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Quantitative Analysis of Chemical Components of Plant Fiber

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ABSTRACT

With the shortage of petrochemical resources and the constant pursuit of high quality of life, natural cellulose fiber is widely used in the papermaking field because of its good comfort, resource availability and environmental friendliness. At the same time plant resources as an important source of natural cellulose fiber, also received attention. China is rich in plant resources, paper that made by plant fiber and a wide range of raw materials, such as cotton, linen, mulberry bark, Eulaliopsis, palm leaves, all kinds of bamboo, coconut shells, straw and so on. Quantitative analysis of the chemical composition of plant fiber and its resources is usually carried out by the national standard GB/T5889-865 quantitative analysis of chemical composition of wheat straw but the standard is formulated for wheat straw raw materials, in the analysis of other plant fiber raw materials will inevitably exist some of the problems, therefore, require a relatively accurate and generally applicable method for chemical analysis of plant fiber raw materials.

KEYWORDS: plant fiber; raw material; wheat; straw; standard chemical composition; quantitative analysis

1. Introduction

Natural plant fiber can meet the requirements of environmental protection and meet the requirements of sustainable development of energy, but also has excellent performance, so more and more attention, development and utilization of new plant fiber resources to become the future development of the paper industry main direction.

Plant fiber raw materials as a source of plant fiber widely distributed in nature, rich in resources, a wide range, and the chemical composition of different content, in order to efficient development, the use of this resource must be accurate quantitative analysis of its chemical composition, and knowledge of plant fiber raw materials has become the basis for quantitative analysis of chemical composition.

1.1. Plant fiber raw materials

1.1.1 Introduction to Plant Fiber Ingredients

Industrial, the fiber refers to the soft and slender filament, there is a certain strength, elasticity and hygroscopicity, is the raw material of paper, and plant fiber is the name from the plant resources of fiber, it is widely distributed in the seed plant in a thick wall organization. Its cells are slender, both ends of the sharp, with a thick secondary wall, the wall often have a single pattern hole, mature generally no living protoplasts, it is mainly in the plant from the mechanical support. The raw material of plant fiber refers to the biological raw materials containing cellulose, hemicellulose and lignin, which is fast and has huge regeneration and is the most abundant biomass resource on the earth. There are about 400,000 species of plants that live on the planet, but there are hundreds of plants that can produce plant fiber. There are about 500 species in our country. There has cultivated fiber crops around the world only about 30 species.

1.1.2 Classification of plant fiber raw materials

The classification of plant fiber raw materials is divided according to different standards. According to the genus is divided into the main branches of the mallow, nettle Branch, linden tree, flax, Sangke, legumes, Indus Branch, Euphorbiaceae, Yu Branch, spear Branch, Rui Xiang Branch, Radix, Agariculata, Bamballae, Pineapple, Liliaceae, Palms and Gramineae [l]. According to the morphological characteristics of raw materials, the main points are wood fiber, gramineous fiber, bast fiber, seed fiber, leaf fiber, fruit fiber, root fiber and cob fiber.

1.1.3 Application of plant fiber raw materials

Plant fiber raw materials are mainly used in papermaking, feeding and used in other light industry, the traditional application research focused on paper pulp. As the two of the effective requirements of plant fiber components are cellulose, so the fiber processing process will produce a lot of additional products, such as furfural and xylose, in addition to plant fiber can also be used for particleboard, a variety of composite panels, catering with a variety of fiberboard and so on.

Domestic and foreign have been used in paper or research and development of plant fibers are: cotton fiber, wheat straw fiber, Apocynum fiber, flax fiber, kenaf fiber, kapok fiber, hemp fiber, pineapple leaf fiber, native bamboo fiber, banana stalk fiber, nettle fiber, sisal fiber, sisal fiber, jute fiber, agave fiber, palm leaf fiber, Eulaliopsis binata fiber, Ura grass fiber, coconut fiber, peach fiber, cotton stalk skin fiber, mulberry fiber, bark fiber and so on.

1.2. Chemical composition of plant fiber raw materials

1.2.1 Introduction to chemical composition of plant fiber raw materials

Plant fiber raw materials, the main chemical composition of cellulose, slightly followed by hemicellulose, pectin, lignin, in addition to other small components such as fat, wax, inorganic salts.

In the paper industry, plant fiber raw material components are usually divided into: cellulose, hemicellulose, lignin, pectin, fat wax, water and ash, rather than part of the cellulose is also known as the gum, is prepared paper fiber must be removed, they act together to support the complex from the plant. Different plant fiber, the content of different components is also relatively large, such as cotton fiber content of 95%, little gum, most of the paper fiber plant fiber raw material fiber content of 55% -75% of the fiber content of only about 45%.

1.2.2 Chemical composition of plant fiber raw materials

Plants are mainly composed of fibrous cells and other parts of the cells through the intercellular layer of material interconnection, through a certain method to remove the remaining cells after the remaining fiber called plant fiber. The composition of a wide variety of plant cells, mainly fibroblasts, parenchyma cells, catheters, stone cells and so on. Parenchyma cells generally have only a small thickness of the primary wall; fibroblasts, catheters and stone cells known as the posterior wall cells, with thickened secondary wall, is the plant support organization, but can be used as paper fiber cell. Fibroblasts are mainly thickened cell walls, whose cytoplasm is decomposed after apoptosis, and the cell wall is divided into primary and secondary walls, and the secondary wall has three layers.

M is the intercellular layer and P is the primary wall. S1, S2 and S3 are the outer, middle and inner layers of the secondary wall. The structure between cells is an intercellular layer, mainly composed of lignin, hemicellulose and a small amount of pectin, and has a large amount of lignin in the intercellular layer of ligninous wood such as woody wood. The thickness of the primary wall is generally about 80nm, the composition is mainly lignin, hemicellulose and pectin. Secondary wall is the main body of fibrous cells, the thickness of about 5-10μm, which secondary wall S2 layer as the main body. In the secondary wall of 16 fiber macromolecules connected by hydrogen bonding into microfibrils, and then microfibrils rely on lignin and hemicellulose through the chemical bond to form a certain length and thickness. Lignin and cellulose, hemicellulose through the chemical bond structure known as the lignin-carbohydrate complex (LCC), this structure is relatively close, the use of chemical methods for the dissolution of some of the material can only be partially dissolved or excessive dissolution, it is difficult to quantify a single component, which resulted in a chemical analysis method can only be relatively accurate quantitative components.

1.3. Research Status of Quantitative Analysis Methods for Chemical Components of Plant Fiber Ingredients

In order to rational and effective use of plant fiber raw materials, it must be a quantitative analysis of chemical composition. The process of component analysis is actually the process of gradual degumming of plant fiber. There are many kinds of methods for the analysis of individual components of plant fiber, such as biological method, colorimetric method, spectrophotometer method, gas chromatography, high performance liquid chromatography and thin layer chromatography, which only partially reflect the composition of the fiber.

In order to solve the defects such as long testing period, high water and electricity consumption, high cost and cumbersome operation, the microwave method was applied to the determination of pectin and hemicellulose in wheat straw in 2008, and the results were statistically tested with wheat straw standard contrast, no significant or significant difference, but the reliability and accuracy still need to be further improved and improved.

In the analysis of other plant fiber raw materials, there are scholars according to their own methods were discussed, and played a good effect, such as: Lanzhou University of Technology Jiang Shaojun and other quantitative analysis of chemical composition of the method for some research, for flax some of the properties were improved on the basis of wheat straw standard, and a variety of chemical kit analysis methods were introduced to obtain a hemp analysis method. Compared with the results, we can see that the method improved the composition of flax precision; Gansu Academy of Sciences Institute of Biology Zhao Xiaofeng for some of the characteristics of flax, wheat straw on the basis of the standard of their organic combination. The systematic analysis of plant fiber raw materials abroad is also from the perspective of the paper industry, such as James S. Han, and so on in the analysis of plant fiber, the first plant fiber raw materials in the main components of a more detailed discussion, for the next step of the specific analysis of the theory of the bedding, and then the plant fiber raw materials are divided into carbohydrates, lignin, inorganic, protein and extracts, and the various components were quantitatively analyzed, the method is more general and some outdated, in the composition analysis is also different from our paper industry, mainly for papermaking wood to set, of course, many of the test theory in the method, the test method is still a great reference value, but its accuracy and applicability need to continue to explore.

2. Moisture content and ash test

2.1. Moisture content

In the wheat straw standard, the characterization of the fiber absorbance is determined by using the moisture content index. The moisture content is the percentage of the wet weight of the fiber contained in the fiber. The wheat straw standard is the wet weight of the fiber and the dry weight of the fiber and the ratio of the wet weight of the fiber. The water content of different fibers is not the same, and the moisture content of different seasons due to different moisture content of the measured results are not the same, such as in summer and autumn season, wheat straw water content of about 8%, reed water content of 7% , the water content of rice is about 9%, while in the winter and spring wheat straw moisture content is about 6.5%, we can see the moisture content of raw materials on the impact of water is still relatively large.

At room temperature, fibers, especially natural plant fibers, are in a balance between moisture absorption and moisture, and their hygroscopicity varies with ambient temperature and air humidity. The presence of water molecules in the fiber has three states: direct absorption of water, indirect absorption of water and capillary water. Direct water absorption refers to the polar molecules on the fiber molecules adsorption of water molecules, intermolecular force is greater, more difficult to remove, the need for higher temperature drying. And indirect absorption of water and capillary water is through the water molecules or between water molecules and fiber molecules between the van der Waals force together, the lower binding force, the general low-temperature baking can be removed (Table 2.1 is the same temperature at the same the moisture content measured in the wheat straw sample).

Table 1. Comparison of moisture content of wheat straw samples measured at different temperatures

Drying temperature	60 ℃ baking	105 ℃ baking
The moisture content	6.54%	8.29%

It can be seen from the above table: the same wheat straw samples in two different temperatures for drying, 105 ℃ baking out the water significantly more than 60 ℃ baking out, indicating that some drying at 60 ℃ water molecules are not baked dry, so the sample moisture content test requires a relatively high temperature, but too high drying temperature will make the fiber damage or even carbonation. Therefore, both in the wheat straw standard or in the papermaking standard, the fiber moisture content is obtained through 105 ℃ drying, so that the thermal movement of water molecules, the water molecules in the fiber will be baked out, after a period of time put out Into the dryer at room temperature for about half an hour to weigh, and then into the oven bake 1h, until the two before and after weighing the results of the error does not exceed 0.02%, weight loss and fiber wet weight ratio is the fiber moisture content, the results remain two bit effective figures, the test showed that dry sample drying about 5h can be weighing, wet sample drying about 7h can be weighing.

2.2. Ash

Plant fiber raw materials, in addition to carbon, hydrogen, oxygen and other basic elements, there are many other elements. These elements are indispensable substances in plant cell life activities such as nitrogen, sulfur, phosphorus, calcium, magnesium, iron, potassium, sodium, copper, zinc, manganese, chlorine and so on; and plant fiber types and growth environment, contains the elements of the type and content will be very different. These inorganic elements form inorganic substances or organic complexes together with the adsorbed dust and the like constitute the ash material of the raw material of the plant fiber.

In the papermaking industry, the raw material samples are usually crushed and placed in a crucible. The carbides are carefully burned and then carbonized. The crucible is then transferred to a high-temperature furnace at about 575 ℃. The carbon, hydrogen, oxygen and sulfur components in the raw materials in the form of gaseous compounds lost, the rest of the things that is ash. In the standard wheat straw standard samples are usually made each weighing about 1g of a total of three samples, were placed in the known weight of white porcelain crucible, bake to constant weight. Remove quickly into the dryer to cool, weigh and record. And then placed in a high temperature electric furnace, burning at 575 ± 25 ℃. When the ashes are white or light gray when the furnace. When the furnace temperature dropped to below 250 ℃, remove the asbestos online cooling 2-3min after moving into the dry cooling, weighing and recording. The weight ratio of the remaining material to the original weight is the ash content.

2.3. Summary of this chapter

Moisture content, ash test methods are relatively simple and mature, moisture and ash of the test method is more uniform, and the results page is more accurate. Some of the new test methods, compared with chemical methods, have a certain degree of environmental friendliness, but their operation is relatively complex, and the experimental equipment requirements are higher, and some even have to add some physical mechanical processing, is not yet generally applicable sex, its promotion there are still some difficulties. The test for the rate of plastic as a result of chemical technology is relatively mature, simple operation, and easier to promote the use, it is still subject to attention.

3. Test and Improvement of Main Components of Plant Fiber Ingredients

The various components described in this chapter are the main part of the analysis of plant fiber composition, which mainly includes hemicellulose, lignin and cellulose. Whether this part can accurately analyze whether the analysis of the whole plant fiber raw material is effective. Therefore, on the basis of summarizing the experience and conclusion of the previous experiment, we have obtained the method of analyzing the composition by experimentation. The analysis process of each component will be described in detail.

3.1. Pectin testing and improvement methods

3.1.1 Brief introduction of pectin and its test method

Pectin A colloidal polysaccharide, including the original pectin, water-soluble pectin (pectinized derivatives) and pectic acid, which are present in the leaves, stems and fruits, can be used for food industry and pharmaceutical industry for thickeners, stabilizers and jelly [44]. In the chemical classification of pectin is a carbohydrate derivatives, the basic composition of the unit is D-galacturonic acid, they are α-1,4-glucoside linkage to form a polymer compound (ie, polygalacturonic acid) and molecular weight of 50-300KD.

Pectin can regulate the body of water in the plant, but also the plant body of cellulose, hemicellulose and lignin growth of nutrients. It's content with the growth of plants and gradually reduced, but in the late growth of plants and a slight rebound. Therefore, by measuring the pectin content, can be real-time monitoring of plant growth. In addition, in order to obtain the spunbond fiber, it is necessary to degum the raw material of the plant after harvest, and the degree of fiber degumming directly affects the technical indexes of post-processing. Pectin is one of the main components in the glial of plant fiber raw material.

Usually use calcium ion chelating agent (oxalate money, EDTA) and so on in the case of large bath ratio of raw materials pectin extraction, straw standard pectin test is to extract the water after the sample, were added with 150ml, the concentration of 5g/L oxalic acid solution of the Erlenmeyer flask, equipped with ball-type condenser, boiling 3h. Remove, in the sorting screen in the wash, into the known weight of the weighing bottle, bake to constant weight. Remove quickly into the dryer to cool, weigh and record. Weight loss is the pectin content. However, according to the standard test of mango will ignore the previous step in the operation of water soluble pectin content.

Wuhan University of Science and Technology (Wuhan 430070, China), the determination of pectin content by microwave-assisted extraction method was carried out in the short time (12min), and the test speed was improved. Start the pectin test after the hot water treatment of the sample, it cannot solve the water-soluble pectin measurement problems.

3.1.2 Pectin test method improvement

Taking into account the use of oxalic acid method to extract pectin is relatively accurate, and simple operation, practicality is strong, so the subject of wheat straw standard extraction of pectin obtained weight loss, coupled with water soluble extraction of water soluble pectin and subtracting the amount of hemicellulose degraded in this step, such as the pectin content of the plant fiber material.

3.1.3 Summary of this section

In the plant fiber raw materials: can be used as paper for the effective parts (stems and leaves) contains the amount of pectin is not a lot but not a trace of ingredients, wheat straw can account for 4% -5%, the test principle comparison is quite simple, methodological research is also relatively simple. The quantitative method of pectin in this subject is a bit more complicated than the standard of wheat straw, and the basis of the standard weight loss of wheat straw is slightly changed. The main changes are reflected in the quantitative and degradable hemicellulose quantification of water-soluble pectin.

This method makes the real content of pectin relatively accurate quantitative, for the application of plant fiber raw materials in papermaking, and even pectin single component in other aspects of the application provides a reference.

3.2. Hemicellulose testing and improvement methods

3.2.1 Brief Introduction to Hemicellulose and Its Testing Methods

Hemicellulose is one of the richest renewable resources on earth, and hemicellulose refers to a class of non-cellulosic polysaccharides that are closely associated with cellulose, especially with lignin, which, in plant cells, and lignin closely intertwined with each other, is one of the main components of plant fiber raw materials.

The degree of polymerization of hemicellulose is low, generally 150-200, there are many kinds of hemicellulose glycogen, such as: xylosyl, arabinosyl, rhamnosyl, fucosyl, galactosyl, mannosyl, glucosyl and uronic acid, but the xylose-based chitosan in the nature of the most widely distributed, the highest amount of existence. Hemicellulose can be produced by hydrolysis of xylose, arabinose, and galactose. Sugar can further produce industrial products such as fuel ethanol, xylitol, organic acid, single cell protein, furfural, and ethyl phthalate.

Different types of plant fiber raw materials, hemicellulose content and structure are very different. Generally, the hemicellulose content of coniferous wood is 15% -20%, mainly with polyglycan mannose, and a small amount of xylan. The hemicellulose in hardwood generally accounts for 20% -25% of the wood. Up to 35%, mainly xylan, but also accompanied by a small amount of polyglycan mannose and poly rhamnose galacturonic acid xylose, of which poly-xylose mainly poly-0-B cool base - (4 1-methylglucuronic acid) xylose. The content of hemicellulose in corn stalks was 28.0% that of barley grass was 34.9%, 38.8% in wheat straw, 35.8% in straw and 36.9% in ryegrass.

In the papermaking standard, there is no separate hemicellulose test method, only in GB / T2677.5-93 papermaking raw materials 1% Na0H extract and the final GB / T2677.10-1995 comprehensive cellulose in the test involved the In many studies, hemicellulose was obtained by removing lignin from the remaining cellulose, and then dissolving the hemicellulose in a dilute alkali, and then precipitating it in a certain way to obtain hemicellulose.

The hemicellulose content in the wheat straw standard was obtained by placing the samples after the pectin was extracted into a Erlenmeyer flask with 150 mL of a 20 g/L sodium hydroxide solution, boil for 3.5h, remove it in a sorting sieve and put it in a weighing bottle of known weight, bake to constant weight, remove quickly and cool it in a desiccator, weigh and record, lose weight is hemicellulose of the content.

Hemicellulose polymerization is very low, containing a large number of branched, poor stability, easy to acid and alkali solution dissolved. The cellulose chemical composition of polydextrose, with a high degree of polymerization, insoluble in dilute alkali solution. Wheat straw standard is based on this nature of the removal of pectin after the sample in dilute sodium hydroxide solution dissolved in the material that is hemicellulose, and then sulfuric acid method to obtain lignin content, the cellulose content through 100 reduction to the other chemical composition of the content obtained.

However, the wheat straw standard in the development of hemicellulose content standards ignored an important material - lignin. Lignin is a three-dimensional polymer organic compound that binds phenylpropane through C-C bonds and C-0 bonds, and cannot dissolve even in strong acids. However, under alkaline conditions, the Na0H solution is present in an ionic state, such as the presence of 0H- and Na + in the solution, wherein OH is capable of nucleophilic reaction with certain of the bonds in the lignin leaving the bond, degradation of macromolecules. According to the nature of lignin, when the finished pectin is extracted from the sample, not only the hemicellulose material is dissolved by the lye, but also the lignin is dissolved by the nucleophilic reaction. The material content should be changed to hemicellulose plus part of the lignin content, so the resulting hemicellulose content is too large, thus affecting the cellulose content, so that small. In the experiment, it was found that the content of lye lignin was about half that of total lignin for wheat straw, as shown in Table 3.2, so the error caused by lignin must be eliminated.

	The content of total lignin			The content of alkali soluble lignin		
Extraction yield	1.99%	2.01%	2.04%	0.94%	1.02%	1.10%

As can be seen from the table, because the sesame lye lignin content can reach about half of its total lignin, so its presence, will make the use of wheat straw standard plant fiber raw materials in the hemicellulose and cellulose quantitative analysis of the results and the real value of the error between, in particular, for the higher content of lignin raw materials, the error is more obvious, directly affect the development of degumming process and the correct evaluation of plant raw materials.

3.2.2 Determination of hemicellulose test methods

According to the standard of wheat straw, the remaining residue after treatment with lye is lignin and cellulose, and according to the analysis of the previous section it can be concluded that some of the lignin and cellulose are left after alkali cooking, not all of the lignin, then the determination of hemicellulose content need to remove the impact of lye lignin, so the main task of this subject is how to quantify the content of lye lignin.

In the case of quantitative analysis of the chemical composition of plant fiber raw materials, the improvement test of hemicellulose is as follows: firstly, the total lignin content of the sample is measured by sulfuric acid method, and then the chlorinated method or sulfuric acid method the content of the remaining lignin in the sample after the extraction of the hemicellulose was measured, and then the difference was the content of the lye lignin, and the content of the hemicellulose was reduced by alkali boiling weight loss. On the water-soluble hemicellulose that is. According to the experimental time and the final effect, the chlorination method is more suitable for the analysis of this step, so the subject of hemicellulose test will be cited [54] method to carry out the specific operation as follows:

1. In accordance with the requirements of the standard wheat straw, the finished pectin samples were placed in a round bottom flask, boiled with 20 g/L sodium hydroxide solution for 4 h, washed with distilled water and placed in an oven at 105 ℃. Weight loss.

2. Remove the remaining residue from the sieve, brush with a brush into a 250mL Erlenmeyer flask, treated with chlorination, weight loss of alkali-insoluble lignin, and finally the remaining residue is cellulose.

Chlorination method: To the conical flask by adding 65mL of distilled water, 0.5mL glacial acetic acid and 0.6g sodium chlorite shake. In the conical flask on the back of a capacity of 25ml Erlenmeyer flask, placed in 75 ℃ constant temperature water bath heating for 1h. Rotate and shake the conical flask during heating. After 1h then add 0.5mL glacial acetic acid and 0.6g sodium chlorite, shake, continue heating in 75 ℃ water bath for 1h, so repeat until the sample goes white.

3. Pour the treated sample into a glass wool core of known weight and rinse continuously with distilled water. Will be washed with the sample together with the glass core into the oven at 105 ℃ in the drying, weighing, the weight loss is the weight of alkali-insoluble lignin, the remaining weight is the weight of cellulose, lye lignin Is equal to the amount of total lignin minus the amount of alkali-insoluble lignin.

4. Hemicellulose content = water soluble, pectin measured in the process of measuring the content of hemicellulose + wheat straw standard alkali weight loss - lysis lignin content.

3.2.3 Summary of this section

This section solves the problem of the determination of lignin in the raw materials of plant fiber by standard wheat straw standard, and accurately tests the hemicellulose and cellulose content, which plays an important role in the development and utilization of plant fiber raw materials.

3.3. Lignin test method improved

3.3.1 Brief Introduction to Lignin and Its Testing Methods

Lignin (Lignin), also known as lignin, is composed of phenylmethane structural units (such as C6-C3 unit) through the ether bond, carbon-carbon bond from the aromatic polymer compounds, which mainly by pine alcohol, Isosorbide propane, lignin propane, and a monomer such as light-based phenylpropane, and the chemical structure of the monomer is shown in Figure 3.4. As the aromatic polymer lignin has a high acid resistance, in the general concentration of strong acid cannot be dissolved, but also insoluble in dilute alkali, dilute acid solution.

The chemical structure of lignin is the most troublesome of natural macromolecules. Its research history is very long and can be traced back to the 1930s. Its research mainly focused on the pulp of wood. From the confirmation of the presence of lignin from Payen, to the development of the sulfite pulping process, to the Klason lignin quantification method and his proposed pineapple theory, until the late 20th century, a variety of lignin purification, separation test, the structure and nature of lignin were clearly understood, which further promoted the development of lignin chemistry and

pulping chemistry, and lignin had a wide range of uses in industry. With the development of science, lignin application, lignin chemistry in the national economy will be increasing.

Lignin as a three-dimensional structure of the natural polymer, widely present in the higher vascular plants in the door, they and cellulose, hemicellulose through each other, together constitute the entire plant skeleton, and it's content with different plant species and the same species of different morphological parts and have greatly changed.

There are two methods for the quantitative analysis of lignin: one is to lignin itself dissolved or become its derivatives and dissolved, measured weight loss, such as the traditional Brahms natural lignin method, Beckman lignin method, organic solvent extraction lignin method, alkali lignin method, chlorinated lignin method and nitric acid ethanol method. In recent years there has been a high boiling method to extract rice hull, peanut shell lignin, made some progress; the other is the lignin other than the composition of the dissolved, the remaining residue weight is lignin content, such as sulfuric acid lignin method (Klason lignin method), hydrochloric acid lignin method, cellulose decomposition enzyme method, copper ammonia lignin method and periodic acid method. Among them, the sulfuric acid lignin method is one of the most commonly used and more accurate quantitative analysis method, is considered the most trusted method for the world as a standard method used, the only drawback is that a small part of the acid soluble lignin cannot be determined , So that the measured lignin content is lower than the actual content, if the UV spectrophotometer method of measuring the content of acid-soluble lignin combined with sulfuric acid method can be more accurate lignin content.

3.3.2 Lignin test method improvement

In the existing plant composition analysis method, due to the accuracy and recognition of sulfuric acid method, for the lignin test are used sulfuric acid method. In the paper industry lignin extraction process is as follows.

Accurately weighed 1g (quasi-0.0001g) sample, extract the wax after drying and weighing, into the capacity of 250mL with a ripple velvet bottle. Add pre-cooled to 12-15 ℃ 72% sulfuric acid 15mL, plug the stopper, shake 1min, so that the sample was completely acid impregnated. The conical flask was then placed in a constant temperature water bath pre-conditioned at a temperature of 18-20 ℃ and held at this temperature for 2.5 hours and the contents of the conical flask were often slanted. After the specified time has elapsed, the contents of the flask are transferred into a 1000 ml Erlenmeyer flask and the Erlenmeyer flask is rinsed with distilled water. All the residue and lotion are washed into 1000 ml Erlenmeyer flask and then diluted with water to a concentration of 30 %, the amount of distilled water added and the total volume of water in the rinse flask are 560 ml.

The large conical flask is equipped with a reflux condenser, boiled for 4 h, and allowed to stand so that it cannot be deposited. With the constant weight of the quantitative filter paper (filter paper should be pre-washed with 3% sulfuric acid solution 3-4 times, then hot distilled water to wash the liquid does not show acidic reaction, and then dried to constant weight) filter, then hot distilled water , to the lotion with 10% barium chloride solution try not turbidity. And then the filter paper together with the residue into a weighing bottle, placed in 105 ℃ 3 ℃ oven drying to constant weight that lignin weight.

Such as non-wood raw materials, it is necessary to determine the ash contained in lignin. So that can be dried to constant weight with the residue of the filter paper into the constant weight of the dust, before the lower temperature burning to the filter paper all carbonation, and then into the high temperature furnace, no more than 575 25 ℃ at the temperature of the burning to the ash without black carbon, and constant weight so far.

Wood raw materials in the content of lignin directly with the weight of the residue divided by the dry weight of the sample, and for non-wood raw materials will use the weight of the residue minus the weight of the residue after the residue divided by the dry weight of the sample.

Can be seen in the paper standard for the lignin test quite trouble, and most of the paper fiber plant fiber materials are non-wood, if tested in accordance with this method, have to test ash and remove the whole process is too cumbersome, it is difficult to avoid human error impact.

In the wheat straw standard, the lignin content was obtained by the following steps: from the spade, random selection of 4-6 points, take about 5g of the sample, extract the wax after the air dry crushed (length of not more than 1.5mm), weighed each weighing about 1g of the sample, a total of three. Then, placed in a known weight of the embossed Erlenmeyer flask, baked to constant weight removed quickly placed in the cooler to cool, weigh, record. And then slowly add 30mL 72% sulfuric acid solution. Placed at 8-15 \u0026 lt; 0 \u0026 gt; C for 24 h. Then, it was transferred to a Erlenmeyer flask, diluted with distilled water to 300 mL, filled with ball-type condenser tube boiling 1h, slightly cold, with a known weight of the glass sand core filter repeatedly filter, washed until the filtrate does not contain sulfate ions. Remove the glass sand core filter to dry weight, remove quickly put in the dryer cooling, weighing and recording. The lignin content is expressed as the ratio of the weight to the total weight of the material in the sand core.

It can be seen that the principle of wheat straw standard and papermaking standard test lignin is the same, wheat straw standard in the operation is relatively simple, but the use of a long time. Gunes UCAR and so on with 77% sulfuric acid overnight soak the sample 12-14h, and then diluted to 25%, and in the 55 ℃ water bath for 2h, and then diluted to about 12% in 95 ℃ or so in the water bath heated 1h, After the cold filter, the remaining residue is lignin.

In this paper, based on the standard of wheat straw, according to the papermaking standard, the size of the sample was over 40 mesh, but the soaking time and the boiling time were changed. The soaking time was changed to 12h, and the boiling time for 4h, the results (as shown in Table 3.3) indicate that this method is feasible.

Methods	Soak 12 h boil 4 h			Soak 24 h boil 1 h		
The content of lignin	1.99%	2.01%	2.04%	1.98%	2.00%	2.07%
	1.66%	1.65%	1.63%	1.67%	1.63%	1.66%

As can be seen from the above table, the improved method can achieve the effect of wheat straw standard, but also save time, the results are relatively stable, can be used as plant fiber raw acid insoluble lignin content of the test method.

For the above-mentioned problem of lignose, papermaking standards and other studies have also been related to the solution, generally cited UV spectrophotometer. In the national standard GB / T10337-89 'papermaking raw materials and pulp in the determination of acid-soluble lignin' is in the paper standard GB / T2677.8-93 'paper raw material insoluble lignin content determination' based on the reference UV spectrophotometer, and the introduction of a light absorption coefficient (obtained from the average of different raw materials and pulp), this method is mainly for papermaking wood may be, so its application in the papermaking material also need further improvement.

3.3.3 Summary of this section

In this section, the problem is too long for the wheat straw standard, in the extraction of the wax samples were crushed, put into the plug of the grinding mouth conical flask, first with 72% sulfuric acid overnight soak for about 12h , must start to shake the cone to make the sample and sulfuric acid completely contact. After the scheduled time, transfer to the 500 mL Erlenmeyer flask according to the requirements of the standard wheat straw. After the condensing device is installed, boil for 4 hours. The following steps continue to operate according to the requirements of the wheat straw standard until the final weight of the residue is lignin.

3.4. Cellulose testing and improvement methods

3.4.1 Introduction to Cellulose and Its Testing Methods

Cellulose is the main component of the cell wall of higher plants and is the most abundant renewable organic resource in nature. Its chemical composition is β (1-4) -D-polyglucose, and the chemical structure is shown in Fig. The term 'cellulose' as used herein refers to a chain-like polymer compound which is insoluble in water, dilute and dilute D-glucosyl groups at room temperature with β-1,4 camp bonds.

High degree of polymerization of cellulose, with a large molecular weight, the nature of relatively stable, water, a variety of strong alkali, polar liquid can make it swell, high concentrations of strong acid to dissolve, cellulose can also be dissolved to ZnCL2 / DMAc solution, copper ammonia solution, copper ethylenediamine solution, ionic liquid medium. In the plant fiber raw material is not only the presence of cellulose, other components of the interference will make many methods are not suitable for chemical analysis of plant fiber raw materials quantitative analysis.

Plant fiber raw materials in the extraction of cellulose components, testing and so on at home and abroad have a lot of research. C.Ververis and other paper for the use of different plant materials, lignin, cellulose content test, the cellulose content obtained by the following means: first sample with nitric acid / acetic acid (volume ratio of 1: 8) at 100 ℃ Treatment of 1h, removal of lignin, hemicellulose and pentosan, with 67% sulfuric acid dilution, and then at 620nm with onion oil colorimetric method to obtain cellulose content. This method is suitable for large quantities of samples, can also be used in other plant fiber raw materials on the quantitative analysis of cellulose components.

In the wheat straw standard, the cellulose content was obtained by reducing the amount of fat wax, water, pectin, hemicellulose and lignin with 100It is clear that the amount of cellulose is accurate or not directly related to the amount of the other components, so it is necessary to accurately test the content of each part.

Because the thermal stability of cellulose is strong, 150 ℃ below the continuous heating will only make its strength decreased, the heating temperature below 240 ℃, the cellulose mass loss is less, in the plant fiber raw material composition analysis of the temperature are at temperatures below 150 ℃, the quality of the cellulose is not normally changed, so that the cellulose can be accurately quantified according to the standard operating mode of the wheat straw.

3.4.2 This test system method of cellulose testing

In this test system, the acquisition of cellulose content can continue to follow the standard method of wheat straw, but also in the hemicellulose test with chlorination method to deal with the weight of the sample residue and the original sample dry weight ratio of cellulose content. Theoretically, the two results of the resulting cellulose should be the same, and the accuracy of the experiment can be measured by their difference size. Table 3.4 shows the relationship between the cellulose obtained by the chlorination of the wheat straw sample and the cellulose content obtained by subtraction.

The dry weight of sample	The amount of cellulose obtained from the method	The amount of cellulose in the subtraction	Two Numbers of direct approximations
5.0484	71.71%	71.21%	0.9932
3.7719	72.06%	71.59%	0.9934
4.9671	71.63%	71.07%	0.9923

From the table can be seen: the results obtained by the experimental ratio is smaller than 1, with the ideal results or a certain gap, but also can see that their approximate rate of 0.99 or more, the test results are still relatively accurate.

3.4.3 Summary of this section

Several quantitative methods of cellulose are described in this section, but they are basically quantitative for pure cellulose and therefore cannot be fully applied to the chemical analysis of plant fiber raw materials. In this paper, the content of cellulose is finally determined as the weight ratio of the remaining sample after the chlorination process, and the content of cellulose obtained by subtraction can be used as a reference for verifying the accuracy of the experiment.

3.5. Summary of this chapter

In this chapter, the test of pectin, hemicellulose, lignin and cellulose content in the method: pectin, lignin test is carried out in this subject, based on the existing theory, after a large number of experimental screening , the first two kinds of experimental workload but increased the accuracy of the quantitative analysis, lignin testing to ensure the accuracy of the circumstances, the screening process to shorten the test time and improve the test efficiency; hemicellulose The test is to refer to other scholars have the test method, to a large extent have improved the accuracy of the test; cellulose content of the test continued the results of hemicellulose testing, and can be used to verify the traditional wheat straw method, High operability.

Due to the complexity of plant components and the incomplete nature of the test methods, the testing of various components cannot be completely expressed, only the process of continuous optimization. Experiments show that the organic combination of each method can be relatively accurate test of the content of wheat straw, there is a certain degree of progress, for other plant fiber raw materials testing will be given in the following sections.

4. Summary and Application of Test Methods for Plant Fiber Materials

4.1. The system framework and the specific operation process of this research method

Which is not the main content of this subject, so it has not done much research in the experiment process. The following part of the dashed box is the core of this subject, their specific operation and arrangements are as follows:

1. Sample handling:

The samples were randomly sampled and the samples were pulverized into micro-sized pulverizers to a size of 20 mesh but 60 mesh sieves, wrapped in a 200-mesh sieve cloth (weight M) with a known weight extracted with an organic solvent, each sample about 3-5g, a total of three, wrapped sample sieve cloth bag height is lower than the fat extractor overflow about 10-15mm, until the sodium hydroxide solution to extract the sample, the sample is removed from the sieve and transferred to a conical flask.

2. Pectin content:

The samples were extracted with water, and the mixture was placed in a round bottom flask containing 150 mL of 5 g / L of oxalic acid, and the mixture was boiled for 3 hours. Remove the distilled water, and then put into the oven at 105 ℃ bake constant weight. Remove the charge quickly placed in the cooler to cool the weight and record it as M3.

The extract was quantitatively reduced to the amount of hemicellulose degraded by spectrophotometry according to the method described in 3.2.2. The sample pectin content according to formula (3):

$$W_3 = \frac{M_2 - M_3 + P_1 - H_2}{G_0} \times 100 \quad (3)$$

Where: the pectin content of the W3- sample, %;

M3- extraction of pectin after the sample and sieve dry weight, g;

H2 - Degradation of hemicellulose in the process of pectin degradation, g.

3. Lignin content:

Will be crushed by the size of the crusher over 40 mesh, but 60 mesh sample sieve samples wrapped with silk sieve cloth, extracted with phenylethanol 6h after removal. Weigh three specimens of about 1 g each weighing, placed in a known weight of a stoppered flask, bake to constant weight removed quickly placed in the dryer cooling, weighing and recording. Then slowly add 30 ml 72% sulfuric acid solution, at 8-15 ℃ overnight for 12h.

The treated sample was transferred to a 500 ml Erlenmeyer flask, the conical flask was rinsed with distilled water, poured into a flask, and diluted with distilled water to about 300 ml, and the condenser was boiled for 4 h. After cooling, pour it into a glass wool core of known weight and recycle and rinse it with distilled water until the filtrate does not contain sulfate ions (tested with 10% barium chloride solution). Remove the glass sand core bake to constant weight, remove quickly put in the dryer to cool, weigh and record. Lignin content according to formula (4):

$$W_4 = \frac{G'' - G'}{G_0'' - G_0'} \times 100 \quad (4)$$

Where: W4 sample lignin content, %;

G '- the sample of lignin and glass sand core total dry weight, g;

G'-glass sand core dry weight, g;

G0 '- the total dry weight of the sample with a stoppered conical flask, g;

G0'-plugged conical flask dry weight, g.

4. Hemicellulose content:

A sample of pectin was extracted and placed in a round bottom flask containing 150 mL of sodium hydroxide solution at a concentration of 20 g / L. The mixture was boiled for 4 h. The mixture was washed with distilled water and placed in an oven at 105 ℃, dry to constant weight after weighing, recorded as M4.

The residue was removed from the sieve, brushed into a 250 mL Erlenmeyer flask, treated with chlorination (see 3.4.2), and the treated sample was poured into a known glass wool core, and rinsed with distilled water. And then rinse the finished sample together with the glass core into the oven at 105 ℃ baking to constant weight after weighing, recorded as M5.

The hemicellulose content according to formula (5):

$$W_5 = \frac{M_3 - M_5 + G' + H_1 + H_2}{G_0} \times 100 - W_4 \quad (5)$$

Where: W5 - hemicellulose content of sample, %;

M5-chlorination method to treat the remaining cellulose and glass sand core dry weight, g.

5. Cellulose content:

After treatment of the sample with chlorination, the remaining white residue is cellulose, according to the operation in 6 drying, weighing and recording, the content can be calculated according to formula (6):

$$W_6 = \frac{M_5 - G'}{G_0} \times 100 \quad (6)$$

Where: W6 - the cellulose content of the sample, %.

Finally, the amount of cellulose obtained by subtracting pectin, hemicellulose and lignin from 100 was compared with the amount of cellulose obtained in the experiment to verify the accuracy of the experiment. The closer their ratio was, the more accurate the experiment was.

For plant fiber raw materials with high pigment content, such as Apocynum venetum, it is necessary to extract the pigment further with organic solvent to avoid the effect of the pigment on the result of the spectrophotometer when the water extract is tested, the dilution factor of the liquid can be adjusted according to the actual situation according to the different plant fiber.

5. Conclusions

In this paper, wheat straw was used as the research object. Based on the existing theories and methods, a method for quantitative analysis of the chemical composition of plant fiber raw materials was determined. The feasibility and accuracy of other raw materials were worth definitely. Through the experimental process and the results of the analysis of the following conclusions:

(1) The raw material sample crushed, the size of the sample through the 20 mesh sieve, but 60 mesh (1 mm or so), with the organic solvent extracted from the known weight of 200 mesh sieve wrapped cloth package, so not only can effectively extract the components, but also to prevent leakage during the extraction process and loss, and in the whole process of silk screen weight loss is only 0.82%, so its distribution to the steps of the error is negligible.

(2) The use of spectrophotometer to determine the application of plant fiber raw material water extraction method, compared with the wheat straw standard more complex operation, but more accurate quantification of the water content, and also for the accurate test pectin and half cellulose content laid the foundation. When using a spectrophotometer, the treatment of the extract is critical before the absorbance is measured. The establishment of the standard equation and the choice of the maximum absorption wavelength are determined depending on the instrument used.

(3) The quantitative method of pectin in this subject is two steps higher than that of wheat straw standard, and the basis of the standard weight loss in wheat straw is slightly changed. The main changes are reflected in the quantitative and degradable hemicellulose quantification of water-soluble pectin. This method makes the real content of pectin relatively accurate quantitative, for the application of plant fiber raw materials in papermaking, and even pectin single component in other aspects of the application provides a reference.

(4) Hemicellulose test to solve the standard test of plant fiber raw materials with lignin problems, accurate hemicellulose, cellulose content of the test, the development and utilization of plant fiber raw materials has an important role in guiding.

(5) The problem of lignin testing in wheat straw standard is too long. In this paper, the immersion time and boiling time of different concentrations of sulfuric acid are changed under the premise of ensuring the accuracy of extraction.

Of course, the use of sulfuric acid method of testing lignin or there are many problems, such as lysis lignin, sulfuric acid original concentration, dilution concentration, fiber size of particles, the size of the filter glass core size will be more or less affect the final results.

(6) Several methods of quantification of cellulose are described in this paper, but they are not fully applicable to the quantitative analysis of the chemical composition of plant fiber raw materials. In this paper, the content of cellulose is finally determined as the weight ratio of the remaining sample after the chlorination process, and the content of cellulose obtained by subtraction can be used as a reference for verifying the accuracy of the experiment.

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