Academic Journal of Engineering and Technology Science
ISSN 2616-5667 Vol.2, Issue 4: 99-106, DOI: 10.25236/AJETS.2019.020614

Study on Extraction of Pentosan from Phragmites Communis

Qian Wang*, Guoming Chen and Caixia Zhang

School of Chemical Engineering and Safety, Binzhou University, Binzhou 256600, Shandong, China
**Corresponding author e-mail: wq_bzu@126.com*

ABSTRACT. *Pentosan is a non-starch polysaccharide, which has a good application prospect in food, pharmaceutical and other industries. In this paper, cheap and easily available reeds are used as experimental materials, sodium hydroxide extraction is used, and the pentosan content in the product is determined by Duffau distillation. The effect of temperature on the extraction of water-soluble pentosan was studied, and the effect of boiling water bath time on water-soluble pentosan was studied. Through the analysis of the results of orthogonal experiments, the optimum technological conditions for the extraction of water-insoluble pentosan in the research area were determined. The experimental results show that the optimal water bath time for the extraction of water-soluble pentosan is 2h, and the effect of different factors on the extraction of water-soluble pentosan is in order of NaOH concentration, water bath temperature, and water bath time.*

KEYWORDS: *pentosan, phragmites communis, Duffau distillation*

1. Introduction

Pentosans are condensates of pentosans, which are widely present in grasses. Studies have found that pentosans are also found in pineapple skin, beer tanks, and reeds. Pentosans are not composed of glucose units, so they are non-starch polysaccharides, which occupy a large proportion in plant cell wall constituents. It is composed of β-L pyranyl xylose residues connected by a β-1,4 glycoside chain as a main chain, and α-L-furan arabinose as a side chain. Pentosans can be generally divided into water-soluble pentosans and water-insoluble pentosans, and the division is based on the difference in water solubility.

Pentosan's special structure gives pentosan some important physical and chemical properties. Pentosans can freely stretch into a helical rod-like structure similar to surfactants in water, which greatly increases the viscosity of the aqueous solution; in the presence of an oxidant, pentosans are easily oxidized in water to form a three-dimensional network The structure and spatial configuration are more

ISSN 2616-5667 Vol.2, Issue 4: 99-106, DOI: 10.25236/AJETS.2019.020614

complicated, which can make pentosan more hydrophilic, so that it can absorb ten times its own weight of water; it can degrade by pentosanase catalysis; when baking foods such as bread, The presence of pentosan can increase the strength and ductility of the dough, make the food more uniform and detailed, and improve the taste. In human foods, pentosan is a type of dietary fiber that is not digested and broken down in the human body. It can absorb water in the intestine and moisturize the bowel. Medical research has found that pentosans can also prevent and suppress breast cancer, control weight and relieve the "three highs". After further research and development, pentosan may also be added as a functional factor to a new generation of health products [1].

Pentosans have attracted widespread attention due to their good function of intestinal laxative, improving the properties of noodle products, and preventing and controlling diseases [2]. Up to now, there are more researches on pentosan detection methods, but less research on pentosans extraction, and it is limited to the wastewater of wheat, beer tank, pineapple peel, and gluten [3-6]. Pentosan detection methods can be summarized into the following categories: chromatography [7], Douglas method [8], Duffau distillation method; extraction methods mainly include the following two types: solution dissolution method and enzymatic method. Finding cheaper raw materials, finding more energy-saving ways, and determining the optimal extraction process conditions have become the goals of experimental research on pentosan extraction. The main purpose of this study was to find the optimal level combination of pentosans extracted from reeds and extract the crude pentosans to improve the utilization of reeds.

2. Experimental Part

2.1 Reagent

$Na_2S_2O_3$, Na_2CO_3, 12% HCl (aq), acetic acid, aniline, furfural.

0.1mol/ L Na2S2O3 standard solution: Roughly weigh 26g of sodium thiosulfate and dissolve it in a small amount of distilled water, add 0.1g of Na2CO3, dilute the distilled water to 1000 mL, shake and transfer to a brown reagent bottle, place in a dark and dark place for one week, filter Calibrate the concentration with the reference material potassium dichromate.

12% hydrochloric acid solution: Measure 307mL of analytical reagent hydrochloric acid and dilute to 1000 mL.

Aniline acetate solution: Measure 2mL of freshly distilled aniline into a dropper bottle, add 18mL of glacial acetic acid and mix.

ISSN 2616-5667 Vol.2, Issue 4: 99-106, DOI: 10.25236/AJETS.2019.020614

2.2 Principle of Experiment

In this experiment, the pretreated sample was transformed into furfural through multiple steps such as thermal decomposition in a 12% hydrochloric acid medium, and then furfural was distilled off by heating distillation, and the furfural was titrated by a titration method to further determine the pentosan content in the sample. When the optimal extraction conditions of pentosans are obtained, pentosans are precipitated by ethanol precipitation, and crude pentosans are obtained by constant temperature drying, and the pentosans content in the crudes is determined.

2.3 Steps of Experiment

2.3.1 Material pretreatment

(1) Material pretreatment of water-soluble pentosan

i Weigh about 20g of reed, wash, filter dry, put it in a beaker, add 100mL of distilled water, and put it in a boiling water bath in a thermostatic water bath for 1h.

ii After the water bath is completed, transfer it to the juicer and pulverize for 10 minutes. After the pulverization, transfer the treatment solution to the original beaker, wash the juicer with 100mL of distilled water, and transfer the washing solution to the beaker. The beaker was placed in a water bath and treated in a boiling water bath for 2 h and cooled.

iii Vacuum filter the cooled treatment solution for 10 min to obtain a filtrate.

(2) Material pretreatment of water-insoluble pentosan

i Weigh about 15g of reed, wash, filter dry, put it in a beaker, add 100mL of distilled water, and put it in a boiling water bath in a thermostatic water bath for 1h.

ii After the water bath is completed, transfer it to the juicer and pulverize for 10 minutes. After pulverization, transfer the treatment liquid to the original beaker, rinse the juicer with 100mL of water, and transfer the washing liquid to the beaker. The beaker was placed in a water bath in a thermostatic water bath for 2 h and cooled.

iii Vacuum-filter the cooled post-treatment solution to obtain a filter residue, place the filter residue in a beaker, and add the same volume of NaOH to dissolve the water-insoluble pentosan.

2.3.2 Removal of starch and protein

(1) Removal of starch

Use 1mol / L NaOH to adjust the pH of the filtrate or filter residue treatment solution to about 6.60, and calculate the amount of α-amylase used based on the amount of starch in the reed. Starch [9].

(2) Removal of protein

i Removal of protein from water-soluble pentosan treatment solution

The treatment solution for removing starch was heated in a water bath at 85°C. for 10 min, and then heated in a boiling water bath for 15 min [10] to remove the protein dissolved in water and excess α-amylase. The treatment solution was centrifuged at 3000 r / min for 15 minutes to remove the precipitate and collect the supernatant, that is, the sample.

ii Removal of protein from water-insoluble pentosan treatment solution

The starch-removed filter residue treatment solution was heated in a water bath at 85°C for 10 min, and then heated in a boiling water bath for 15 min [11] to remove the protein and excess α-amylase dissolved in NaOH. The treatment solution was centrifuged at 4000 r / min for 20 minutes, and the precipitate was removed to obtain a supernatant, that is, a sample.

2.3.3 Distillation and determination of furfural

(1) Determination of pentosan content [12] (Duffau distillation method)

i Connect the experimental device and heat the glycerin to about 180°C.

ii Take a 25mL sample with a measuring cylinder, determine the weight of the electronic balance, pour it into a round bottom flask, add 10g NaCl, 100mL 12% hydrochloric acid in order, connect the round bottom flask, the condenser tube, and the dropping funnel to the dropping funnel. 100mL of 12% hydrochloric acid was added to start the furfural distillation. During the distillation, 12% hydrochloric acid was continuously added to the dropping funnel, and the distillation rate was controlled at 30mL / 10min.

iii When 300mL of distillate is distilled off, collect 1mL of distillate in a graduated cylinder and place it in a beaker, add 1-2 drops of phenolphthalein indicator, neutralize to 1 red with 1mol / L NaOH, and then add 1mL of aniline acetate and let it stand 1 minute, if the solution is colorless, it is proved that the furfural distillation is completed, otherwise the distillation is continued.

iv After the distillation is completed, measure the volume of distillate with a graduated cylinder, then transfer it to a 500mL volumetric flask, rinse the graduated cylinder and beaker twice with 12% hydrochloric acid, and pour the washing liquid

into the volumetric flask together with 12%. Make up to volume with hydrochloric acid, shake well, and wait for determination.

v Using a pipette, measure 100mL of the processed distillate into the iodine volumetric flasks 1 and 2. Measure 100mL of 12% hydrochloric acid into the iodine volumetric flask 3 as a control. Add 12.5mL KBr to 1, 2 and 3 respectively. — KBrO3 mixed solution, quickly stopper the bottle stopper and seal with water for 1 h in a dark place to fully react the hydrochloric acid with Br-.

vi After 1 hour, take out the iodine volumetric flask, dry the water on the stopper with a absorbent cotton, add 5mL of 10% KI solution to it, quickly stopper the stopper, shake well, and let stand for 5min to replace the Br- in the solution.

vii After 5 min, the precipitated iodine was titrated with 0.1 mol / L sodium thiosulfate. When dropping to orange, 1 mL of 1% starch indicator was added to the solution, and continued to drip until the blue color disappeared, which was the end point of the titration.

(2) Determination of total pentosan content in reeds [11]

Cut the reeds with scissors and grind them with a mortar. Accurately weigh about 1g of reed powder on the round bottom

In the flask, add 100 mL of 12% hydrochloric acid to the round bottom flask, connect the experimental distillation device as shown in Figure 1, and heat the glycerin bath to 180 ° C. Add 100mL of 12% hydrochloric acid to the dropping funnel, control the distillation rate to 30mL / 10min, and continuously add 12% hydrochloric acid to the dropping funnel to distill furfural. After the distillation is completed, the total pentosan content is measured according to the pentosan content determination procedure in the treatment solution.

2.3.4 Extraction of Pentosan

The determination confirmed that the water-soluble pentosan content was low in reeds, so only the water-insoluble pentosan crude was extracted in the experiment. The orthogonal test was used to determine the optimal levels of the three factors: the concentration of NaOH in the extract, the time of the bath, and the temperature of the bath. Under the optimal combination of factor levels, a high-concentration water-insoluble pentosan filtrate was obtained.

3. Results and Analysis

3.1 Single factor experiment results

The experiment is based on the measurement of about 20g of reed, and the result is converted to 100g as the reference, as shown in Table 1.

Table 1 Single factor experiment results

No.	Bath time / h	Results /g
1	2	1.64
2	3	8.38
3	4	1.98

According to experimental data, the optimal water bath time is about 2h, between 1h and 2h. As the water bath time increases, the solubility of water-soluble pentosan increases. After 2h, the solubility decreases with time. This shows that the pentosan solubility rate has basically reached the maximum at 2h.

3.2 Orthogonal experimental results

Table 2. Orthogonal experiment and result analysis

No.	A	B	C	Result
1	1	1	1	1.072
2	1	2	2	1.624
3	1	3	3	2.022
4	2	1	2	2.548
5	2	2	3	2.554
6	2	3	1	2.113
7	3	1	3	2.987
8	3	2	1	3.099
9	3	3	2	3.066
K_1	4.718	6.607	6.284	
K_2	7.215	7.277	7.238	
K_3	9.152	7.201	7.563	
k_1	1.573	2.202	2.095	
k_2	2.405	2.426	2.413	
K_3	3.051	2.400	2.521	
Range R	1.478	0.224	0.426	
Best level	A_3	B_2	C_3	
Factor	1	3	2	

Table 2 was prepared based on the results of the orthogonal test. By analyzing the results of Table 5 in the orthogonal experiment, the influence of the concentration of NaOH in the extract, the time of the water bath, and the temperature of the water bath on the extraction of the water-insoluble pentosan can be determined, as well as the optimal factor level combination. The main factors affecting the extraction of water-insoluble pentosan in reeds are as follows: A (NaOH concentration)-C (water bath temperature)-B (water bath time). Therefore, the optimal factor level combination for the extraction of water-insoluble pentosan is

A3B2C3: the NaOH concentration is 2mol / L, the water bath time is 3h, and the water bath temperature is 80°C.

3.3 Determination of total pentosan content in reed

According to the determination procedure of the total pentosan content in the reed in the experimental part, the result obtained was 9.77%.

3.4 Determination of pentosans in water-insoluble pentosan products

The obtained pentosan product was measured in accordance with the determination procedure of total pentosan content in reed, and the result was 32.68%.

4. Discussion and Outlook

The following issues need to be further studied: narrowing the scope of research, studying the water bath time required for the solubility of water-soluble pentosan to reach a higher level, increasing the scope of the study, studying whether the optimal value of NaOH concentration affecting the extraction of water-soluble pentosan, The optimum value of water bath temperature that affects the extraction of water-insoluble pentosan. After solving the above problems, determine the optimal factor level combination for pentosan extraction.

References

[1] Dong Bin, Zheng Xueling, Wang Fengcheng. Research progress of wheat pentosan at home and abroad [J]. Henan Agricultural Sciences, 2005 (5): 8-10.
[2] Yang Hailian, Chen Guocang, Zhang Chenglie. Analysis of nutritional composition of reeds in different habitats [J]. Acta Prasae, 1994, 3 (1): 1-6.
[3] Sun Yuanlin, Li Wenduo, Zhang Shengwan, Liu Huifang, Zhao Liping. Extraction, purification and composition analysis of pentosan from wheat bran [J]. Chinese Journal of Food Science, 2010, 10 (5): 54-59.
[4] Wang Jinhua, Li Dongsheng, Chen Xiong. Extraction and purification of water-soluble pentosan in beer tanks [J]. Food Industry, 2003 (4): 10-12.
[5] Wu Jing, Huang Huihua. Ultrasonic-assisted Extraction and Physicochemical Properties of Pentosan from Pineapple Peel [J]. Science and Technology of Food Industry, 2010 (4): 260-265.
[6] Ma Fumin, Xu Meiling, Guo Xiaorui, Guo Naifei, Shan Shuyan, Bao Shibao. Study on the Water-Insoluble Pentosan Extraction Process of Whole Wheat Flour [J]. Science and Technology of Food Industry, 2013, 34 (03): 269-273.
[7] R. Houben, CFde Ruijter and K. Brunt. Determination of the Pentosan Content of Wheat Products by Hydrolysis, Glucose Oxidase Treatment and Analysis by HPAEC / PAD [J]. Journal of Cereal Science, 1997 (26): 37--46.

[8] S.G.DOUGLAS.A rapid method for the determination of pentosans in wheat flour [J] .Food Chemistry, 1981 (7): 139-145.
[9] Chen Fenglian. Study on Enzymatic Preparation of Wheat Bran Pentosan [J]. Food Science, 2008, 29 (11): 391-394.
[10] Lin Lin. Optimization of determination method of pentosan content [J]. Food and Fermentation Industry, 2005, 31 (6): 101-104.
[11] Li Limin, Zhu Yongyi, Yao Huiyuan. Comparative Study on Determination Methods of Pentosan Content in Grains [J]. Journal of the Chinese Cereals and Oils Association, 2004, 19 (6): 76-79.
[12] Chen Huiping, Tan Zhiliang, Xing Ting milling. Chemical determination method of pentosan in mixed bran [J]. Feed Research, 1996 (7): 22-23.