

Study on the degumming process of *Abelmoschus manihot* (L.) Medic fiber

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Abstract: *Abelmoschus manihot* (L.) Medic has drawn much attention recently due to its potential beneficial health effects. The roots, flowers and seeds of *A.manihot* play an important role as medical materials. In this paper, the result of the chemical composition testing shows that the main constituent of *A.manihot* bast of the stems is cellulose (41.80 %). *A.manihot* fibers are extracted from *A.manihot* bast by the treatment of degumming. In the degumming process, the key parameters have been optimized. The results of FTIR and XRD analysis indicated that hemicellulose, lignin, pectin and other impurities can be effectively removed by this degumming process, and the main components of *A.manihot* fiber was cellulose much the same as cotton, the crystallinity of *A.manihot* bast and fiber were 50.45% and 63.73% respectively.

Keywords: *A.manihot*, composition, degumming, fiber

INTRODUCTION

Abelmoschus manihot(L.) Medic belongs to the Malvaceae family and widely distributed in the east tropical and subtropical zone. The roots, stems, flowers and seeds of *A.manihot* can all be exploited, collecting edible, medicinal, ornamental values in one. The flower of *A.manihot*, a traditional Chinese medicine, has a broad spectrum of applications as anti-inflammatory, antibacterial, anticoagulant and diuresis effects, which is used to treat several diseases, such as chronic renal disease, diabetic nephropathy etc (Xue *et al.*, 2011). The extensive phytochemical and pharmacological investigation of *A.manihot* reveals that the major bioactive compounds to be the flavonoids such as hyperoside, isoquercitrin, myricetin, hibifolin, gossypetin, quercetin and quercetin-3'-O-glucoside (Lai *et al.*, 2007). Other studies show that the seeds contain crude protein 16.82%, fat 31.16%, amino acids 13.56%, of which the content of the human body essential unsaturated fatty acid 91.8% (Wen-qun *et al.*, 2002). The mucilage extracted from *A.manihot* roots reaches 16% , composed of Arabin 12.30%, Galactan 13.19%, Rhamnosan 8.08%, fucula 16.3%, protein 6.38%, calcium oxalate 17.61% etc., (Xian-rong and Zhao-quan, 1981). Some medicinal substances are obtained from the mucilage.

The studies on secondary structure of the *A.manihot* stems show that a large number of fasciculi distributes in the secondary bast, composed of the fibers by a number ranging from 12 to 50 per fasciculus. Only 1~2 layers parenchyma cells exist between adjacent

fasciculi. Therefore, the fibers extracted from the stems of *A.manihot* can be used as textile materials (Gang-rong, 2003). However, *A.manihot* bast has not yet being exploited at home and abroad. In this paper, the key factors influencing on the degumming are explored based on the chemical composition of *A.manihot* bast.

EXPERIMENTS

Materials: *A.manihot* bast were obtained from Zibo, Shandong Province, China.

Chemical composition of *A.manihot* bast: The chemical composition of *A.manihot* bast is tested according to the GB5889-86 "Ramie Chemical Composition Quantitative Analysis Method".

Degumming process: Fiber extraction process was as follows:

A.manihot bast → pretreatment → washing → alkali treating → washing → bleaching → washing → scutching → drying → *A.manihot* fibers

Process parameters:

- **Pretreatment:** H₂SO₄ (98%) 1ml/L, liquor ratio 1:20, time: 1h, temperature 50°C
- **Alkali treating:** temperature 100°C, liquor ratio 1:20, NaOH: variable (6, 8, 10, 12, 14g/L), time: variable (90, 105, 120, 135, 150 min).
- **Bleaching:** MgSO₄·7H₂O:0.1g/L, liquor ratio 1:20, NaOH 5g/L, H₂O₂: variable (6, 8, 10, 12,

14g/L), time: variable (60, 75, 90, 105, 120 min), temperature 100°C.

- **Washing:** warm water at 40-50°C.

Residual gum content: Referring to the GB5889-86 “Ramie Chemistry Composition Quantitative Analysis Method”, residual gum content M (%) is calculated by Eq. 1:

$$M = \frac{G_a - G_b}{G_a} \times 100\% \quad (1)$$

where, G_a and G_b are the weight of the sample before and after alkali cooking, respectively.

Whiteness: The fiber whiteness were tested with X-Rite 8400 computer-chroma apparatus.

FT-IR spectroscopy (FTIR): The FTIR spectra was carried out with a Nicolet Centaurus IR Microscope in a wavenumber range of 400-4000/cm, distinguish quotiety:2/cm.

X-ray diffraction (XRD): The wide-angle X-ray diffractograms were recorded on a Japan Rigaku D/max 2550 PC X-ray diffractometer operated at 40 kV, 250 mA. The Bragg angle was scanned from 5° to 60°.

RESULTS AND DISCUSSION

Chemical composition: The chemical compositions of A.manihot bast are collected in Table 1. It can be seen that the content of waxy lipid is only 1.76 wt%. Pectin, ash and water-soluble material are 6.88 wt%, 5.21 wt% and 18.5 wt%, respectively. The key factors of the degumming are to remove the large amount of the lignin (13.29 wt%) and the hemicellulose (17.72 wt%). The main constituent of A.manihot bast is cellulose (41.80wt%).

The degumming effects with varial parameters: Alkali treatment plays an important role in the degumming process. The NaOH concentration, treatment time are the key parameters that could affect the degumming effects. The residual gum content is used to check the degumming effects. Figure 1 shows the relation curve between NaOH concentration and residual gum content. At the beginning stage, the gum is removed obviously with the increase of the NaOH concentration. When the NaOH concentration keeps rising, the fiber structure gets ruined, the residual gum content becomes bigger. Considering the cost of the stuff and the intensity of the fiber, the optimum NaOH

concentration is 10g/L. Figure 2 shows the relation curve between alkali treating time and residual gum content. When the treating time gets to 150min, the residual gum content is the lowest. Therefore, we choose 150min as the treating time.

Bleaching is another important part as well as alkali treating in the degumming process. The H_2O_2 can remove the gum and bleach the fiber at the same time. The A.manihot fiber after alkali treatment exhibited a light brown color. Figure 3 shows the relation curve between H_2O_2 concentration and residual gum content, whiteness. The residual gum content represents the degumming effects, and the bleaching effects are demonstrated by the fiber whiteness. The concentration of 12 g/L left the lowest residual gum content. Considering the degumming effects and the fiber whiteness, the optimized H_2O_2 concentration is 12 g/L. The relation curve between bleaching time and residual gum content, whiteness is obtained from Fig. 4. It is clear that the 90min-treating time is the most effective in removing gum. The whiteness reaches the third highest, closing to the highest. According to the results of the figure, 90min was chosen as the bleaching time.

FTIR analysis: Figure 5 gives detailed information regarding the FTIR spectra of;

- A.manihot fiber
- Cotton fiber
- A.manihot bast

In FTIR spectra, the characteristic absorption peaks of cellulose around $1432cm^{-1}$, $1163cm^{-1}$ and $1058cm^{-1}$ shows the same trend among this three samples. The characteristic absorption band of hemicellulose around $1737cm^{-1}$ in curve ‘c’ disappeared in curve ‘a’, this indicated that the degumming treatment can effectively remove hemicellulose from the A.manihot bast. The characteristic absorption band of lignin at about $1637cm^{-1}$ was greatly weakened after delignification. Therefore, based on FTIR results, the hemicellulose, lignin, pectin and other impurities have been effectively removed by degumming process, and the main components of A.manihot fiber was cellulose same as cotton

XRD analysis: Figure 6 shows the X-ray diffraction spectrum of A.manihot fiber (a) and bast (b), which are similar to other natural fibers such as cotton. The appearance of (101) and (002) planes ($2\theta = 15.96^\circ$, 22.60°) determines A.manihot fiber and bast as crystalline cellulose I. The crystallinity of A.manihot

Table 1: Chemical composition of A.manihot bast

Ingredient	Cellulose	Hemicellulose	Lignin	Waxy lipid	Water-soluble material	Pectin	Ash
Content/(%)	41.80	17.72	13.29	1.76	18.5	6.88	5.21

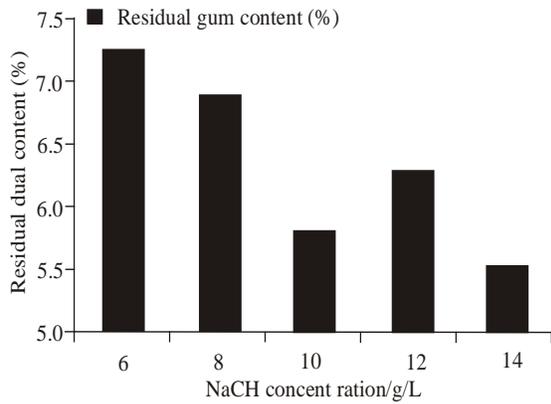


Fig. 1: The relation curve between NaOH concentration and residual gum content

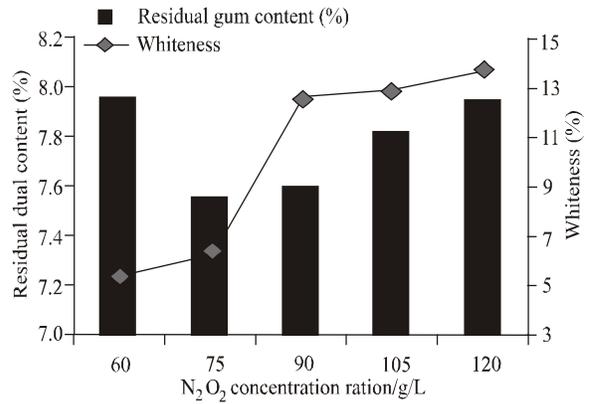


Fig. 4: The relation curve between bleaching time and residual gum content, whiteness

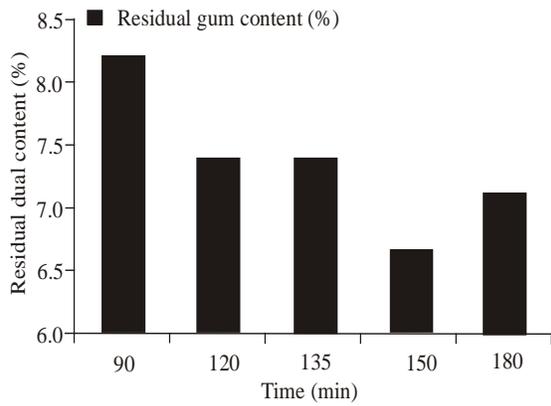


Fig. 2: The relation curve between alkali treating time and residual gum content

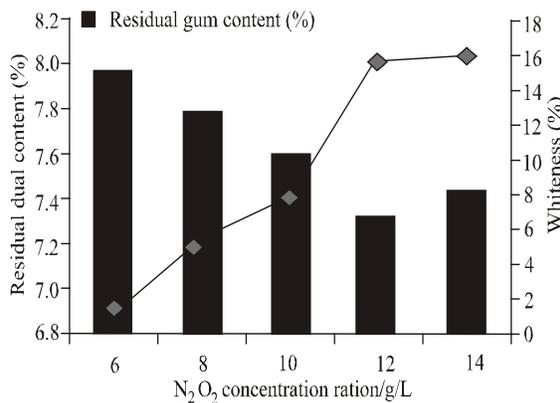


Fig. 3: The relation curve between H₂O₂ concentration and residual gum content, whiteness

fibers and bast are 63.73% and 50.45%, respectively. The crystallinity of A.manihot fibers is closing to cotton (65-67%). The increase of the crystallinity indicates that the hemicellulose, lignin, pectin and other

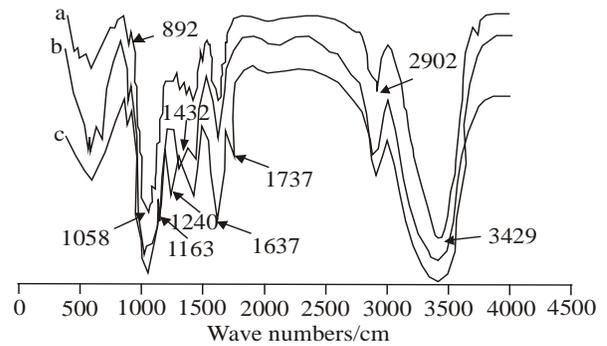


Fig. 5: (a) FTIR spectra of A. manihot fiber (b) Cotton fiber (c) A. manihot bast

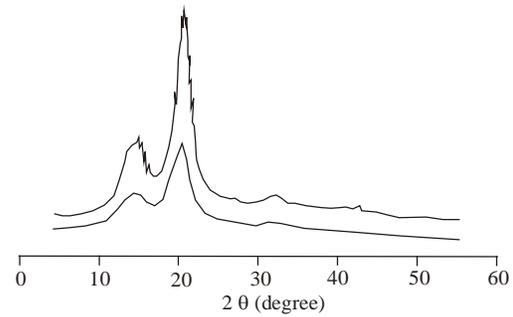


Fig. 6: (a) XRD of A. manihot fiber and (b) A. manihot bast

impurities have been effectively removed from A. manihot bast by degumming process.

CONCLUSION

Based on the analysis of the composition of A.manihot bast, degumming results and FTIR results, the main findings can be summarized as follows:

- Cellulose content of A.manihot bast is 41.80%, and it would have good prospects on textile application after degumming.

- In the degumming process, the key parameters have been optimized, NaOH concentration (10g/L), treating time (150 min), H₂O₂ concentration (12g/L) and bleaching time (90 min) were chosen in the Alkali treating part.
- The results of FTIR and XRD analysis indicated that hemicellulose, lignin, pectin and other impurities can be effectively removed by this degumming process, and the main components of A.manihot fiber was cellulose much the same as cotton, its crystallinity was 63.73% closing to cotton (65-67%).

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