long, although can achieve excellent whiteness, but the extension of time will increase the reaction of hydrogen peroxide and cellulose in the fiber, and even cause fiber damage. In addition, it will cause a waste of energy. Figure 4 shows the effect of time on whiteness, breaking tenacity, weight loss ratio and capillary effect of flax roving bleached using 8 g/L H_2O_2 , 5 g/L NaOH, 4 g/L PAA-Mg, 3 g/L Na_2CO_3 and 3 g/L NaHSO_3 under 90 °C for 15, 30, 45, 60, 75, 90 min.

As can be seen from Fig. 4, with the prolonging of bleaching time, the weight loss ratio and capillary effect of the flax roving increased continuously, the reason was that the impurities in the flax roving were gradually removed, the crystallinity of the fiber became uniform, and the moisture absorption was enhanced. It was also possible that the fibers have been excessively damaged and the weight loss ratio continued to increase (Fig. 4b). The whiteness of flax roving improved obviously with prolonging of bleaching time, but after bleaching 60 min, and the whiteness increased not obviously, on the contrary, the breaking tenacity of the fiber decreased significantly (Fig. 4a). The main reason was that the effective bleaching components in the working solution reacted more thoroughly with the impurities in the fiber with the bleaching time increase, at the same time, the damage degree of the fibers increased either. However, after reached a degree similar to saturation, even if the bleaching time was extended, the bleaching effect would not be significantly improved, but only the consumption increased. Therefore, considering the bleaching effect and production cost, the bleaching time should be 60 min.

Bleaching temperature

It is very important to control the bleaching temperature of flax roving to achieve good bleaching effect. Figure 5 shows the effect of time on whiteness, breaking tenacity, weight loss ratio and capillary effect of flax roving bleached using 8 g/L H_2O_2 , 5 g/L NaOH, 4 g/L PAA-Mg, 3 g/L Na_2CO_3 and 3 g/L NaHSO_3 under 60, 70, 80, 90, 95, 100 °C for 60 min.

As can be seen from Fig. 5, when the bleaching temperature varies in the range of 60 - 100 °C, with the increase of the bleaching temperature, the whiteness (Fig. 5a), weight loss ratio and capillary effect (Fig. 5b) of flax roving increased continuously, but its breaking tenacity decreased obviously (Fig. 5a). This was mainly because higher temperature helped the decomposition of hydrogen peroxide, and the produced effective bleaching components reacted with the impurities in the flax roving, so that the impurities were removed thoroughly. But above 90°C, the hydrogen peroxide decomposed so fast that its active component HOO^\cdot was released into the air before it could interact with the impurities, making the increase in whiteness less noticeable. The decrease in breaking tenacity indicated that the fibers had been damaged. Considering the bleaching effect and production cost, the bleaching temperature should be at about 90 °C.

PAA-Mg concentration

The synthesized non-silicon OBS PAA-Mg is a product of oligomer acrylic acid compounding with magnesium salt, which can effectively inhibit the catalysis of metal ions, thereby inhibiting the ineffective decomposition of hydrogen peroxide. The purpose of using this stabilizer is to solve the silicon scale problem caused by traditional stabilizer sodium silicate. Fig. 6 shows the effect of time on whiteness, breaking tenacity, weight loss ratio and capillary effect of flax roving bleached using 8 g/L H₂O₂, 5 g/L NaOH, 1, 2, 3, 4, 5, 6 g/L PAA-Mg, 3 g/L Na₂CO₃, and 3 g/L NaHSO₃ under 90 °C for 60 min.

As can be seen from Fig. 6, with the increase of PAA-Mg concentration, the whiteness increased gradually, the breaking tenacity decreased first and then increased slightly (Fig. 6a). The weight loss ratio of the bleached flax roving decreased gradually, and the decomposition rate of H₂O₂ decreased gradually (Fig. 6b). The reason was that when the concentration of PAA-Mg was lower, it also had less stabilizing effect on hydrogen peroxide, and the violent decomposition of H₂O₂ were not controlled, so that some effective component escaped into air without time to interact with the impurities, and the good bleaching effect was not achieved. With the increase of PAA-Mg concentration, it also increased the stability of H₂O₂ decomposition, the active component reacted with the impurities in the fiber, and achieved the best bleaching effect. When the concentration of PAA-Mg was too high, which over-inhibited the decomposition of H₂O₂, thus the whiteness reduced and the breaking tenacity increased slightly. Therefore, the concentration of PAA-Mg should be about 5 g/L.

Orthogonal optimization of application process of non-silicon OBS PAA-Mg

Based on the influences of single factor on bleaching effects of the flax roving, the optimal scouring and bleaching process of the synthesized non-silicon OBS PAA-Mg was determined by orthogonal experiment of L₉ (3⁴). Table 1 shows the orthogonal experiment and range analysis.

Table 1 Orthogonal experiment and results

No.	A (Temperature) °C	B (H ₂ O ₂ Con.) %	C (NaOH Con.) %	D (OBS Con.) g/L	Whiteness %	Breaking tenacity cN/tex
1	85 (1)	7.5 (1)	4.5 (1)	4.5 (1)	64.29	60.65
2	85 (1)	8.0 (2)	5.0 (2)	5.0 (2)	66.35	54.10
3	85 (1)	8.5 (3)	5.5 (3)	5.5 (3)	66.81	62.60
4	90 (2)	7.5 (1)	5.0 (2)	5.5 (3)	65.54	57.65
5	90 (2)	8.0 (2)	5.5 (3)	4.5 (1)	65.93	60.95
6	90 (2)	8.5 (3)	4.5 (1)	5.0 (2)	66.75	55.15
7	95 (3)	7.5 (1)	5.5 (3)	5.0 (2)	63.70	59.70
8	95 (3)	8.0 (2)	4.5 (1)	5.5 (3)	66.49	63.40
9	95 (3)	8.5 (3)	5.0 (2)	4.5 (1)	67.11	56.00
k ₁	65.82	64.51	65.84	65.77		
k ₂	66.07	66.26	66.33	65.60		
k ₃	65.77	66.89	65.48	66.28		
R	0.30	2.38	0.85	0.68		
k ₁ '	59.12	59.33	59.73	59.20		
k ₂ '	57.92	59.48	55.92	56.32		
k ₃ '	59.70	57.92	61.08	61.22		
R'	1.78	1.56	5.16	4.90		

It can be seen from Table 1, for the whiteness, the order of primary and secondary factors was B <math>\lt;math>C <math>D A, the optimization scheme was A₂ B₃ C₂ D₃. For the breaking tenacity, the order was C <math>\lt;math>D <math>A B, the optimization scheme was A₃ B₂ C₃ D₃. Although the analysis of the two indexes appeared different optimization process, but considering the influence of the evenness of flax roving on the test results, as well as the specific requirements of breaking tenacity in production practice, namely, the strength of the bleached flax roving should be within a reasonable range of proper spinning, and the requirement of whiteness should be appropriately reduced in order to prevent the flax fiber from being seriously damaged. On the basis of the above analysis, combined with the performance of flax fiber and the stability of hydrogen peroxide, the optimal technological conditions for the application of the synthesized non-silicon OBS PAA-Mg to the one-bath scouring and bleaching process of the flax roving were obtained

as follows: 30% H₂O₂ 8.5 g/L, NaOH 5 g/L, Na₂CO₃ 3 g/L, NaHSO₃ 3 g/L, PAA-Mg 5.5 g/L, scoured and bleached at 90 °C for 60 min.

The optimal process determined by orthogonal experiment was used for verification experiments, and the properties of the samples were determined. The results showed that the whiteness was 68.75%, and the breaking tenacity was as high as 113.4 cN/tex, which was much higher than any one of the results on single factor and orthogonal experiments, so the optimized process was feasible.

Comparison of application effects of different stabilizers

The performance of the treated flax roving using various stabilizers was tested, and the results were shown in Table 2.

Table 2 Comparison of application effects of different stabilizers

Stabilizer	Whiteness (%)	Capillary effect (cm/30 min)	Breaking tenacity (cN/tex)
Sodium silicate	68.53 ± 0.20	14.4 ± 0.1	101.2 ± 6.7
DM-1403	67.39 ± 0.18	14.3 ± 0.2	109.5 ± 7.4
PAA-Mg	68.75 ± 0.25	15.4 ± 0.2	113.4 ± 7.8

As can be seen from Table 2, the application performance comparison of the three stabilizers in scouring and bleaching one-bath process to flax roving shows that the synthesized non-silicon OBS PAA-Mg is the best no matter the whiteness, the capillary effect or the breaking tenacity. DM-1403 is one of the substitutes to sodium silicate, which is a high molecular polymer containing hydroxyl, carboxyl and thiamine group. It is a non-silicon oxygen bleaching stabilizer that integrates micelle adsorption, inter-molecular and intra-molecular chelation. PAA-Mg belongs to the macromolecule anion stabilizer, the main component carboxylate radical of the poly(acrylic acid) molecule can chelate to the metal cation, and the component magnesium salt can easily form magnesium hydroxide colloid in the alkaline medium, the metal ions in solution mainly exist in the form of complex ions with negative charge, such as hydroxide. The complex ions are also easily adsorbed by magnesium hydroxide micelles and lose their catalytic activity. Most of the complex ions will be removed in the washing process and will not adhere on the fiber surface, so the treated flax roving has high capillary effect and good feel.

XRD of unbleached and bleached flax roving

The X-ray diffraction pattern was used to characterize the difference in crystal structure of flax roving before and after bleaching with different stabilizers. It can be seen from Fig. 7 that the X-ray diffraction curves and diffraction peaks of flax roving treated with different stabilizers are very similar to the sample untreated. The crystalline structure of flax cellulose is cellulose I β , the diffraction peaks of (100) plane and (010) plane appear at 14.83 and 16.04, respectively, and the diffraction peak of (110) plane emerge at 22.68 (French 2014, 2020). This is the basis for conclusion that the bleached flax roving has not changed

the crystal structure of the original cellulose, and the skeleton structure of the fibers was not damaged. The differences between the samples are that the intensity of all diffraction peaks in bleached flax roving is slightly weakened. The possible reason is that the flax fiber has been damaged to different degrees by the scouring and bleaching with various stabilizers, which leads to the slightly decrease of the crystallinity of flax fiber. It is concluded that the bleached flax fibers basically preserve natural crystal regions distribution.

SEM of unbleached and bleached flax roving

SEM examination of the flax roving was made with S-3400 Scanning Electron Microscope by Hitachi Co. (Japan). SEM images of the samples unbleached and bleached with different stabilizers were shown in Fig. 8. The conclusion is that the surface of flax fibers has changed significantly, the surface of the flax fiber became relatively clean and smooth, and the impurities were mostly removed, but the smoothness by using different stabilizers was inconsistency. There were lots of granular solids on the fiber surface (Fig. 8a), which may be the colloidal impurities existed in the flax fiber itself. The surface of the flax fiber bleached using traditional stabilizer sodium silicate was almost no granular solids (Fig. 8b), it implies that the impurities have been removed completely. Stabilizer DM - 1403 was an alternative product of sodium silicate, the surface of flax fiber was smooth and the impurities were removed thoroughly (Fig. 8c). The surface of flax fiber bleached using this synthesized PAA-Mg as stabilizer was smooth and the impurities were also removed completely (Fig. 8d). It showed that the synthesized non-silicon OBS PAA-Mg can replace sodium silicate and achieve the application effect of other substitutes.

Conclusions

In this study, the optimal process of the synthesized non-silicon OBS PAA-Mg in the scouring and bleaching of flax roving was determined, namely, 30% hydrogen peroxide 8.5 g/L, sodium hydroxide 5 g/L, PAA-Mg 5.5 g/L, sodium carbonate 3 g/L, sodium bisulfite 3 g/L, scoured and bleached at 90 °C for 60 min, the bath ratio 25:1. By using the synthesized non-silicon OBS PAA-Mg as the stabilizer, the bleached flax roving obtained higher whiteness, and the decrease of breaking tenacity was obviously reduced, the damage to the fiber was reduced. Especially the silicon scale problem caused by the use of sodium silicate as the stabilizer was solved, which is beneficial to the subsequent dyeing and finishing process. The future work is to study the effect of this synthesized non-silicon OBS PAA-Mg on the dyeing properties of flax fibers.

Abbreviations

PAA-Mg Poly(acrylic acid) magnesium

OBS Oxygen Bleaching Stabilizer

Declarations

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Contributions

Jie Liu and Wenqi Jiang performed the experiments. Jie Liu, Wenqi Jiang, Ling Sun and Chun Lv planned the experiments and analysed the data. Jie Liu supervised the work. Jie Liu wrote the manuscript. Jie Liu, Wenqi Jiang, Ling Sun and Chun Lv corrected the manuscript. The presented data was discussed by all authors prior to submission and all authors agreed to submit.

Ethical Statement

All authors state that they adhere to the Ethical Responsibilities of Authors. In addition, the work is compliance with ethical standards.

Conflicts of Interest Statement

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. All the authors listed have approved the manuscript enclosed.

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Figures

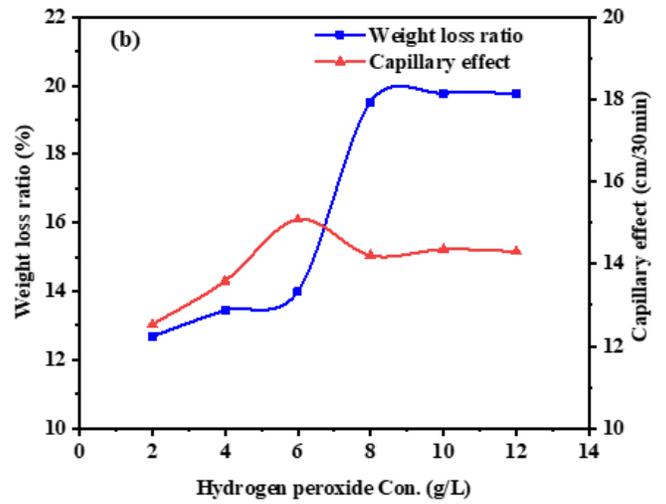
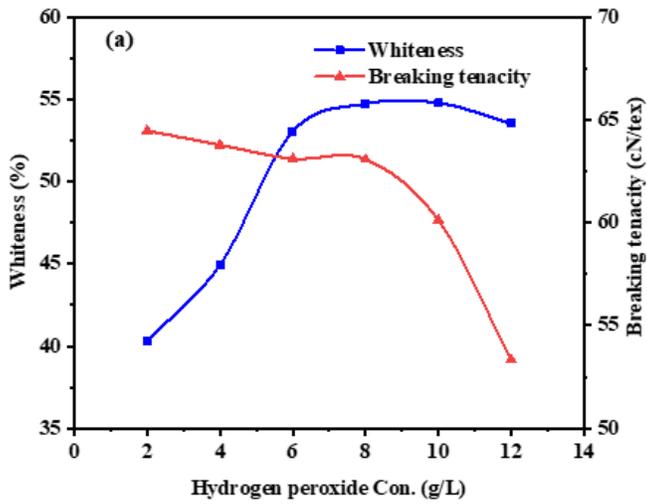


Figure 1

Effect of hydrogen peroxide concentration on: (a) whiteness and Breaking tenacity, (b) weight loss ratio and capillary effect

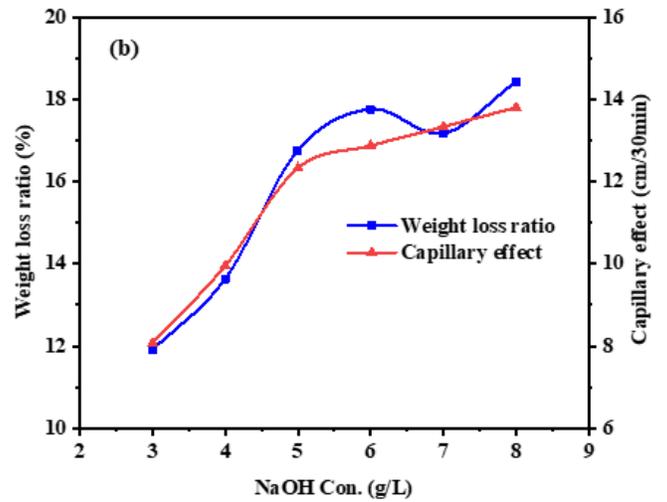
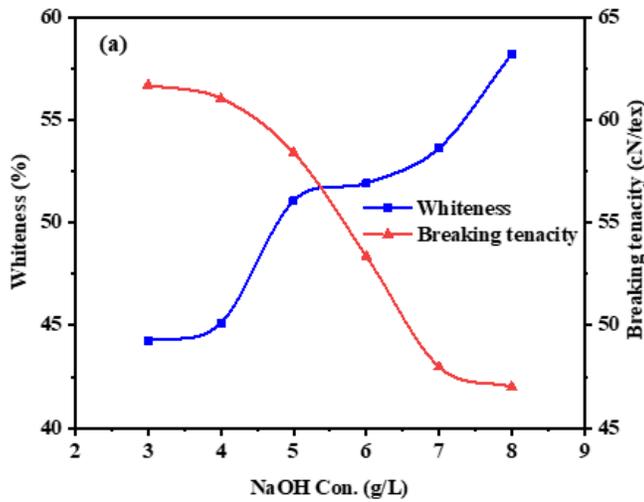


Figure 2

Effect of NaOH concentration on: (a) whiteness and Breaking tenacity, (b) weight loss ratio and capillary effect

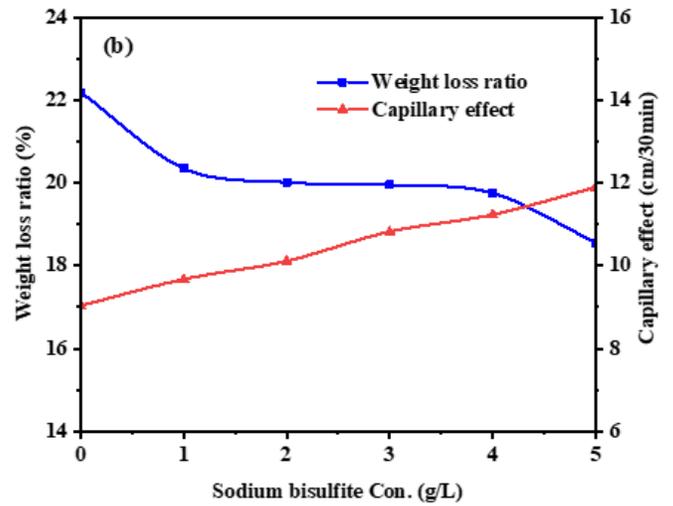
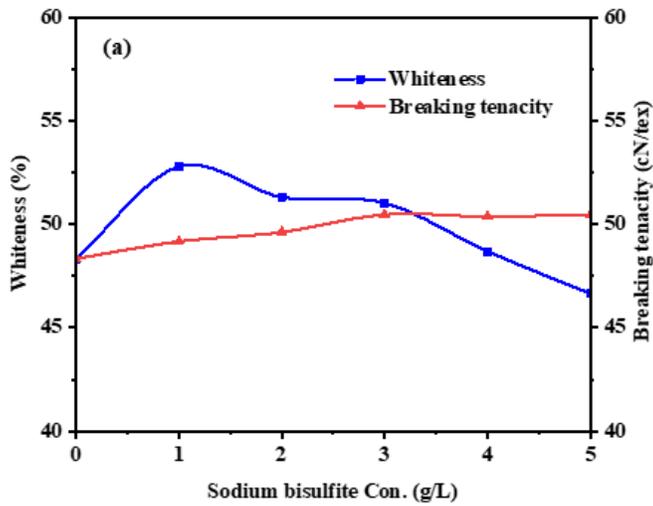


Figure 3

Effect of NaHSO₃ concentration on: (a) whiteness and Breaking tenacity, (b) weight loss ratio and capillary effect

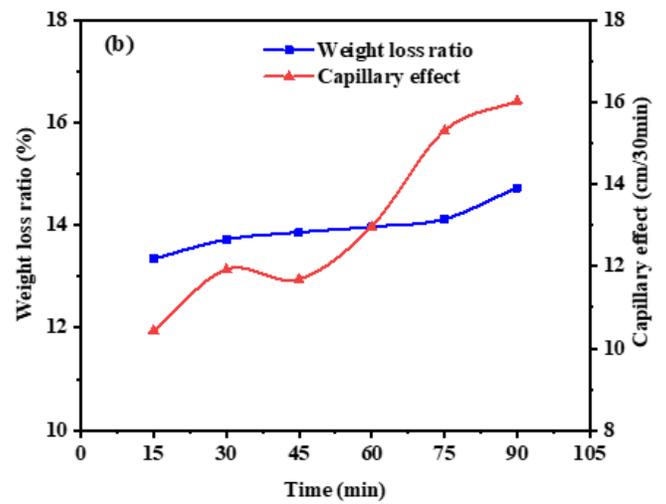
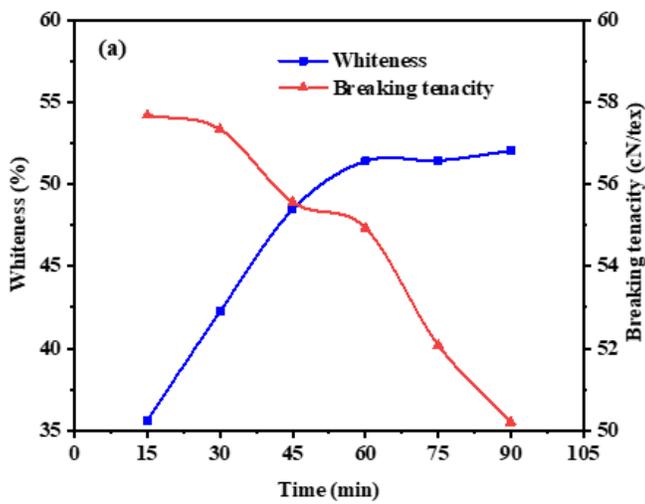


Figure 4

Effect of time on: (a) whiteness and Breaking tenacity, (b) weight loss ratio and capillary effect

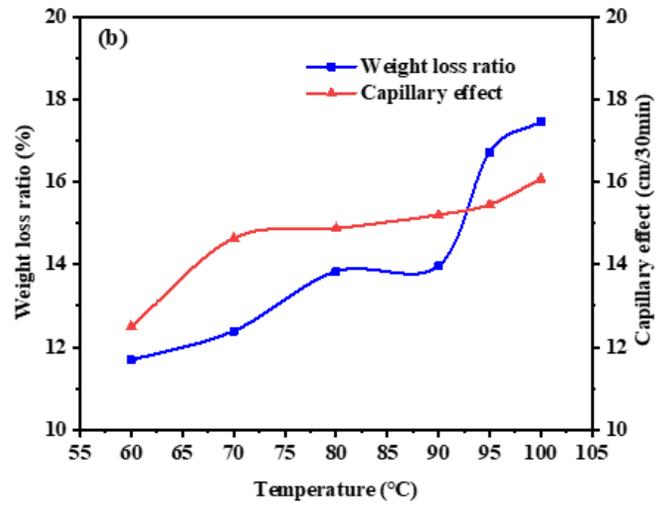
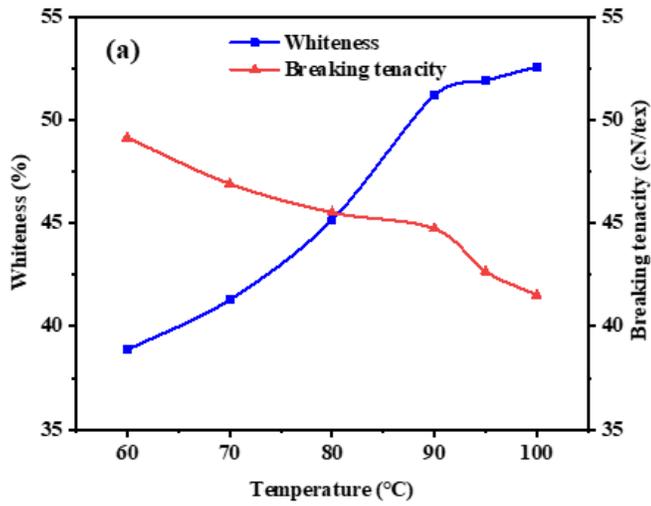


Figure 5

Effect of temperature on: (a) whiteness and Breaking tenacity, (b) weight loss ratio and capillary effect

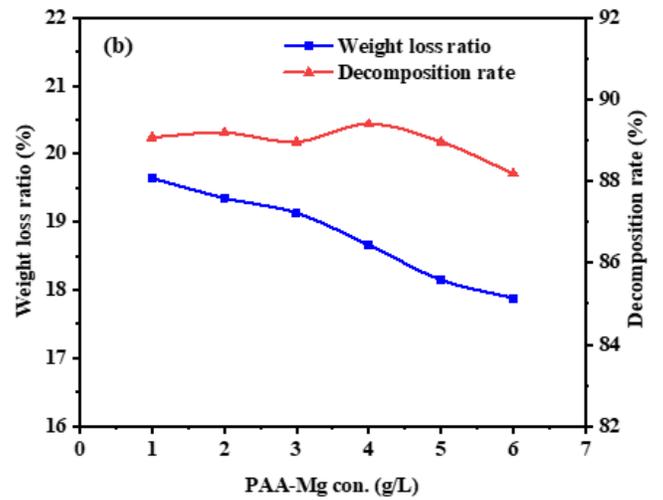
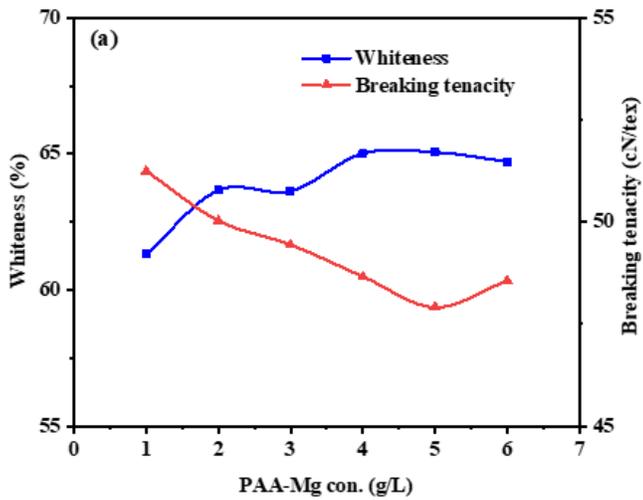


Figure 6

Effect of PAA-Mg con. on: (a) whiteness and Breaking tenacity, (b) weight loss ratio and capillary effect

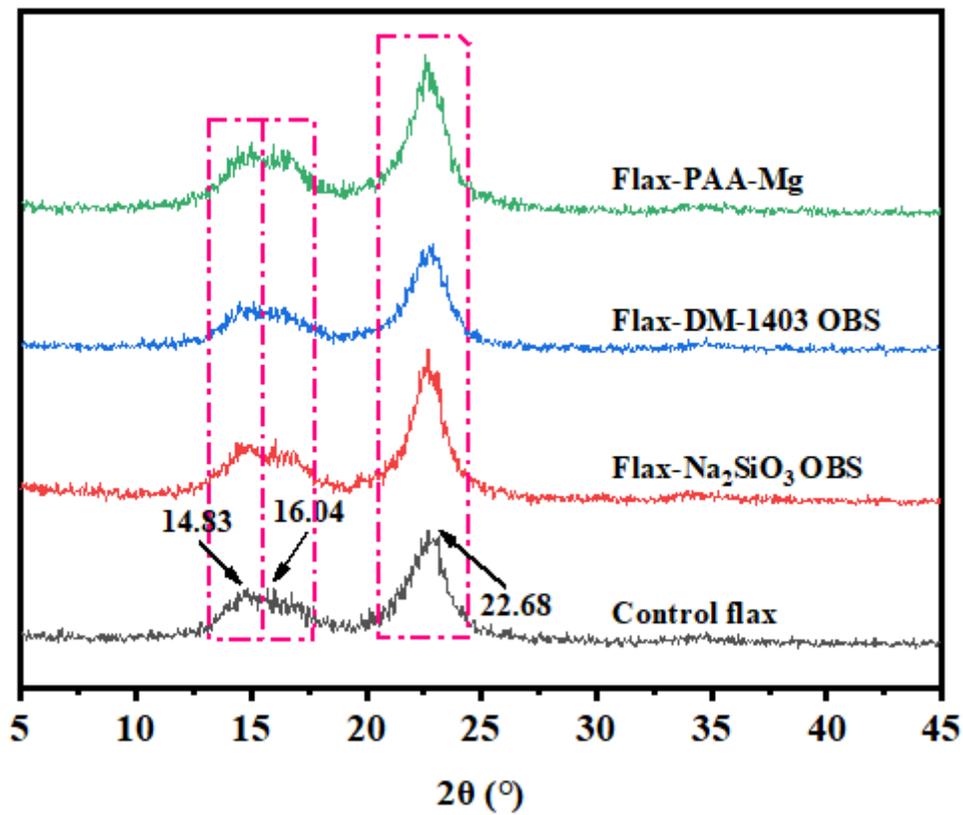


Figure 7

X-ray diffraction patterns of flax roving

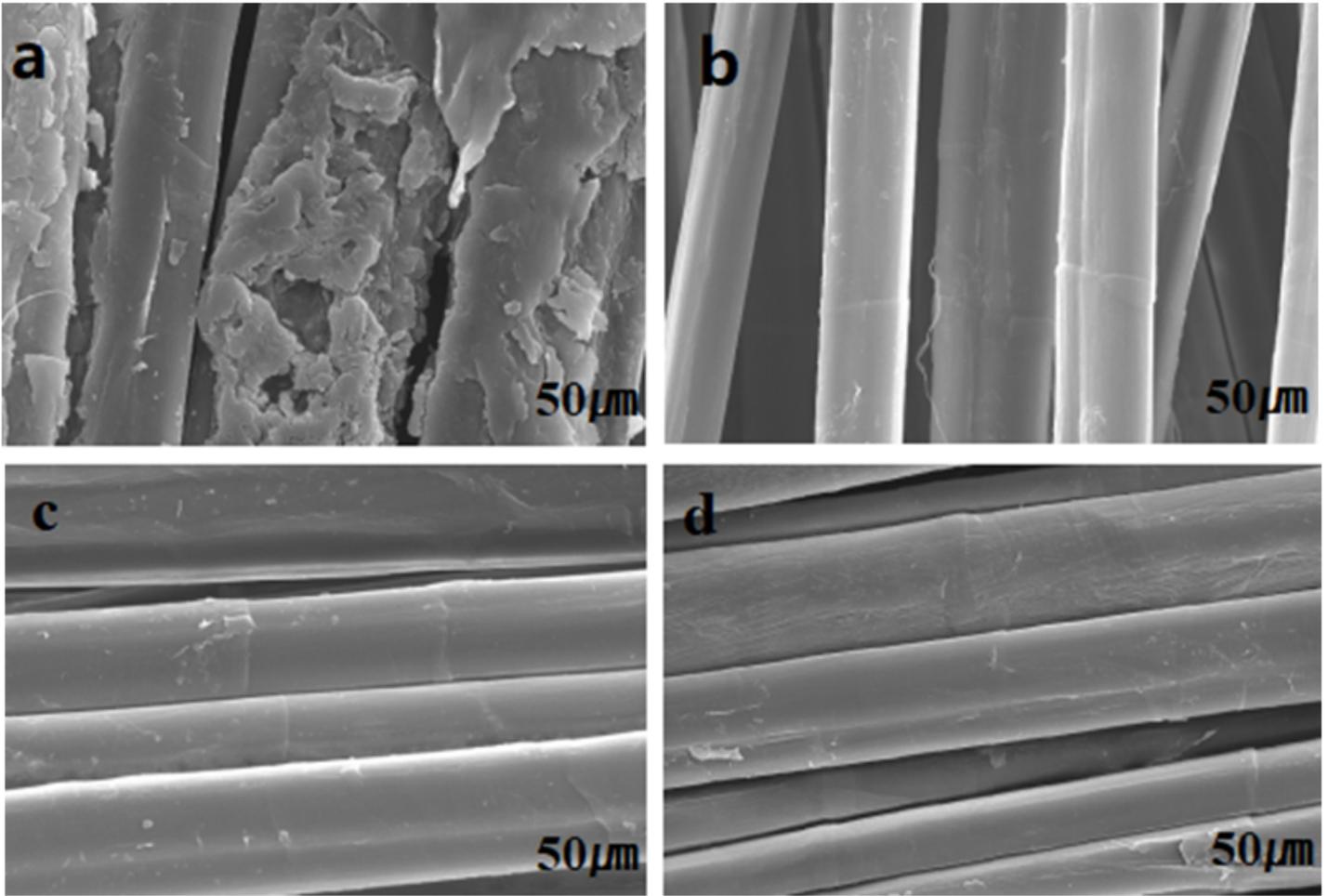


Figure 8

SEM photos of the samples with different stabilizers (a) Untreated; (b) Stabilizer sodium silicate; (c) Stabilizer DM-1403; (d) Stabilizer PAA-Mg