



Effect of Microwave Radiation on The Macromolecular, Morphological and Crystallographic Structures of Plantain (*Musa paradisiaca*) Fibre

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Abstract

The physical properties of natural fibres extracted from plantain (*Musa paradisiaca*) fibres were modified using microwave irradiation under different conditions in terms of power and time. To investigate the effect of this modification, macromolecular parameters of the fibre are characterised by wide angle X-Ray Diffraction (XRD), Fourier Transform Infra-red (FTIR), Scanning Electron Microscope (SEM) and Thermogravimetric analysis (TGA). XRD analysis shows a significant change in the macromolecular and the crystallographic parameters of the fibre, while SEM image reveals the serious damaging effect on the surface of the fibre, due to degradation and rupturing of fibre cells at higher power and time of microwave irradiation. FTIR spectra confirm that the chemical structure of the microwave treated fibre does not change. Thermal analysis shows increase thermal stability at optimised microwave power and time. However, the degradation of the structural component and crystallinity of the fibre are observed at higher power and higher treatment period.

1. Introduction

The use of chemical and physical methods has been found to reduce the inherent hydrophobicity of natural fibre [1]. Environmental and health issues are major concerns on the use of hazardous chemicals for modifying the fibre and its disposal. Hence, the interests in non-hazardous chemical and physical modification of the natural fibres are becoming an attractive area of research. In this regard, the use of microwave irradiation on natural fibre is gaining substantial attention. Microwaves are electromagnetic waves that lie between radio and infrared frequency regions in the electromagnetic spectrum. The majority of the microwave frequencies are dedicated for communications and radar purposes, 915 MHz, 2.45 GHz, 5.8 GHz, and 20.2–21.1 GHz are frequencies designated for industrial, scientific, and medical uses [2,3]. The ability of some materials to convert microwaves into heat makes them suitable for microwave processing [4]. Unlike conventional heating where the heat is concentrated along the materials surface rather than the interior, microwave energy is absorbed by the material and converted to heat [5-6].

Over the years different natural occurring fibres such as hemp [7], jute [8], flax [9], kenaf [10], coconut [11, 12] and banana fibre [13,14] have been studied. Xue *et al.* [15] reported in their study of microwave irradiated wool fabric, that microwave heating is more efficient than conventional heating. Mahmoodi *et al.* [16] reported the potential production of new environment-friendly textile fibres in their study of silk degumming using microwave irradiation. Annapurna *et al.* [1], reported that Microwave irradiation could affect the macromolecular parameters, crystallographic structures and morphology of sisal fibre. Singh *et al.* [17] reported the suitability of Microwave irradiation on joining of a green composite. The complete transformation of cellulose lattice type I into cellulose lattice type II without any heating using microwave was also reported [18]. Researchers such as Chimekwene *et al.* [19], Okafor *et al.* [20], Alvarez-López *et al.* [21], Edith *et al.* [22] and Imoisili *et al.* [23], have all studied Plantain as a source of natural fibre and their various applications. The above research findings have indicated that microwave radiation is an effective treatment method for natural fibre as well as fibre polymer composite due to their beneficial effects on processing time and mechanical properties. There is, however, hardly any report on the effect of the microwave irradiation on the macromolecular structure of plantain (*Musa paradisiaca*) fibre. The objective of this work is to study the effectiveness of microwave radiation on plantain (*Musa paradisiaca*) fibres modification. Special emphasis is made on the study of the macromolecular parameters of the pristine and microwave (MV) treated fibre. An attempt has been made to analyse the modification of the fibre in terms of macromolecular parameters.

2. Experimental details

2.1. Extraction of fibre

Plantain fibre was collected from a local farm from the south-west Nigeria State of Ondo and extracted from the pseudostem using the water retting methods as reported by Paridah et al [24]

2.2 Treatment of Fibre

The microwave treatment of the fibres was carried out in a microwave oven (Sanyo Electronics Model EM 51052) having an adjustable power of (550 – 750) W with a microwave frequency of 2450 MHz. The extracted fibres were treated with microwave irradiation at a power setting of 550w and 750w for different treatment periods (2 and 4 min). The fibres were removed from the oven and cooled under vacuum for 24 hours [1]. The microwave treated (MV) fibres were designated as 550W2, 550W4, 750W2, 750W4. The prefixes of 'W' denote the power setting whereas the suffixes of it represents the microwave irradiation time on the fibres in minutes, while untreated fibres were designated as UT.

2.3 Characterization

2.3.1 XRD analysis

In order to identify the effect of microwave treatment on the crystallographic structures of plantain (*MusaParadisiaca*) fibres, wide angle X-Ray Diffraction (XRD) was carried out using a Siemens D-5000 powder diffractometer with monochromatic CuK α radiation ($k = 1.5418 \text{ \AA}$), using an acceleration voltage of 40 kV and 40 mA. The diffraction angle was scanned from 5° to $50^\circ 2\theta$, at a step size of 0.05° , and a rate of $5.00^\circ/\text{min}$. The degree of Crystallinity was measured using Bruker/Siemens diffraction software package (Topaz Rietveld Refinement software). While crystallite size was determined using the Debye Sherrer's formula [1].

$$D = K \lambda / \beta \cos \theta,$$

Where; D is the particle size (\AA), K = Constant ($k = 0.89$), λ = Wavelength of the incident X-ray beam ($\lambda_{\text{CuK}\alpha} = 1.5418 \text{ \AA}$), β = Full width at half maximum of the X-ray diffraction peaks (rad) and θ = Bragg angle of X-ray diffraction peak

2.3.2 Bulk density measurement.

The density measurements of the fibres were done as per ASTM D3800-99.

2.3.3 FTIR Analysis

Chemical compositions of the raw and treated fibres were investigated by the Tian Jin Gang Dong FTIR 650-spectrometer spectrum in the mid IR range i.e. from 400 cm^{-1} to 4000 cm^{-1} , by making a KBr pellet with plantain fibre

2.3.4 Thermal analysis (TGA)

Thermal analysis (TGA) of the fibre was determined using NETZSCH thermo gravimetric balance, (model TG – 209).

2.3.5 SEM-EDS analysis

Fibre morphology was examined by Scanning Electron Microscope (SEM) SEM (Zeiss Gemini SEM) at 15.0 kV.

1. Results and Discussion

3.1. XRD analysis

X-Ray Diffraction (XRD) patterns of the untreated and MV fibres are shown in Figure 1, while crystallographic structural parameters are shown in Table 1. The basic crystalline nature of the cellulose was not changed by Microwave Irradiation but affects the crystallinity, crystallite size and density of the fibre. The maximum intensity of the crystalline peak at 22.50 was obtained by 550W4, followed by 550W2 and is least for 750W4. Moderate microwave power of 550W may have been able to rearrange of the fiber structure, effectively releasing the residual stress [25] and causing a decrease in crystal distortion and defects as evidenced by the increase in degree of crystallinity, crystallite size and bulk density of the fiber as shown in Table 1. However at higher power (750W) and higher irradiation period, degradation of cellulose became prominent as the fiber becomes brittle, thus crystallinity, crystallite size and density of the fibres seemed to decrease, with the least for 750W4, which may be due to the rupturing of cellulose particles into new dimensions [1].

3.2. FTIR Analysis

Figure 2, exhibits the FTIR spectra of the untreated and MV fibres. It was observed that the FTIR curve of the MV fibres are almost similar to each other except for 750W4, suggesting that only physical changes took place in the fibres.

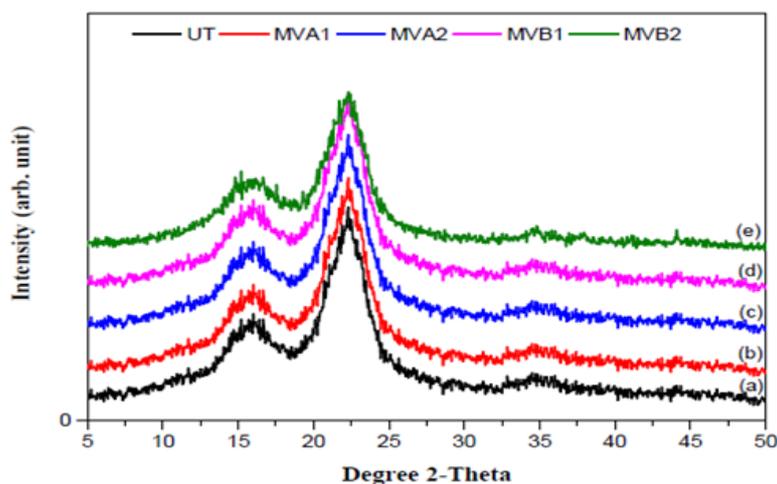


Figure 1: XRD Spectra for Untreated and MV Fibers (a) UT (b) 550W2 (c) 550W4 (d) 750W2 (e) 750W4

Table 1: Various crystallographic and physical parameters of Untreated and MV Fibers

Fiber	Degree of Crystallinity (%)	Crystallite size (Å)	Density (g/cc)
UT	53.07	23.39	1.33
550W2	54.61	29.19	1.44
550W4	56.31	32.10	1.62
750W2	51.27	22.99	1.29
750W4	49.31	20.10	1.21

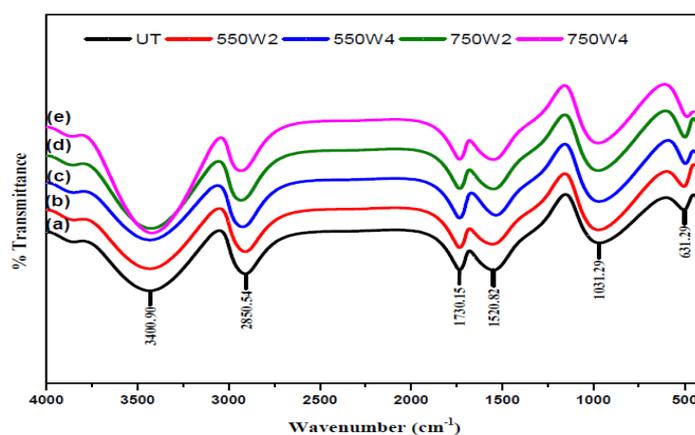


Figure 2: IR Spectra for Untreated and MV Fibers (a) UT (b) 550W2 (c) 550W4 (d) 750W2 (e) 750W4

The broad absorbance peak at 3200– 3400 cm^{-1} corresponds to the O–H stretching of hydrogen bond network [1]. However, at higher treatment power (750W), the intensities of the O–H group tend to increase because of cellulose degradation and incorporation of more polar groups. Hence, at 750W4 the O–H group was highest. All other peaks remain unaffected by the MT. It suggests that no significant chemical changes took place in MV plantain fibres, as only the OH group and absorbed water molecules of the fibre were affected [1]. Thus the hydrophobicity of plantain fibre can be achieved with a proper power setting and treatment period.

3.3. SEM analysis

SEM micrographs of UT, 550W2, 550W4, 750W2 and 750W4 fibres are shown in Figure. 3 (a-e). It is observed that with the increase in irradiation period from 2 min to 4 min keeping the power constant at 550W, the surface roughness of the fibre has increased and uniform surface roughness was achieved. Fibres irradiated at 750W shows a serious damaging effect on the surface of the fibre with the increase in irradiation period from 2 min to 4 min. The severity of damage can be estimated from the exposure of inner central lumen of the fibre shown in Fig 3 (e). The Longer period of high energy radiation at 750W has led to the heavy damage to the fibre surface along with the damage to the strength proving cellulose.

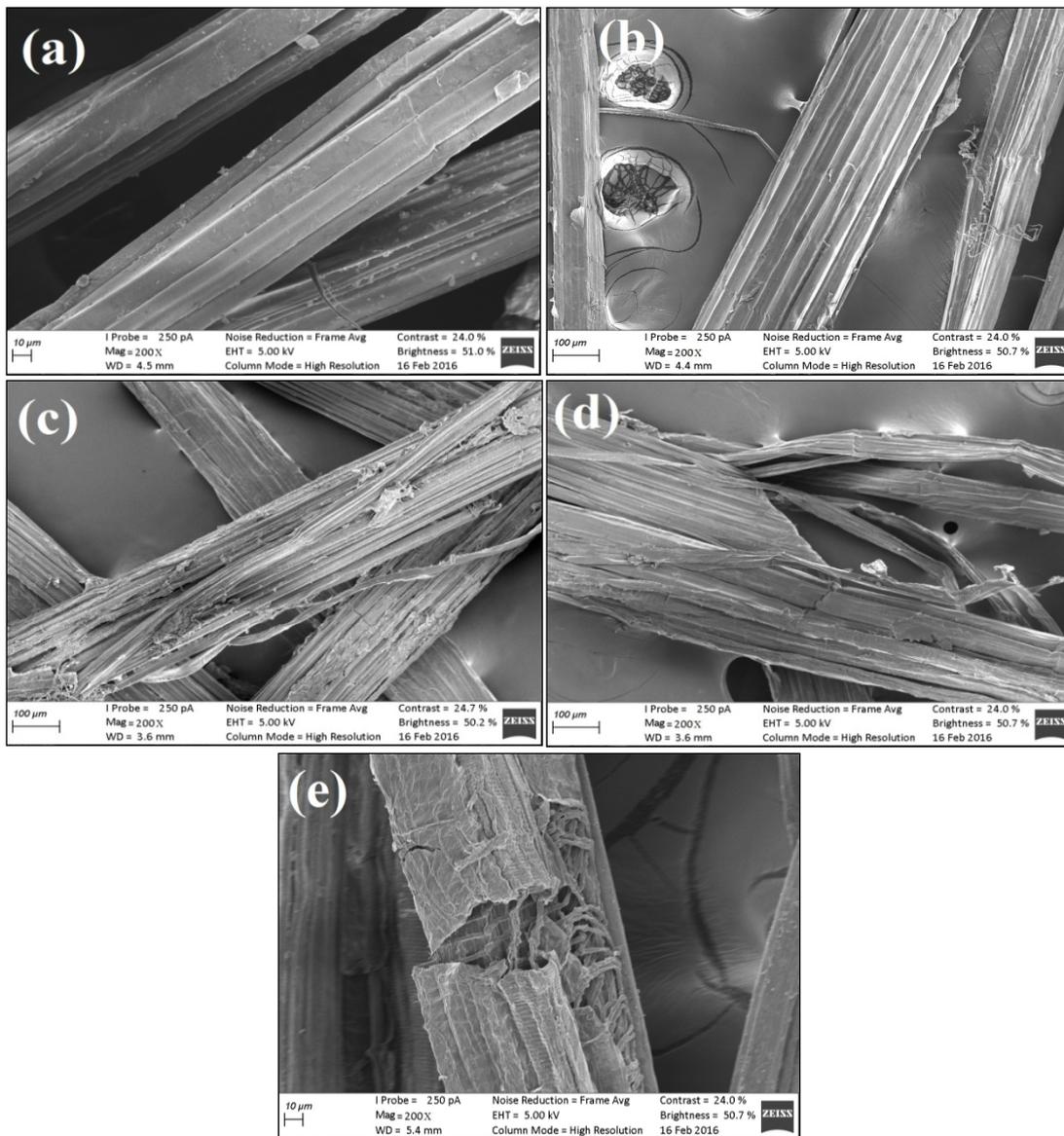


Figure 3: Longitudinal Morphology of Raw and MV fiber (a) UT (b) 550W2 (c) 550W4 (d) 750W2 (e) 750W4

3.4. Thermogravimetry analysis

TGA thermographs of Untreated and Microwave Treated Fibres samples are shown in Figure 4. It can be seen that the curve are almost similar to each other suggesting that the change in the fibre is only physical in nature.

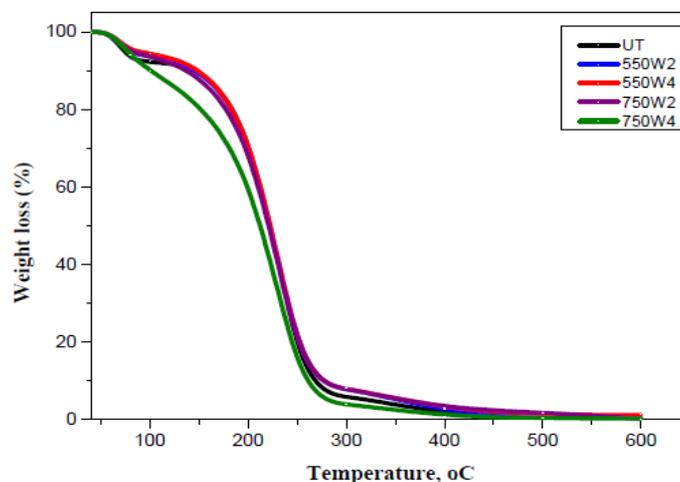


Figure 4: TGA curve for Untreated and Microwave Treated Fibers (a) UT (b) 550W2(c) 550W4 (d) 750W2 (e) 750W4

However, fiber treated at 750W4 shows a decrease in thermal stability this might be due to the degradation of cellulose and incorporation of more polar groups at higher energy radiation thus decreasing the decomposition temperature [26-28].

Conclusions

The applicability and suitability of high energy microwave radiation for the modification of plantain fibre have been investigated. Microwave irradiation has affected the macromolecular parameters of the fibre along with crystallographic structures and morphology of the fibre, an increased degree of crystallinity, crystallite size, and density showing the changes in the physical parameters of the fibre. The chemical structure of the fibre remained unaffected. Adequate surface roughness was achieved at optimised microwave power and time. Finally, it is concluded that the microwave irradiation with 550W power for 4 min treatment period was the best to modify plantain (*Musa paradisiaca*) fibres. This study has shown that microwave modification can significantly modify plantain (*Musa Paradisiaca*) fibres and reduce the inherent hydrophobicity thus making the fibre more permeable to resin when used as composite reinforcement,

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