Vegetos
https://doi.org/10.1007/s42535-023-00807-x

RESEARCH ARTICLES

The structural and performance characterization of bamboo fibers treated with calcium hydroxide solution

Yong Luo[1,2] · Mustafasanie M Yussof[1] · Zhongwei Peng[2]

Received: 19 May 2023 / Revised: 27 November 2023 / Accepted: 23 December 2023

Abstract

This article observes the micro-structure of bamboo fibers treated with water and calcium hydroxide solution for 5 min respectively, using optical microscope, scanning electron microscope (SEM), Infrared analysis, Thermal analysis, Tensile strength analysis, and examines their chemical structure and quality changes. Results show that after treatment with calcium hydroxide solution, the components of the bamboo fiber surface, such as glue, hemicellulose, and pectin, were effectively removed; The thermal stability of bamboo fiber was improved after treatment with calcium hydroxide solution; The treated bamboo fiber products have a nanometer-level diameter, with an average diameter of 10.33 µm; The tensile strength of bamboo fibers treated with calcium hydroxide solution is approximately 1.53 times higher than that of bamboo fibers treated with water, as observed by the tensile tester.

Keywords Thermogravimetric analysis · Infrared analysis · Calcium hydroxide solution · Electronic scanning · Tension resistance

Introduction

There is increasing research on adding various fibers as external additives to improve the mechanical properties of concrete and enhance its durability (Ahmed et al. 2017; Aravind et al. 2021). Most of these fibers are synthetic, such as polypropylene fibers, carbon fibers, glass fibers, basalt fibers, etc. However, synthetic fibers have some drawbacks, such as the harm of glass fibers to human health and the high cost of carbon fibers, which limit their further use and promotion (Anjum et al. 2013; Salehi et al. 2018). In recent years, renewable plant fibers as composite reinforcement have attracted much attention (AOAC 2012; Benkhnigue et al. 2022). Compared with traditional fibers, plant fibers have the advantages of wide sources, low price, low density, environment-friendliness, renewable, and biodegradability (Aye 2012; Norahmad et al. 2019). China has extremely rich bamboo resources, ranking first in the world. Bamboo fiber, which is made from green bamboo as raw material and has a green and environmentally friendly production process, is basically the residual waste of bamboo factories and has high mechanical properties (Bouzid et al. 2017; Nwokoro 2015).

Bamboo fibers have good biodegradability and high ecological stability, which can improve the impact resistance and fracture toughness of concrete. They are low-cost waste materials. However, bamboo fibers may degrade and mineralize in the alkaline environment of the cement matrix, and their water absorption characteristics can weaken their bond strength with cement concrete (Chaachouay et al. 2022; Choy et al. 2019; Ohizua et al. 2017). Therefore, some treatments need to be applied to bamboo fibers, with two methods: (1) alkali treatment, which can remove part of lignin, most hemicellulose, pectin, and wax on the surface of bamboo fibers, and increase their surface roughness to enhance the mechanical interlocking force between them and cement concrete (Dwivedi et al. 2020; HCP 2018; Ansari et al. 2023); (2) cellulose nanofiber-grafted modified bamboo

✉ Mustafasanie M Yussof
cemustafa@usm.my

Yong Luo
luoyong@student.usm.my

Zhongwei Peng
59388898@qq.com

1 School of Civil Engineering, Engineering Campus, Universiti Sains Malaysia, Nibong Tebal, Penang 14300, Malaysia

2 Smart Construction Engineering Center, Fujian Forestry Vocational and Technical College, Nanping 353000, China

Published online: 08 February 2024

fibers, which can modify the original defects on the surface of bamboo fibers, reduce the porosity of the cement matrix, and further enhance the compressive strength of bamboo fiber concrete (Nissen et al. 2021; Orch et al. 2021; Plamada and Vodnar 2022). However, the selection of the alkali treatment solution is a difficult point. Currently, most researchers use sodium hydroxide solution to treat bamboo fibers. However, the concentration of sodium hydroxide solution has not been effectively studied at home and abroad (Singh et al. 2014; Shubham et al. 2019). Sodium hydroxide solution is a strongly corrosive solution. If the concentration is too high, it will destroy the fiber structure of bamboo fibers, which not only fails to enhance the mechanical properties of concrete but also reduces them, becoming a burden (Singh et al. 2020). On the other hand, chemical modification of bamboo fibers is too expensive, which is not conducive to market promotion and application. Therefore, how to treat bamboo fibers remains a difficult point (Tan 2019; Ukpo et al. 2017).

The methods of optical microscope and electron scanning microscope can characterize the microstructure composition of bamboo fibers. Infrared analysis can determine the content of hemicellulose on the surface of bamboo fibers and determine the chemical composition of bamboo fibers. Thermal analysis can characterize the level of stability of bamboo fibers in different states. Tensile strength analysis can measure the tensile strength of bamboo fibers in different states. These testing methods play a very important role in the analysis of the tensile strength mechanism of bamboo fibers, and can explain the deep-level reasons for the changes in tensile strength of bamboo fibers under different treatment methods very well.

This chapter uses a weakly corrosive calcium hydroxide solution to treat bamboo fibers, and analyzes and characterizes the bamboo fibers and their treatment process through scanning electron microscopy (SEM), Fourier transform infrared spectroscopy (FTIR), thermal analysis (TG), and tensile strength testing.

Table 1 Physical property parameters of bamboo fiber

Material	Fiber diameter (mm)	Fibre length (mm)	Tensile strength (MPa)	Modulus of elasticity (GPa)	Density (g/cm3)
Bamboo fiber	1.5	22.5/45/67.5	150	20	0.68

Calcium hydroxide: Food-grade 95% white powder calcium hydroxide is used, take a small amount of powder and dissolve it in water to prepare a calcium hydroxide solution by stirring

Materials and methods

Materials of study

Bamboo fiber: The experiment uses shaving bamboo fibers aged 3–5 years, purchased from Nanping, Fujian. Shape: flocculent bamboo fiber, about 3–4 cm. Its physical properties (theoretical) are shown in Table 1.

Equipment of study

High-speed centrifuge, Neofuge 15, Shanghai Lishen Scientific Instrument Co., Ltd.; Electric Heating Constant Temperature Blowing Drying Oven, DHC-9070 A, Shanghai Jinghong Laboratory Instrument Co., Ltd.; Synchronous Thermal Analyzer, SDTQ600, TA Instruments, USA; Freeze Dryer, FD-2, Beijing Boyikang Instrument Equipment Co.,Ltd.; Fourier Transform Infrared Spectrometer, AVATAR330, Nicolet, USA; Scanning Electron Microscope, TESCAN, Czech Tescan Instrument Company; X-ray Diffraction Instrument, D8, Bruker, Germany.

Preparation of bamboo fiber

Take about some bamboo fibers and treat them with water and calcium hydroxide solution separately for 5 min, then put them in a drying oven for 12 h (at 90 degrees Celsius) and get them out for later using.

Structure characterization method

Optical microscopy analysis: Adhere a small amount of the sample to a glass slide, observe the surface structure changes of the sample, and analyze the similarities and differences.

Scanning electron microscopy analysis: Adhere a small amount of the sample to conductive adhesive, spray it with gold in a vacuum, observe the surface structure changes of the sample, and characterize the morphology of the sample (Danielsen et al. 2020).

Infrared analysis: Use a Fourier transform infrared spectrometer for infrared spectroscopy scanning. Firstly, grind the sample and mix it with potassium bromide, and then scan it with a potassium bromide crystal as a control, scanning 28 times with a resolution of 4 cm^{-1} (Chaachouay et al. 2023).

Thermal analysis: Test using a synchronous thermal analyzer, with a nitrogen flow rate of 50 mL/min,heating rate of 100 C/min, and a temperature range of 30 – 1,000 °C (Bouzid et al. 2017).

Tensile strength analysis: Hang the sample on the hook of the tensile tester, keeping the tension direction consistent with the spring direction. After the bamboo fiber is broken, observe the tensile strength.

Nano Measurer 1.2 software is used for bamboo fiber diameter statistical analysis to calculate the average diameter.

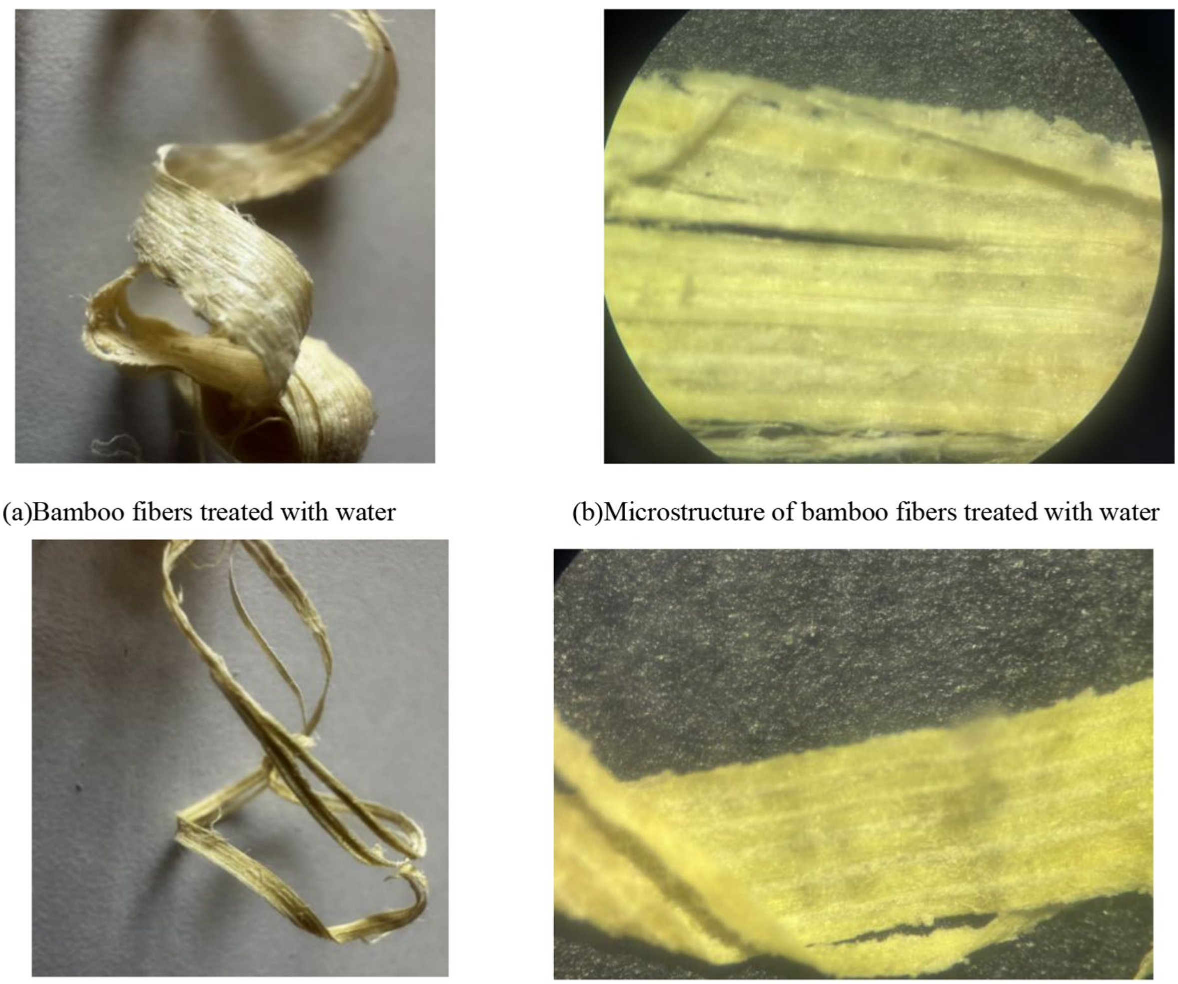
Results and discussion

Analysis of optical microscope structure

The magnified structures of bamboo fiber treated with water and treated with calcium hydroxide solution is shown in Fig. 1.

From the image, it can be observed that the bamboo fiber treated with water appears to have a light yellow color, while the one treated with calcium hydroxide solution has a deeper yellow color. Moreover, there are more impurities attached to the surface of the bamboo fiber treated with water, such as pectin and waxy substances, whereas the surface of the fiber treated with calcium hydroxide solution has fewer impurities, with no visible pectin or waxy substances. The surface is flat and clean.

Reason: The bamboo fiber treated with water merely removes surface dust and simple adhesions, while solid adhesions and epiphytic impurities are not effectively removed. In contrast, the bamboo fiber treated with calcium hydroxide solution has a deeper color and is cleaner, not only removing simple adhesions but also eliminating the pectin, sugar and other impurities on the surface. In addition, the treated bamboo fiber is lighter in weight, has a rougher surface, and exhibits a more detailed and prominent fiber structure, which enhances its tensile strength. This is because calcium hydroxide solution is an alkaline solution that easily dissolves impurities such as pectin and sugar, while preserving the fiber structure of bamboo and



(a)Bamboo fibers treated with water

(b)Microstructure of bamboo fibers treated with water

(c) Bamboo fibers treated with calcium hydroxide solution

(d)Microstructure of bamboo fibers treated with calcium hydroxide solution

Fig. 1 Microstructure of bamboo fiber increasing the fiber content in the bamboo stem, providing better toughness to the bamboo fiber.

Analysis of electronic scanning structure

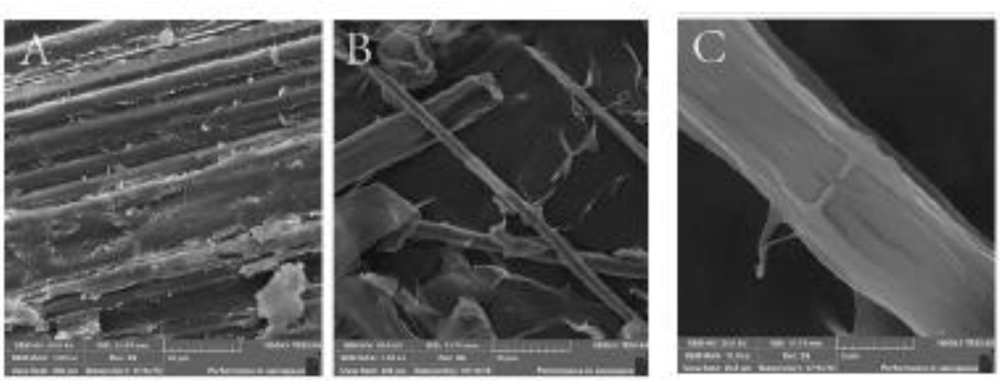
Figure 2 shows the SEM images of the products in each stage of bamboo fiber extraction. As can be seen from Fig. 2A, the bamboo fibers treated with water are arranged in a straight fiber bundle, and there are sheet adhesive materials on the surface of the fiber bundle, or they are formed into chunks. After calcium hydroxide solution treatment, the fiber bundle disintegrates and a single fiber appears. There are bamboo leaf-like substances around the fiber, which may play the role of adhesion and wrapping substances in the bamboo structure, such as gum, lignin or hemicellulost (El Khomsi et al. 2022). The central part of a single fiber (Fig. 2B) is enlarged by 10,000 times, as shown in Fig. 2C, showing bamboo joints and twigs branching out beside the bamboo joints. The diameter of a single fiber here is close to 10 µm. Through statistical analysis, the average diameter of bamboo fiber is 10.33 µm, as shown in Table 2.

Influence of treatment process on chemical structure

The structural and compositional changes of bamboo fiber after treatment were clearly reflected in the infrared spectrum. As shown in Fig. 3, there were changes in the peaks of bamboo fiber raw material and calcium hydroxide solution treated bamboo fiber at 3050 cm^{-1}, with a certain degree of shift. In addition, after calcium hydroxide solution treatment, the peak at 1735 cm^{-1} disappeared, which is the absorption peak of hemicellulose. The peak at around 1650 cm^{-1} of the bamboo fiber raw material was weak, while it was strengthened and formed a double-headed peak after calcium hydroxide solution treatment. Meanwhile, the peak at 1260 cm^{-1} gradually weakened, which is the absorption peak of pectin. The small peak at 1500 cm^{-1} did not disappear after calcium hydroxide solution treatment, which is the peak of lignin. The small peak near 1450 cm^{-1} gradually increased during the treatment and finally formed a prominent peak. The peaks at 1,200, 1,080, and 1,060 cm^{-1} began to strengthen and differentiate after each stage of treatment, which are all characteristic peaks of cellulose. Therefore, these changes indicate that calcium hydroxide solution treatment removes hemicellulose from bamboo.

Influence of the processing process on thermal stability

From the thermal analysis curve in Fig. 4, it can be seen that bamboo fiber begins to lose weight at around 100 ℃, which is mainly due to the evaporation of water. At around 200 ℃, the second stage of weight loss begins. Starting from around 250 ℃, the weight loss of the three samples begins to differentiate. The raw bamboo fiber loses close to 70% of its weight at 380 ℃, with the weight loss rate remaining relatively stable and close to zero until the end of the test period. In contrast, the bamboo fiber treated with calcium hydroxide solution exhibits slow changes in mass starting from around 150 ℃. There is a significant weight loss at around 500 ℃, which continues until around 600 ℃. This is followed by a slow reduction in weight loss rate, indicating an increase in the stability of the bamboo fiber after treatment with calcium hydroxide solution. Previous studies have shown that the presence of hemicellulose and lignin can lower the temperature at which bamboo fiber begins to degrade. Increasing the cellulose content by removing these two components can lead to an increase in the pyrolysis temperature.



A,B,C are raw materials (1 000x), alkali solution treatment (1 000x, 10000x),

Fig. 2 Morphology of raw bamboo fiber

Table 2 Statistical report for diameter of bamboo fiber

Sampling number	1	2	3	4	5	6	7	8	9	10	max	min	aver
Diameter/um	12.02	7.38	11.13	8.32	13.3	12.2	10.1	9.76	10.25	7.67	13.3	7.67	10.2

Influence of tension during processing

Bamboo fibers treated with water and treated with calcium hydroxide solution were divided into two groups. Three experiments were carried out on each group of bamboo fibers, and the average value was taken. The experimental data were shown in Table 3.

As shown in the table, bamboo fibers treated with water underwent three tensile strength tests, with the measured values of 0.31 N, 0.33 N, and 0.32 N, respectively. The average tensile strength calculated from these tests is 0.32 N. On the other hand, bamboo fibers treated with calcium hydroxide solution underwent three tensile strength tests, with the measured values of 0.48 N, 0.50 N, and 0.49 N, respectively. The calculated average tensile strength from these tests is 0.49 N. The tensile strength of bamboo fibers treated with calcium hydroxide solution is higher than that of bamboo fibers treated with water, as shown in the bar chart and line chart in Fig. 5, respectively. The calculated tensile strength of bamboo fibers treated with calcium hydroxide solution is approximately 1.53 times higher than that of bamboo fibers treated with water.

Reason: The surface of bamboo fibers contains impurities such as pectin. The treatment with water only cleans away the surface dust and other simple impurities, but does not remove pectin, sugar, and other impurities, increasing the overall weight of the bamboo fibers and reducing their tensile strength. The treatment with calcium hydroxide solution, however, dissolves the pectin and sugar on the surface of bamboo fibers, making the fiber structure more prominent, reducing the overall weight of the bamboo fibers, and enhancing their tensile strength.

Discussion

Bamboo fibers treated with calcium hydroxide solution remove impurities such as pectin and hemicellulose from the surface of the fibers. Therefore, the bamboo fiber surface observed under an optical microscope and an electron scanning microscope appears clean and free of impurities. Infrared analysis shows that bamboo fibers treated with water and bamboo fibers treated with calcium hydroxide solution exhibit different waveforms. This is because the calcium hydroxide solution removes the hemicellulose on the surface of the bamboo fibers, resulting in the disappearance of its characteristic peaks. This further proves the importance of calcium hydroxide in purifying bamboo fibers. Thermogravimetric analysis reveals that the overall mass of bamboo fibers treated with calcium hydroxide solution is lower than that of bamboo fibers treated with water. This is because the calcium hydroxide solution removes impurities

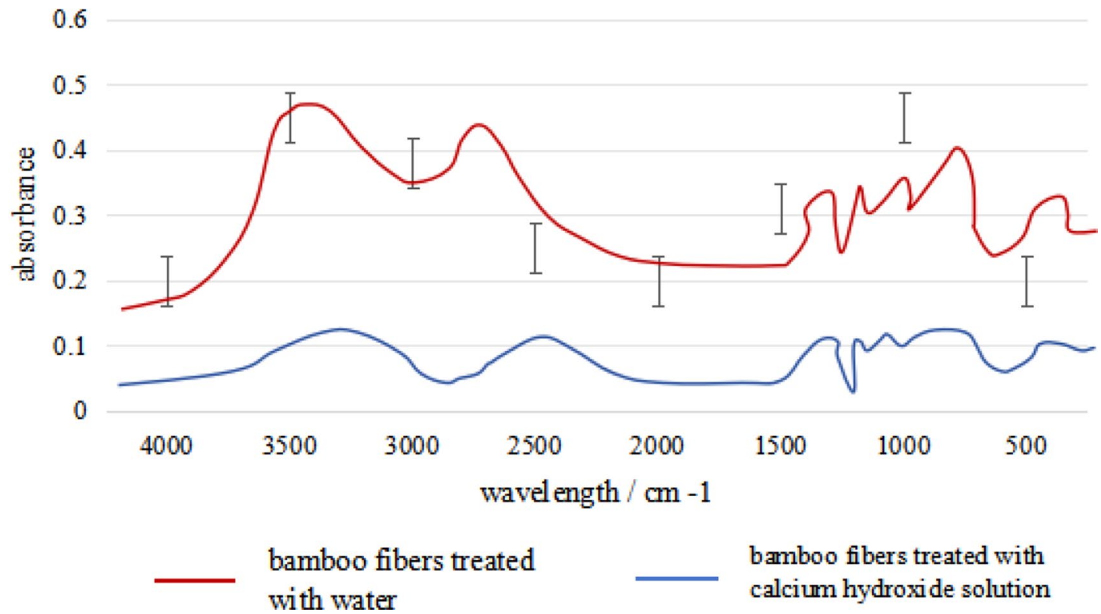


Fig. 3 FTIR spectra of bamboo samples at all stages

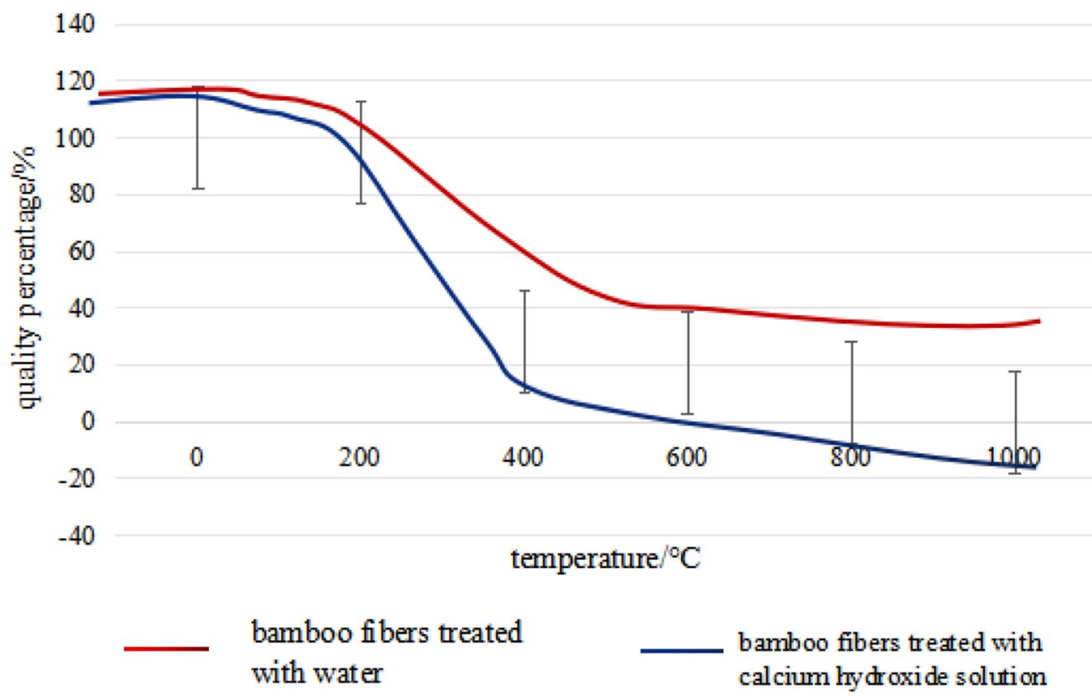


Fig. 4 TG analysis of bamboo at different stages

Table 3 Tensile force of bamboo fiber (N)

Type of bamboo fiber	Sample 1	Sample 2	Sample 3	Aver
Bamboo fiber treated with water	0.31	0.33	0.32	0.32
Bamboo fiber treated with calcium hydroxide	0.48	0.50	0.49	0.49

from the surface of the bamboo fibers, reducing their overall mass and demonstrating stronger stability. Tensile strength analysis of bamboo fibers shows that those treated with calcium hydroxide solution exhibit significantly higher tensile strength compared to those treated with water. This indicates that calcium hydroxide solution enhances the tensile strength of bamboo fibers.

Li Ming aimed to improve the compatibility between well cement and bamboo fibers by modifying the fibers with

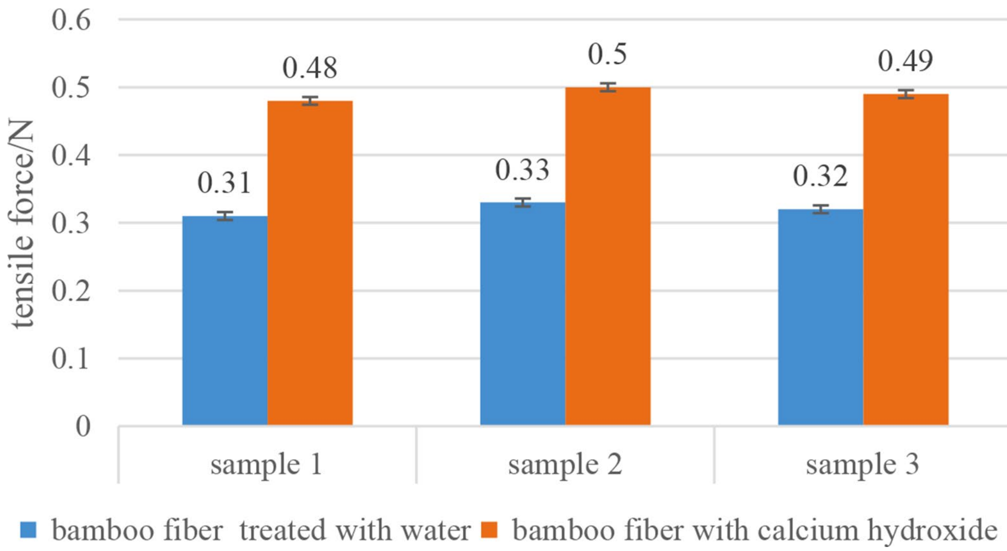


Fig. 5 Tensile strength of bamboo fibers in different treatments

sodium hydroxide solution. Through infrared spectroscopy analysis and scanning electron microscopy (SEM) observation, it was found that the surface of the alkali-treated bamboo fibers showed deep grooves, with hemicellulose and lignin being removed. The fiber visibility and surface area increased, while the surface polarity decreased, thereby improving its dispersibility, affinity, and adhesion with cement. However, this study did not thoroughly explore the enhanced performance of cement with bamboo fibers modified using different concentrations of sodium hydroxide solution, nor did it find the optimal sodium hydroxide concentration for fiber modification. Further research is needed to investigate whether higher concentrations of sodium hydroxide can cause corrosion of the bamboo fibers (Li Ming et al. 2014). This study uses a weak alkaline calcium hydroxide solution, which removes impurities from the surface of bamboo fibers without damaging the fiber structure, thereby further enhancing their tensile strength.

Conclusion

This paper adopted two different treatments using water and calcium hydroxide solution and characterized bamboo fibers. The treated bamboo fiber products have a nanometer-level diameter, with an average diameter of 10.33 μm; After treatment with calcium hydroxide solution, the components of the bamboo fiber surface, such as glue, hemicellulose, and pectin, were effectively removed; Thermal analysis shows that the thermal stability of bamboo fiber was improved after treatment with calcium hydroxide solution; The tensile strength of bamboo fiber treated with calcium hydroxide solution is approximately 1.53 times higher than that of bamboo fiber treated with water.

Author contributions Conceptualization: YL; Methodology: YL; Formal analysis and investigation: ZP; Writing - original draft preparation: YL; Writing - review and editing: YL; Funding acquisition: Smart construction Engineering Center project (2021LK11), Fujian Forestry Vocational & Technical College, Nanping 353000, China; Resources: none; Supervision: MBMY.

Funding This study was funded by Smart construction Engineering Center project (2021LK11), Analysis and research on Influencing factors of special concrete mix design, Fujian Forestry Vocational & Technical College, Nanping 353000, China.

Data availability The datasets generated during and analysed during the current study are available from the first author on reasonable request.

Declarations

Ethical approval This is an observational study. The Research Ethics Committee has confirmed that no ethical approval is required.

Financial interests The authors declare they have no financial interests.

References

Ahmed RA, He M, Aftab RA, Zheng S, Nagi M, Bakri R, Wang C (2017) Bioenergy application of Dunaliella salina SA 134 grown at various salinity levels for lipid production. Sci Rep 7(1):8118

Anjum V, Ansari SH, Naquvi KJ, Arora P, Ahmad A (2013) Development of quality standards of Carica papaya Linn. Leaves Pharm Lett 5:370–376

Ansari MKA, Iqbal M, Chaachouay N, Ansari AA, Owens G (2023) The concept and status of medicinal and aromatic plants: history, pharmacognosy, ecology, and conservation. Plants as medicine and aromatics. CRC Press, New York p, pp 129–144

AOAC (2012) Ofcial methods of analysis, Association of ofcial analytical chemist 19th edn. Washington D.C., USA

Aravind SM, Wichienchot S, Tsao R, Ramakrishnan S, Chakkaravarthi S (2021) Role of dietary polyphenols on gut microbiota, their metabolites and health benefts. Food Res Int 142:110189

Aye PA (2012) Efects of processing on the nutritive characteristics, anti-nutritional factors and functional properties of Cnidoscolus aconitifolius leaves (Iyana Ipaja). Am J Food Nutr 2(4):89–95

Benkhnigue O, Chaachouay N, Khamar H, El Azzouzi F, Douira A, Zidane L (2022) Ethnobotanical and ethnopharmacological study of medicinal plants used in the treatment of anemia in the region of Haouz-Rehamna (Morocco). J Pharm Pharmacogn Res 10(2):279–302

Bouzid A, Chadli R, Bouzid K (2017) Étude ethnobotanique de la plante médicinale Arbutus unedo L. dans la région de sidi Bel Abbés en Algérie occidentale. Phytothérapie 15(6):373–378

Chaachouay N, Azeroual A, Douira A, Zidane L (2022) Ethnoveterinary practices of medicinal plants among the Zemmour and Zayane tribes, Middle Atlas, Morocco. S Afr J Bot 151:826–840

Chaachouay N, Azeroual A, Ansari MKA, Zidane L (2023) Use of plants as medicines and aromatics by indigenous communities of Morocco: pharmacognosy, ecology and conservation. Plants as medicine and aromatics. CRC Press, New York, pp 33–44

Choy KW, Murugan D, Leong XF, Abas R, Alias A, Mustafa MR (2019) Flavonoids as natural antiinfammatory agents targeting nuclear factor-kappa B (NFκB) signaling in cardiovascular diseases: a mini review. Front Pharmacol 10:1295

Danielsen M, Nebel C, Dalsgaard TK (2020) Simultaneous determination of land D-amino acids in proteins: a sensitive method using hydrolysis in deuterated acid and liquid chromatography-tandem mass spectrometry analysis. Foods 9(3):309

Dwivedi MK, Sonter S, Mishra S, Patel K, Singh PK (2020) Antioxidant, antibacterial activity, and phytochemical characterization AOAC. Ofcial methods of analysis of AOAC international, 17thedn. AOAC international, Washington DC, USA

El Khomsi M, Dandani Y, Chaachouay N, Hmouni D (2022) Ethnobotanical study of plants used for medicinal, cosmetic, and food purposes in the region of Moulay Yacoub, Northeast of Morocco. J Pharm Pharmacogn Res 10(1):13–29

HCP (2018) Haut-commissariat Au plan, Monograpie De La région Tanger Tétouan Al Hoceima. Direction Régionale de Tanger-Tétouan-Al Hoceima

Li M, Meng L et al (2014) Study on toughening of well cement with alkali-treated modified bamboo fibers. Funct Mater 45(13):13087–13091

Nissen SH, Schmidt JM, Gregersen S, Hammershøj M, Møller AH, Danielsen M, Stødkilde L, Nebel C, Dalsgaard TK (2021) Increased solubility and functional properties of precipitated alfalfa protein concentrate subjected to pH shift processes. Food Hydrocolloids 119:1–12

Norahmad NA, Razak MR, Misnan NM, Jelas NH, Sastu UR, Muhammad A (2019) Efect of freeze-dried Carica papaya leaf juice on infammatory cytokines production during dengue virus infection in AG129 mice. BMC Complement Altern Med 19(44):1–10

Nwokoro SO (2015) From the Known to the Unknown: Some Glimpses and Dances of a Scientist. In: 160th Inaugural Lecture Series of the University of Benin, pp 26–27

Ohizua ER, Adeola AA, Micheal AI (2017) Nutrient composition, functional, and pasting properties of unripe cooking banana, pigeon pea, and sweet potato four blends. Food Sci Nutrition 5:750–762

Orch H, Chaachouay N, Douiri EM, Faiz N, Zidane L, Douira A (2021) Use of medicinal plants in dermato-cosmetology: an ethnobotanical study among the population of Izarène. Jordan J Pharmaceutical Sci 323–340

Plamada D, Vodnar DC (2022) Polyphenols—gut microbiota interrelationship: a transition to a new generation of prebiotics. Nutrients 14(1):137

Salehi B, Mishra AP, Nigam M, Sener B, Kilic M, Sharif-Rad M, Fokou PVT, Martins N, Sharif-Rad J (2018) Resveratrol: a double-edged sword in health benefts. Biomedicine 6:91

Shubham S, Mishra R, Gautam N, Nepal M, Kashyap N, Dutta K (2019) Phytochemical analysis of papaya leaf extract: screening test. EC Dent Sci 18(3):485–490

Singh S, Varshney VK, Wahl N, Khan LH (2014) Isolation and biochemical analysis of leaf protein concentrates from the leaves of Shorea robusta. Pak J Nutr 13(9):546–553

Singh SP, Kumar S, Mathan SV, Tomar MS, Singh RK, Verma PK, Kumar A, Kumar S, Singh RP, Acharya A (2020) Therapeutic application of Carica papaya leaf extract in the management of human diseases. Daru 28(2):735–744

Tan SS (2019) Papaya (Carica papaya L.) seed oil. In: Ramadan M (ed) Fruit oils: chemistry and functionality. Springer, Cham

Ukpo GE, Owolabi MA, Imaga NO, Oribayo OO, Ejiroghene AJ (2017) Efect of Carica papaya (Linn) aqueous leaf extract on pharmacokinetic profle of ciprofoxacin in rabbits. Trop J Pharm Res 16(1):127–134