Biological Treatment of Raw Flax with Fungus

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Abstract: Flax is preferred by consumers and used widely in clothing and garments owing to its merits, such as fast moisture absorption and carry-off, natural grain, unique style, etc. While flax, as one kind of natural bast fiber, not only includes cellulose but also includes gum consisting of pectin, hemicellulose and lignin, these materials glue cellulose into stiff sheet bundle fiber, thus, gum must be removed before spinning, through retting process, therefore, retting is the treatment that degrades the pectin-rich middle lamella connecting adjacent fiber cells to release bast fibers, which is the predominant problem in preparation. The original processing of flax is dew retting, which is time-consuming and results in unstable quality of flax. Therefore, Microbe treatment of raw flax is studied in this paper. One strain of fungus screened from soil is used in experiments, and pretreatment of flax is also involved. The evaluation is based on modified Fried Test. Treated and non-treated flax is tested by infrared spectrum and X-ray diffraction. The results manifest that ammonium oxalate is an effective pretreatment chelator to remove calcium, which loosens the tight structure of gum. Therefore, the method in which raw flax is pretreated with chelator followed by treatment with fungus is feasible; furthermore, bast fiber and xylem can be separated fully in 5h.

Keywords: Raw flax, retting; chelator, pretreatment, ammonium oxalate, fungus-treatment.

1. Introduction

Flax, as one kind of bast fibers, is preferred by consumers and used widely in clothing and garments because its fabric has many virtues [1,2], such as, fast moisture absorption and carry-off, natural grain, bland color, unique style, etc. Therefore, the production scale in China is expanded constantly that it has been listed the second globally, next only to Russia now. But the processing technology in China is deficit to meet textile industry's requirements and about seventy percents of flax materials are imported. Retting, the treatment to degrade the pectin-rich middle lamella connecting adjacent fiber cells to release bast fibers [3], is the major limitation in preparation. The original processing of flax is dew retting [4] with the drawbacks of low efficiency and instable quality. As a result of poor quality fibers, considerable effort has been expended to improve dew-retting. The improved retting is water retting and enzyme treatment [5,6], bundles of flax stems were immersed in water(e.g. rivers, ponds), and fermentation by anaerobic bacteria degraded pectins and matrix components in the plant cell wall, thereby retting the flax [7]. The fermentation product constituted such a significant ecological problem that water-retting is no longer practiced, while cost appears to be one major disadvantage in enzyme-retting [8-11]. With the environmental protection being paid more and more attention in the world, use of pollution-free method in textile industry is inevitable. Therefore, we try to develop an environment-benign, efficient retting processing of flax in our research.

2. Experimental

2.1 Materials

Raw flax is from Zhengjiang province all samples are from middle straw and are cut into 10cm. All samples' diameters are more than 0.75mm.

2.2 Experimental design

2.2.1 Pretreatment Experiments

Chelators are used in pretreatment of raw flax to remove calcium in pectin. Elhylene diamine tetraacetic acid, sodium tripolyphosphate, oxalic acid and ammonium oxalate are involved in the pretreatment experiments. Different chelators are used in different temperatures, pH varied from 3 to 10, and temperature from 32℃ to 62℃. Control sample is also treated at different temperatures, pH and treatment time.

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2.2.2 Microbe retting of flax

Standard microbiological techniques are used in screening retting microbe. The pH of culture medium, different carbon source, nitrogen source, cultivation temperature, cultural time, the speed of shaker and the aerobiosis of microbe were involved in experiments. Lastly, a strain of fungus was separated from retted water, it was also proved that it is effective in retting in kenaf [12], the conidial fructification of fungus is listed in Figure 1. Therefore, the fungus is used in the treatment of raw flax. Furthermore, pretreatment and microbe treatment are used in flax simultaneously, then comparisons are done between microbe retting and pretreatment-microbe treatment of flax.

2.2.3 Test criterion

Evaluation of retting is based on mended Fried Test [13,14], a 10cm long straw of flax was put in a test tube and 10mL boiling water was added. The tube was shaken on a vortex in full speed for 10 seconds and thereafter manually shaken in vertical direction 4 times, the samples were then visually graded in a scale from 0-6 based on the fraction length, that is, bast fibers length separated from woody core. The score criterion is listed in table 1. "0" means no bast fibers released, "1" means bast fibers were separated from 0 to 10mm long straw, "2" means fibers released from 10 to 25mm, "3" means 25 to 50mm, "4" means 50 to 75mm, "5" means fibers released from more than 75mm, but still joined in some area, "6" means all bast fibers were released from core. To avoid bias, all the samples were tested thrice and the average was regarded as the final results.

Lastly, control sample and treated sample are tested with infrared and X-ray diffraction.

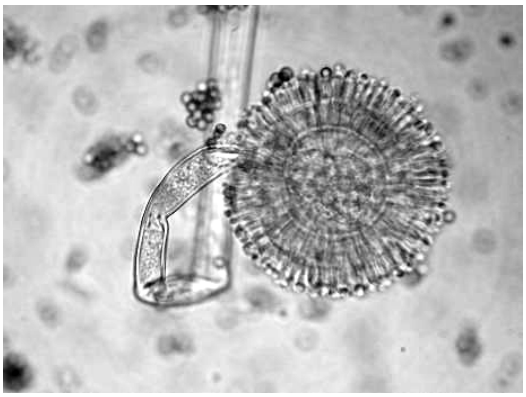


Figure 1 Conidial fructification of fungus.

Table 1 Score criterion

Score	0	1	2	3	4	5	6
Separate length (mm)	0	0-10	10-25	25-50	50-75	>75	100

2.2.4 Test methods

Infrared test is potassium bromide disk prepared under high pressure. Sample and potassium bromide powder of 1~2mg weight are tested at the style of disk.

Powder of sample is tested with X-ray diffraction. The test conditions are as follows: Ni filtration, electric voltage 40kV, electric current 300mA, scanning velocity 2o/min，scanning angle 6^o-60^o.

3. Results and discussion

3.1 Control sample test

Raw flax is treated in water with no agents at different temperatures and different time, respectively. The scores are listed in Table 2 and Table 3. The results prove that rise in temperature does not improve separation of bast fibers from core, the score is still "0", so does extension of time, which indicates that retting of raw flax is not finished without agents or other factors. Therefore, agents are involved in the following experiments.

Table 2 Scores of control sample at different temperatures

temperature(℃)	32	42	52	62
score	0	0	0	0

Table 3 Scores of control sample at different time

time(h)	2	3	3.5	4	4.5	5
score	0	0	0	0	0	0

3.2 Pretreatment of raw flax

Raw flax is treated with EDTA of different concentrations, different pH at ℃2 for 6h and different temperatures at 0.2g/L for 6h, then the treated flax was scored based on modified Fried Test, the scores are listed in Table 4 and Table 5. It is obvious that score does not change with increase of concentration of EDTA, which shows that EDTA is not suitable for pretreatment of flax. The trend of temperature experiments are similar to that of concentration (listed in Table 6). Therefore, EDTA is not an effective agent for pretreatment.

Table 4 Scores of EDTA at different concentrations (g/L)

concentration	0.05	0.06	0.08	0.10	0.13	0.16	0.18	0.2
score	0	0	0	0	0	0	0	0

Table 5 Scores of EDTA at different pH

pH	3	4	5	6	7	8	9	10
score	0	0	0	0	0	0	0	0

Table 6 Scores of EDTA at different temperatures

Temperature(℃)	32	42	52	62
score	0	0	0	0

Sodium tripolyphosphate, oxalic acid are also involved in experiments, while the results are not satisfactory,and are similar with that of EDTA, that is, the change of temperature, concentration and treatment time does not separate bast fibers from the woody core, while ammonium oxalate is useful for separation of bast from core.

Figure 2 shows that score increases with increase in concentration of ammonium oxalate at the same temperature in 5h. Score increases quickly at initial stage, while it has little changes when concentration is more than 5g/L. Furthermore, temperature is notable to start retting, the seperation of bast from core is slower at low temperature, seperation speeds up at higher temperature. But oversize concentration does not improve score at some temperatures.

It is obvious that the more treatment time, the higher score is under concentration 5g/L (shown in Figure 3), which indicates that extension of time is in favor of seperation of bast fibers from woody core, but over a long time does little help in score at lower temperature.

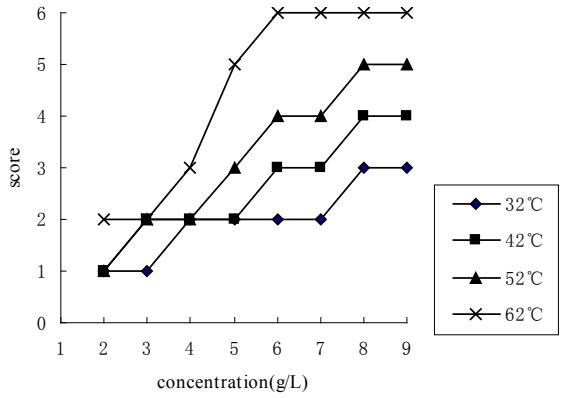


Figure 2 Scores of treatment with ammonium oxalate of different concentrations.

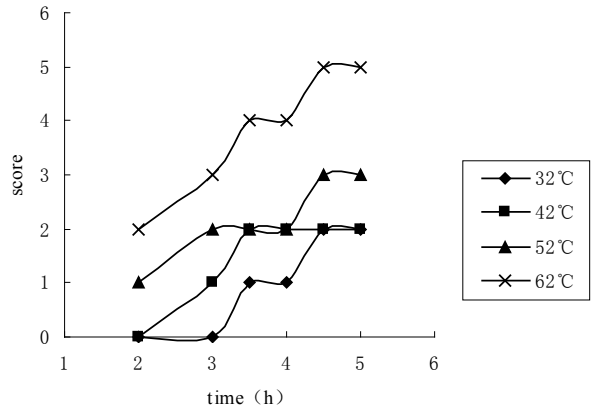


Figure 3 Scores of treatment with ammonium oxalate at different time.

Therefore, ammonium oxalate is a useful chelator in retting of flax. The reason is that two main classes of pectins have been distinguished from each other according to their mode of extraction from plant cell walls. The pectins released with boiling water are highly methylated. They have been reported to be linked with one another, or with other polysaccharide compounds, by covalent bonds [15-17]. Apart from possibly affecting multiple nonspecific chemicals and biochemical degradative processes, treatment by boiling water mainly causes the breaking of pectic molecules by p-elimination. More acid pectins can be solubilized using cation chelators, especially calcium chelators, and are therefore considered to be ionically bonded with each other [18-20]. The calcium ion distribution is shown in Figure 4[21] and the structure of Chelated pectin macromolecule is shown in Figure 5. While ammonium oxalate is one kind of calcium chelator, it can chelate calcium ion from pectin macromolecule, thus the close structure of pectin macromolecule is damaged and retting is easier.

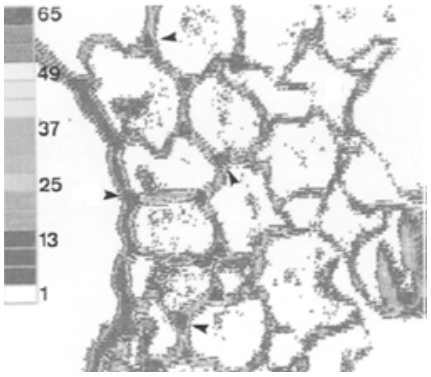


Figure 4 The distribution of calcium ion in pectin.

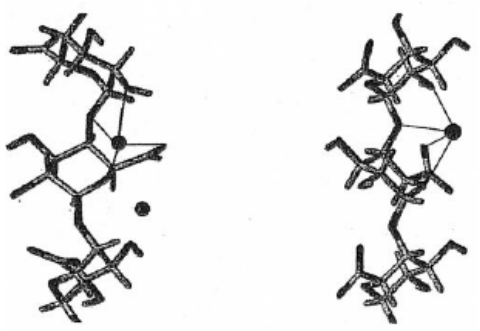


Figure 5 The relative location of calcium ion and pectin.

3.3 Microbe treatment of raw flax

Microbe treatment of raw flax with or without pretreatment of ammonium oxalate is employed. The Fried Test scores of retted flax are listed in Table 7. The results indicate that pretreatment is effective. The reason is that pectin macromolecule in flax is chelateded by calcium ion, which makes gum complex structure difficult to remove. While ammonium oxalate is the chelator and it can remove calcium, then bast and woody core is separated partly. The following processing is microbe retting with fungus that degrades the gum resulting in bast and core separate fully.

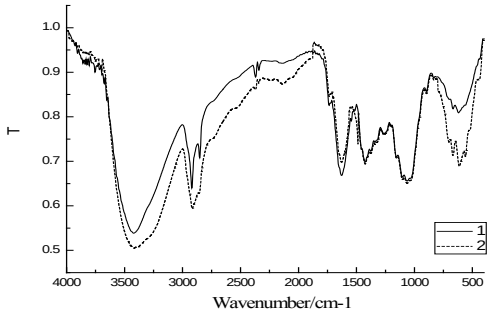
Table 7 Scores of treated flax

Time(h)	1	2	2.5	3	3.5	4	5
Scores(non-pretreatment)	0	0	1	1	2	3	3
Scores(pretreatment)	2	3	3	3	4	5	6

Figure 6 show that the characteristic peak of 1640cm^{-1} weakens, which is the characteristic peak of pectin [22-24], while other characteristic peaks have few changes. Therefore, the content of pectin is reduced, which is due to chelator and fungus. Ammonium oxalate, as one kind of chelator, can damage the tight structure of noncellulose by chelateding calcium ion, then the growth and propagation of fungus consumed pectin and other oligomer in flax resulting in the separation of bast and stem, thus retting of flax is completed.

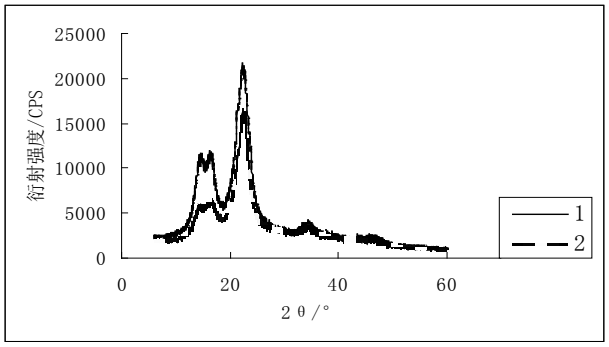
3.4 X-ray diffraction Test

The results (shown in Figure 7) demonstrate that both positions of scanning angle do not change, about 17.0°and 22.8° occur in the two curves of raw flax and treated flax, which indicates that no essential change was produced in microcosmic structure of flax, but the intensity of scanning angle weakens after the treatment, that is, crystallinity of flax changes resulting from treatment, crystallinity is calculated based on X-ray diffraction curve, the crystallinities of raw flax and treated flax are 73.57%, 71.27%,respectively. The reduction of crystallinity attributes to content change of gum of flax.



1: raw flax; 2: treated flax

Figure 6 Infrared of raw and treated flax.



1: raw flax; 2: treated flax

Figure 7 X-ray diffraction of raw and treated flax.

4. Conclusion

Chelator and fungus are involved in flax retting. The results indicate that ammonium oxalate, as a chelator, can remove calcium in pectin macromolecule, thus the close structure of gum is destroyed, which makes it easy for fungus to degrade gum and quicken microbe retting of flax, then the bast and stem of flax can be separated fully by pretreatment with ammonium oxalate for 3h followed by treatment with fungus for 5h.

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