Asian Journal of Chemistry; Vol. 26, No. 6 (2014), 1622-1624

ASIAN JOURNAL OF CHEMISTRY

http://dx.doi.org/10.14233/ajchem.2014.17304

Optimization of Pre-Treatment Condition of Luffa Cylindrica using Orthogonal Experiment†

YANHUA TENG, MAN LU, HONGYAN LI, XIYOU WANG, CHANGGUO XUE* and MINGXU ZHANG

School of Material Science and Engineering, Anhui University of Science and Technology, Huainan 232001, Anhui Province, P.R. China

*Corresponding author: E-mail: chgxue@foxmail.com

Published online: 10 March 2014; AJC-14846

An optimization study on the pre-treatment of luffa cylindrica was described in this paper based on orthogonal experiments. The luffa fibers were treated by mixed treatment (sodium hydroxide and hydrogen peroxide). The effect of treatment on the structure of fibers was inspected by SEM and XRD. Based on the results of the range analysis, the maximum adsorbing yield was found at mixed solution of 20 % NaOH, 10 % H_2O_2 and reaction time 3 h. The SEM and XRD results revealed that the treatment wiped off lignin, pectin and hemicelluloses, more holes and grooves appeared on the surface and decreased the crystallinity index of treated fibers.

Keywords: Luffa fibers, Chemical treatment, Electron microscopy, X-Ray techniques.

INTRODUCTION

The luffa sponges, as dry fruits of luffa cylindrica, are composed of 60 % cellulose, 30 % hemicelluloses and 10 % lignin possessing a netting-like fibrous vascular system with excellent mechanical strength. Recently, the luffa cylindrical fibers have been widely used to produce a new system of sorption to remove the heavy metals, for instance, the removal of copper(II)[1], nickel(II) and dye[2] from aqueous solution. Some other studies also characterized the luffa fibers for other technical applications such as reinforcing composite materials[3].

The reason why luffa fibers can be an ideal adsorbent is its physical property. The valid separation of hemicelluloses and lignin from the luffa sponges makes the fibers more effective in adsorption. Thus, alkalized by sodium hydroxide solution is the popular method and it can change crystalline and dilation of the fibers[3]. In addition, alcohol is necessary, due to the fibers with alkali reaction ability is limited. Although there have been many studies related to the adsorption of alkalized luffa fibers, the optimal conditions have never been thoroughly investigated[4].

In present work, orthogonal testing method was used to study parameters of chemical treatment luffa fibers.

EXPERIMENTAL

Orthogonal array experimental design: The luffa sponges, collected from the locality they grow in, used in this study was selected and reduced dimension into 2-3 mm, then washed with distilled water several times to remove the adhering dirt, dried naturally. Cleaned luffa fibers are treated by mixed solution consisting of both sodium hydroxide and hydrogen peroxide (Table-1) at 25 ºC. The obtained fibers were done in water bath at 90 ºC for 3 h and washed with distilled water until a neutral pH was reached. Then, collected by suction filtration and dried at 70 ºC.

There were the following three variables NaOH concentration (factor A), H_2O_2 concentration (factor B) and reaction time (factor C) that were identified to have larger effects on the results of treatment. Accordingly, applied the orthogonal array and assigned the four factors and three levels as shown in Table-1.

Scanning microscopy: A scanning electron microscopy (SEM) S-3000N is used to examine the morphology and the fibers surface topography before and after chemical treatments.

X-Ray diffraction: The crystallinity index of the tested fibers was calculated from X-ray diffraction patterns recorded on the XRD-6000 X-ray diffractometer.

RESULTS AND DISCUSSION

Adsorbing capacity and statistical analysis: In the present study, the experiments were based on an orthogonal array experimental design. Data analysis was carried out through the range analysis to reflect the optimal reaction conditions and their magnitudes. Optimal conditions were obtained after

†Presented at The 7th International Conference on Multi-functional Materials and Applications, held on 22-24 November 2013, Anhui University of Science & Technology, Huainan, Anhui Province, P.R. China

TABLE-1
LEVELS AND FACTORS AFFECTING THE ALKALIZED TREATMENT

Level	Factors			
	NaOH concentration (in weight, %)	H_2O_2 concentration (in volume, %)	Reaction time (h)	Blank
	10	10	1	1
	15	15	2	2
	20	20	3	3

the orthogonal experiments (Table-2) and subsequent data analysis of adsorbing capacity.

TABLE-2
RESULTS OF ADSORBING CAPACITY (Qe)

Trial No.	Factors				
	A	B	C	Blank	Q_e
1	1	1	1	1	0.114
2	1	2	2	2	0.085
3	1	3	3	3	0.078
4	2	1	2	1	0.121
5	2	2	3	2	0.121
6	2	3	1	3	0.092
7	3	1	3	2	0.164
8	3	2	1	3	0.107
9	3	3	2	1	0.099

As mentioned before, for each factor, a higher mean value k_j indicates that the level has a larger effect on adsorbing capacity. Therefore, the best level for each factor can be determined according to the highest mean value of the experimental condition. In Table-3, the highest adsorbing capacity for each level was clearly distinguished, as the NaOH concentration was 20 %, H_2O_2 concentration was 10 % and reaction time was 3 h, thus the highest at this combination ($A_2B_1C_3$). Compared with the range values (R_j) of different factors, the factors' levels of significance are as follows: reaction time (0.155) > H_2O_2 concentration (0.135) > NaOH concentration (0.075).

TABLE-3
RANGE ANALYSIS DATA OF ADSORBING CAPACITY

Value name	NaOH concentration A	H_2O_2 concentration B	Reaction time C
K_1	0.277	0.399	0.313
K_2	0.334	0.313	0.306
K_3	0.370	0.270	0.363
k_1	0.077	0.159	0.0978
k_2	0.112	0.098	0.093
k_3	0.137	0.073	0.132
R_j	0.075	0.135	0.155

The mean values of each factor were shown in Fig. 1. It should be noted that these graphs were only used to show the trends of each factor, not for predicting other values that were not tested experimentally. Based on the change in the mean value of each factor (Fig. 1a-c), it can be observed that the adsorbing capacity sharply increased from 0.077 to 0.137 in optimal conditions, with the NaOH concentration increasing from 10 to 20 %. For H_2O_2 concentration, the adsorbing capacity decreased dramatically from 0.159 (at 10 %) to 0.073 (at 20 %). Besides, the adsorbing capacity decreased with increasing reaction time, firstly, then increased and reached its maximum point (0.132) at 3 h.

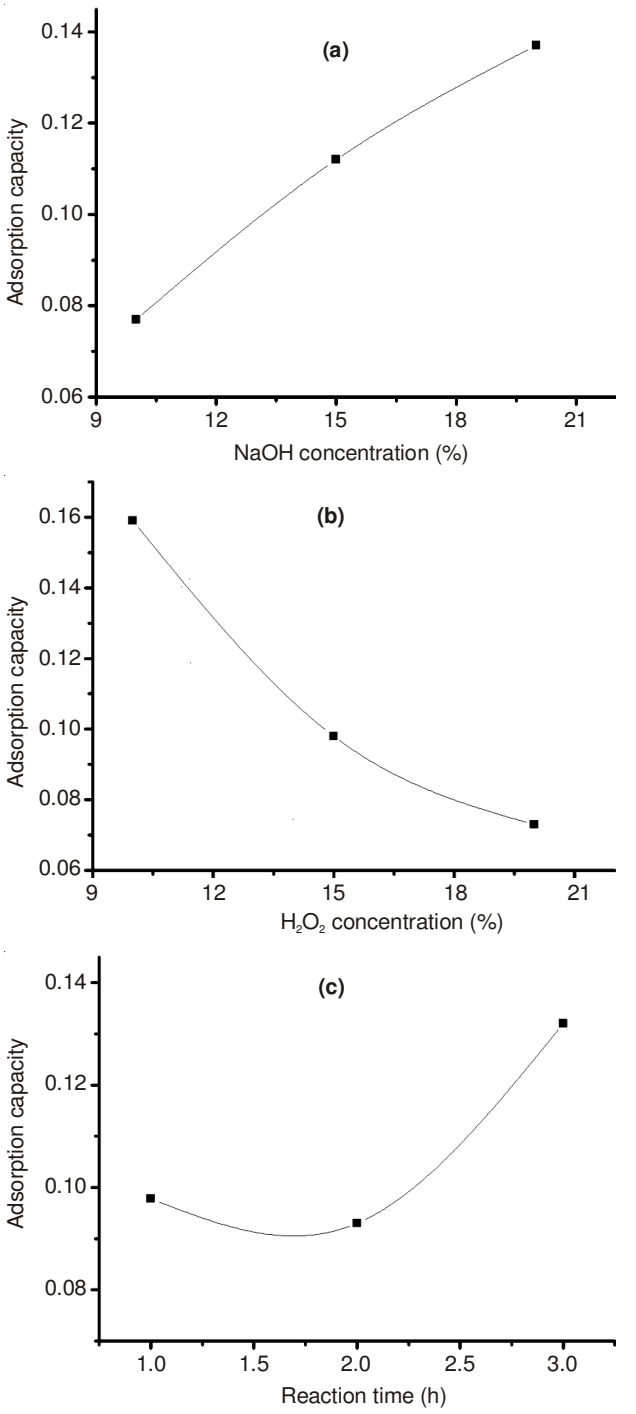

Fig. 1. Relationship between NaOH concentration and adsorbing capacity (a), Relationship between H_2O_2 concentration and adsorbing capacity (b), Relationship between reaction time and adsorbing capacity (c)

Scanning microscopy analysis: The morphology of untreated luffa sample was shown in Fig. 2a. The fibres, formed by fibrils, glued together by gummy substances. Treated this fibers by solution containing 20 % NaOH and 10 % H_2O_2 during 3 h (Fig. 2b). It can be seen that there were holes and grooves on the surface, due to the removal of hemicelluloses, lignin and other substance.

X-Ray diffraction analysis: The results (Fig. 3) showed that the crystallinity index of treated luffa fibers decreased by follow calculation:

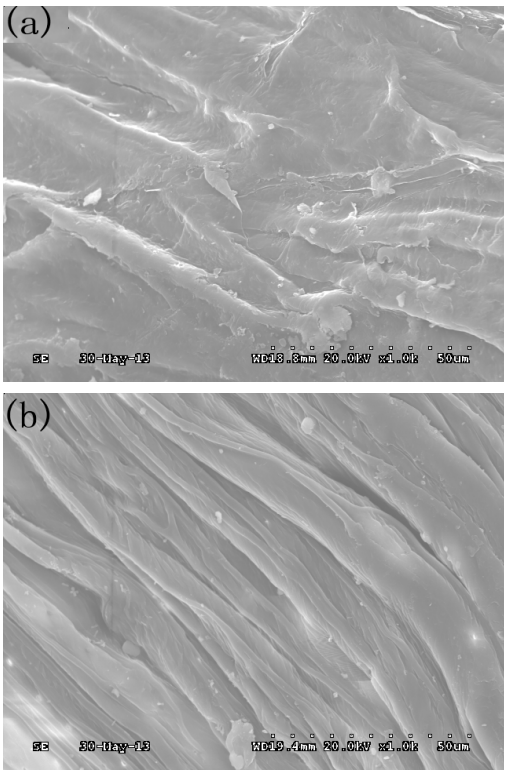

Fig. 2. SEM micrographs of (a) untreated luffa fibres; (b) treated luffa fibers

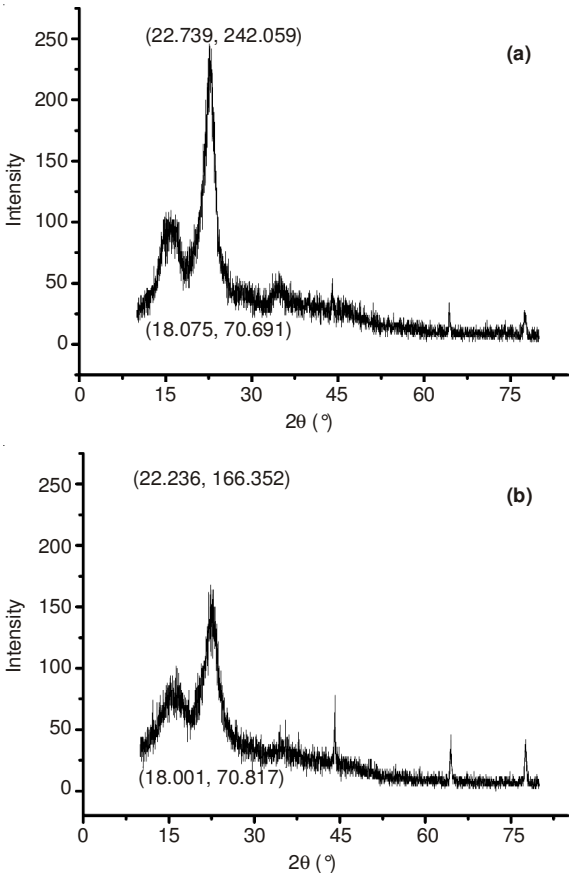

Fig. 3. X-Ray diffraction patterns of untreated (a) and treated Luffa fibres (b)

$$C_1 = \frac{(242.059 - 70.691)}{242.059} \times 100\ \% = 70.796\ \%$$

$$C_2 = \frac{(166.352 - 70.871)}{166.352} \times 100\ \% = 57.429\ \%$$

where C_1, C_2 is the crystallinity of untreated and treated luffa fibers, respectively.

Conclusion

The influences of the chemical treatment parameters on the structure of luffa fibers was investigated in this work. This treatment by using solution containing sodium hydroxide and hydrogen peroxide improves their structural properties and it make them have the better adsorption performance. The optimal treatment process is at 20 % NaOH, 10 % H_2O_2 for 3 h and also the crystallinity reduced.

ACKNOWLEDGEMENTS

This work was supported in part by the National Natural Science Foundation of China (Grant Nos. 11202001), China Postdoctoral Science Foundation funded project (2012M-521210), the Foundation for Young Talents in College of Anhui Province (2012SQRL053ZD) and The College Students' innovation and entrepreneurship training project of China (201210361073 and 201310361059).

REFERENCES

1. Y. Laidani, S. Hanini and G. Henini, *Energy Procedia*, **6**, 381 (2011).
2. H. Demir, A. Top, D. Balköse and S. Ülkü, *J. Hazard. Mater.*, **153**, 389 (2008).
3. C.A. Boynard, S.N. Monteiro and J.R.M. d'Almeida, *J. Appl. Polym. Sci.*, **87**, 1927 (2003).
4. L. Ghali, S. Msahli, M. Zidi and F. Sakli, *Mater. Lett.*, **63**, 61 (2009).