

Chemical Treatments on Hemp Fibre Composites

Hao Wang¹, Mazed Kabir¹, K.T.Lau²

¹Centre of Excellence in Engineered Fibre Composite, Faculty of Engineering and Surveying, University of Southern Queensland, Toowoomba, Australia

²Department of Mechanical Engineering, The Hong Kong Polytechnic University, Hong Kong
Hao.Wang@usq.edu.au

INTRDUCTION

Natural fibres have become an attractive alternative to synthetic fibres in polymer composite structures. This is due to environmental concerns that lead to using more renewable resources in composite structures. Natural fibre constituent contents (cellulose, hemicelluloses and lignin) and hydrophilic nature reduce their compatibility with polymer matrices. Fibre surface modifications using chemical treatments have been successfully used to improve fibre-matrix compatibility. In this study, hemp fibres were treated with alkali and acetyl chemicals. Fibre constituents such as cellulose, hemicelluloses and lignin constituents were separated from treated fibres. The chemical and thermal influences of these constituents on the treated fibres were examined by using scanning electron microscope (SEM) and thermogravimetric analysis (TGA).

EXPERIMENTAL

Chemical treatments

The alkali treatments on hemp fibre used different concentrations of NaOH solutions (0%, 5% and 10%). Hemp fibres were soaked in NaOH solution for 3 hours at room temperature. The fibres were then washed several times with distilled water to allow absorbed alkali to leach from the fibre. The washed fibres were dried at room temperature for 8 hours, and then oven dried at 100°C for another 6 hours. For acetyl treatments, alkali pre-treated hemp fibres were soaked in acetic acid (glacial) and subsequently treated with acetic anhydride at room temperature for 3 hours. Following this treatment, the fibres were washed and then dried in the manner described above.

Chemical analysis

Cellulose, hemicelluloses and lignin constituents from the treated hemp fibre were separated by using sodium chlorite, acetic acid, potassium hydroxide (24% KOH), rectified spirit and sulphuric acid (72% concentration). Treated fibres were crushed into powder and oven dried at 100°C for 2 hours. The dried hemp powder (4 g) was placed in an Erlenmeyer flask and the chemicals were added. The residue from the solution mixture was collected by filtration and washed with distilled water and rectified spirit. The collected residue was oven dried at 105°C for 8 hours before being weighed. The percentage of cellulose, hemicelluloses and lignin were calculated by measuring weight differences.

Microstructure and thermal analysis

SEM micrographs of treated and untreated fibre samples were captured by using scanning electron microscope Model JEOL 6400F. For SEM evaluation, all samples were coated with a layer of gold and 20 kV was used as the accelerating voltage. TGA analysis of fibres was conducted by thermal gravimetric analyser (TGA-Model No. Q500). Between 6 and 10 mg of fibres were taken for analysis. Fibre samples were heated up at a rate of 10°C/min from 10 to 500°C in a helium medium (60 ml/min).

RESULTS AND DISCUSSION

The content of fibre constituents of different treated fibres is presented in Table 1. From Table 1 it can be observed that the hemicelluloses and lignin contents were decreased with NaOH treatment. Removal of hemicelluloses and lignin increased the relative amount of cellulose contents on the treated fibres.

Table I. Fibre constituents before and after chemical treatments.

Fibre treatment	Alkalisation			Alkalisation + acetylation			
	%NaOH	Cellul'	HemiC'	Lignin	Cel'	HC	L
0		77.20	5.40	6.67	81.3	4.96	10.3
5		80.03	4.90	4.86	83.2	4.38	4.0
10		88.35	4.67	3.46	92.6	3.85	2.6

Acetyl treatments on the untreated fibres reduced the hemicelluloses content but the measurement of lignin content increased. This was the result of hemicelluloses initially reacting with acetyl groups, then the acetylated hemicelluloses depositing on lignin. Acetyl treatment on alkali pre-treated fibres showed that the amount of cellulose contents were increased with higher concentrations of (5%, 10%) NaOH pre-treatment. This was due to the extraction of more lignin in two consecutive treatment processes as compared to the NaOH treated samples.

Figure 1 shows the hemp fibre before and after chemical treatments. It can be observed that the gummy polysaccharides of lignin, pectin and hemicelluloses are localized on the surfaces of the untreated fibres (Figure 1a). Alkalisated fibre surfaces (Figure 1b) appeared as bundles of continuous cellulose elementary fibre. Several patterns of partitions formed among these fibre bundles which could not be seen in the original untreated fibres.

This observation clearly indicated that NaOH treatments could remove the hemicelluloses and lignin coverings from the cellulose surfaces. Similarly, acetylated fibre surfaces (Figure 1c) also exhibited the partitioning of fibre bundle and cleaner surfaces in relation to the untreated fibres.

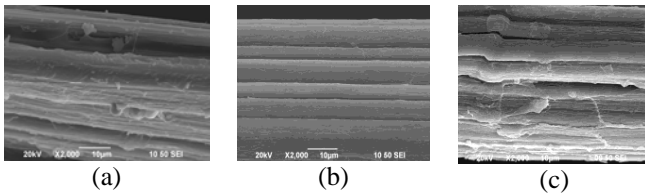


Figure 1. SEM micrograph of (a) untreated (b) alkalisied (c) acetylated fibre surface.

Figure 2 shows the TGA curves of fibre constituents and the untreated fibre. The thermal degradation process of fibre was divided into four stages: moisture evaporation, hemicelluloses degradation, cellulose degradation and lignin decomposition. Moisture evaporates at lower temperature (25-150°C). After the removal of moisture, between the temperature ranges of 150-500°C, the degradation process begins in the cellulose, hemicellulose, and then lignin constituents [1]. The major fibre decomposition was caused in the second stage where large weight losses were recorded. This was primarily caused by the decomposition of hemicellulose and partly by the lignin degradation. The decomposition temperature of cellulose was much higher than hemicellulose and lignin.

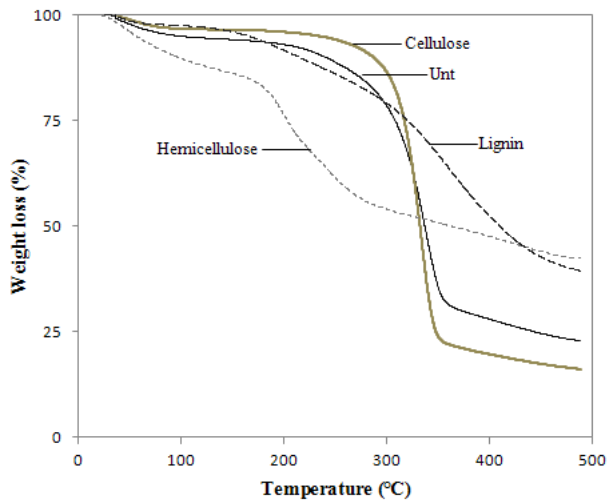


Figure 2. TGA curves of fibre constituents and untreated fibre.

Mechanical properties

The effect of chemical treatments in relation to the interface bonding characteristics between the fibre and matrix can be reflected on their flexural strength properties. Figure 3 shows the bending and shear strengths of untreated and treated composite samples. It is observed that flexural strength of alkali, acetylation and alkali+acetylated fibre composite samples showed 12%,

33% and 17% improvements in strength properties compared to the untreated fibre sample. The improvement in strength can attribute: (1) the increase of cellulose content after treatment; (2) the increase of chemical bonding between fibre and matrix, because the removal of lignin and hemicelluloses expose the more -OH groups to react with matrix [2, 3]. (3) the increase of mechanical bonding because of fibre surface roughness [4].

In the shear test, one side of the V-notched specimen was fixed in the fixture and other side displaced vertically along the fibre direction. It is observed that alkali, acetylation and alkali+acetylated fibre composite samples showed 36.43%, 19.11% and 43.73% higher strength properties compared to the untreated fibre sample. This was presumably due to the higher surface roughness of the treated fibre contributed good bonding with the matrix and provided greater frictional stress transfer along the interface.

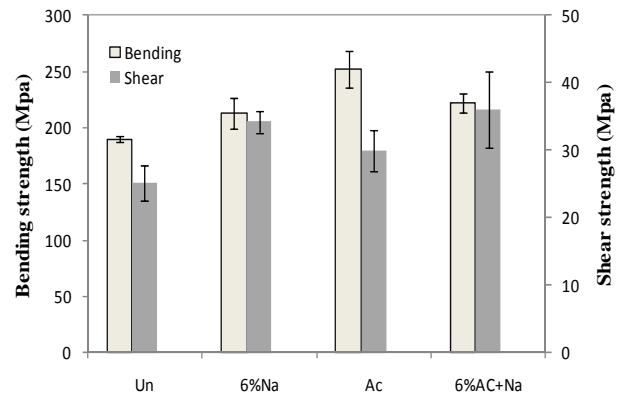


Figure 3. Bending and shear properties of the composites.

CONCLUSION

Chemical treatments change fibre structures by reacting with its constituents. Alkalisiation and acetylation reduce the content of hemicellulose and lignin from the fibre. Thermal analysis of fibre revealed that hemicellulose is the most reactive constituent and degraded faster than that of cellulose and lignin. Cellulose exhibited better thermal stability and lignin was degraded in a wide range of temperatures. The hydrophilic nature of the fibres was predominantly caused by the presence of hemicellulose and then lignin constituents.

KEYWORDS: Hemp fibre, natural fibre composites, chemical treatments

REFERENCES

1. H.J. Kim and Y.G. Eom. *Journal of the Korean Wood Science and Technology*, vol. 20, pp. 59-67, 2001.
2. Y.M. Mwaikambo and M.P. Ansell. *Angew Makromol Chem*, Vol 272, pp 108-16, 1999.
3. M. Abdelmouleh, S. Boufi, M.N. Belgacem and A. Dufresne. *Compos Sci Technol*, Vol 67, pp 1627-39, 2007.
4. G.W. Beckermann and K.L. Pickering. *Composites: Part A*, Vol. 39, pp 979-88, 2008.