

# Modification of the Physical and Thermomechanical Characteristics of Mesocarcal Fibers of Palm Kernel Varieties by Alkaline Treatment

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## Abstract

Oil palm mesocarp fiber (OPMF), a biomass waste product from palm oil production, is rich in polysaccharides that can be transformed into a value-added product. This work aims to evaluate the chemical, physical, and thermomechanical properties of these fibers after a multi-step alkaline treatment, with a view to their potential use as reinforcement in industrial materials. The alkaline treatment was carried out in several stages, with sodium hydroxide (NaOH) at different concentrations (3%, 4%, and 5%) as the main reagent, and a solution of hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>) and potassium hydroxide (KOH) to optimize fiber extraction. The results, obtained by Fourier transform infrared spectroscopy (FTIR), revealed significant spectral changes at certain peaks characteristic of the constituent elements of the treated fibers (absorbance at 2859, 1263, and 1027 cm<sup>-1</sup>), thus demonstrating the partial removal of hemicellulose and waxy substances during the process. In addition to the increase in the crystallinity index, initially 27% for untreated OPMF fibers, which reached 56% for OPMF fibers treated with a 5% NaOH solution, derived thermogravimetric analysis (DTG) revealed an improvement in the thermal stability of the fibers treated at 3% and 4%. Scanning electron microscopy (SEM) highlighted a more friable, stable, and porous surface after pretreatment, indicating the presence of perforations in the cell walls and an alteration of the lignin

structure. In this study, the alkaline treatment was limited to four steps to preserve the lignocellulosic nature of the fiber while improving its physical and thermomechanical characteristics. These results demonstrate that alkaline treatment optimizes the performance of OPMF fibers for applications in composite materials.

## Keywords

Palm Kernel Mesocarp, Fiber, Thermomechanical, Alkaline Treatment

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## 1. Introduction

Oil palm mesocarp fiber (OPMF), also known as pressed palm fiber, is the biomass residue obtained after pressing palm fruit for palm oil extraction. OPMF is a lignocellulosic biomass with a high lignin (20.5% to 30.0%) and cellulose (12% to 42.8%) content [1]. During extraction, pressing the fruit generates several co-products used in numerous applications. The raw fiber from industrial processing can be used for various applications, with or without chemical and physical pretreatments [2] [3]. OPMF is a lignocellulosic material that can be obtained from industrial residues and is a promising candidate for various applications, including polymer reinforcement [2] [4]. However, the presence of oily substances and surface impurities still limits its commercial exploitation. Oil and impurities must be removed from FMPH before use. Like other natural fibers, FMPH has several drawbacks, such as its hydrophilicity, susceptibility to biological attack, and low thermal stability. This incompatibility leads to biocomposites that tend to form aggregates during extrusion, are difficult to homogenize, exhibit low adhesion and limited stress transfer, and show fiber pull-out at fracture surfaces [5]. Various plant fiber extraction methods are used for industrial applications [6], each with its own specific characteristics and impact on the final fiber properties. These include mechanical, chemical, and enzymatic processes, which can be used independently or in combination [7] [8]. Mechanical extraction (dehulling, combing, and brushing) is used for the production of textile fibers, enzymatic extraction for the production of nanocellulose and specialty papers; chemical extraction (chemical retting, alkaline treatment, hydrolysis) is used for surface modification of fibers intended for biocomposites to improve their compatibility with the polymer matrix. Two extraction methods can also be combined; the physicochemical method, which combines mechanical retting with chemical retting using NaOH, allows for complete extraction and fibers with optimized mechanical properties. Chemical extractions (organosolv) [1] (ethanol, methanol), bisulfite process (sulfuric acid), sodium hydroxide process, alcohol-insoluble residues (AIR) [9], Kraft process (NaOH) [10], chlorine process [11], nitric acid process, ionic liquid processes, etc.), sometimes applied after pretreatment [12], have proven more effective for the delignification of lignocellulosic materials. Other fiber delignification meth-

ods exist. Megashah *et al.*, in a comparative study between alkaline treatment and [13] peracetic acid bleaching (PAA), observed that the latter further delignified the mesocarp fibers (from 37% lignin after alkaline treatment to 0% after PA acid treatment). However, this process disrupted the lignocellulose structure, leaving only small microfibrils composed mainly of cellulose.

Fiber extraction is generally followed by a second treatment, as various chromophores present in the fibers must be bleached or removed using bleaching agents. Since the chemical nature of chromophores varies, different types of reactivity are required. Therefore, bleaching treatments, such as  $\text{H}_2\text{O}_2/\text{NaOH}$  treatments, are applied to increase the whiteness of the isolated cellulose fibers. Peroxides are used to decolorize and improve the whiteness of the pulp, but depending on the reaction conditions, they can also degrade the cellulose fiber. The scientific literature contains several studies describing the degradation mechanism and the reactions involved [14] [15].

The sequence of alkaline treatment using  $\text{NaOH}$  and bleaching using  $\text{NaOH}/\text{H}_2\text{O}_2$ , in Under appropriate conditions, the majority of amorphous constituents, such as hemicellulose, pectin, lignin, ash, waxes, and oils, can be removed from plant fibers, thus increasing the purity of the cellulose fibers. Sulfuric acid ( $\text{H}_2\text{SO}_4$ ) is frequently used because this reagent breaks down the polysaccharide chains of the fiber, thereby improving the solubility of the resulting cellulose [16] [17]. Organosolv treatment is an interesting process for producing high-purity, low-molecular-weight lignin and recovering a cellulose-rich solid residue [18]. This method uses an organic solvent (e.g., acetic acid, acetone, methanol, ethylene, glycerol, butanol, or ethanol), but at very high concentrations, which implies a considerable amount of reagents.

Alkaline treatment is a common method for modifying natural fibers due to its low cost and ease of implementation. It improves the wettability and surface roughness of the fibers and reduces their moisture absorption. These effects enhance the material's strength and toughness [19] [20]. The effects of alkaline treatment depend on the  $\text{NaOH}$  concentration and soaking time. Beyond optimal conditions, fiber properties deteriorate when  $\text{NaOH}$  weakens or damages them through excessive delignification [21]. Some studies have divided the treatment into several stages by introducing hydrogen peroxide ( $\text{H}_2\text{O}_2$ ) and observed an increase in the crystallinity index from 33% to 50% and then 65% [11], but this treatment proved less effective with the organosolv process [22], in which the crystallinity of the treated fibers is higher. Potassium hydroxide ( $\text{KOH}$ ) is also used in the alkaline pretreatment of various raw materials [23] to remove residual hemicellulose [24].

This study aims to efficiently extract non-cellulosic amorphous materials from oil palm mesocarp fibers (OPMF) using multi-step pretreatments. Bleaching was performed for three different durations: 1, 2 and 3 hours with a  $\text{NaOH}/\text{H}_2\text{O}_2$  solution, followed by 1 hour with a  $\text{KOH}$  solution. The characteristics of the resulting fibers will be investigated using several analytical techniques, such as X-ray diffraction (XRD), scanning electron microscopy (SEM), Fourier transform infra-

red spectroscopy (FTIR), and thermogravimetric analysis (TGA), to determine the crystallinity, morphology, and thermal stability of the cellulose.

## 2. Materials and Methods

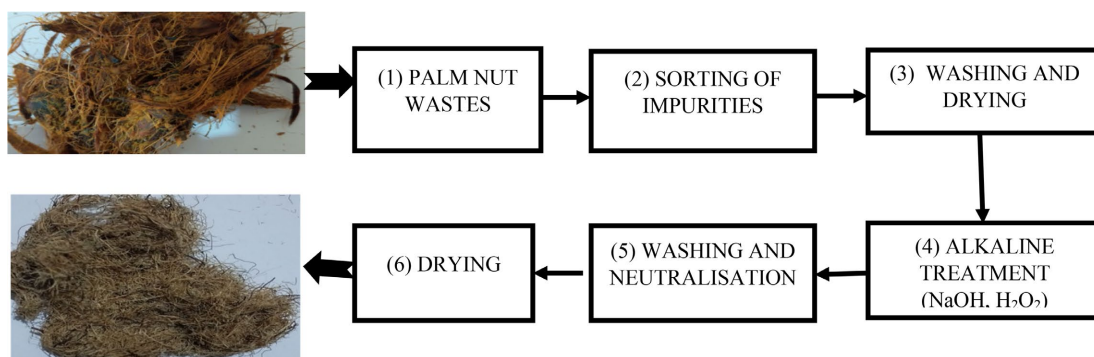
### 2.1. Materials

Samples of palm oil from palm oil production (POPP) were collected from the “ETS Père et Fils” palm oil mill in the town of Eseka, located in the Central Region of Cameroon. After thorough washing under running water, the samples were sun-dried until all moisture was removed to prevent fungal growth. Once dry, they were stored in airtight plastic bags at room temperature before further processing.

### 2.2. Working Methodology

The adopted working methodology is based on considering two important parameters during alkaline treatment: the NaOH concentration and the solution temperature. Beyond the optimal conditions, the fiber properties deteriorate when the NaOH weakens or damages the fiber due to excessive delignification [21]. However, Aris’s [25] work shows that even at high concentrations (3%, 5%, and 10%), one hour of soaking did not have a significant effect on removing residue from OPMF fibers. He did, however, observe a 40% decrease in melting temperature at a 10% NaOH concentration. Some studies have shown that oil palm residue fibers should be treated with 3% NaOH when intended for composite manufacturing [22] [26]. At a 5% NaOH concentration, improvements in the composite’s mechanical properties were observed [27]. These observations led us to adopt a very specific working methodology.

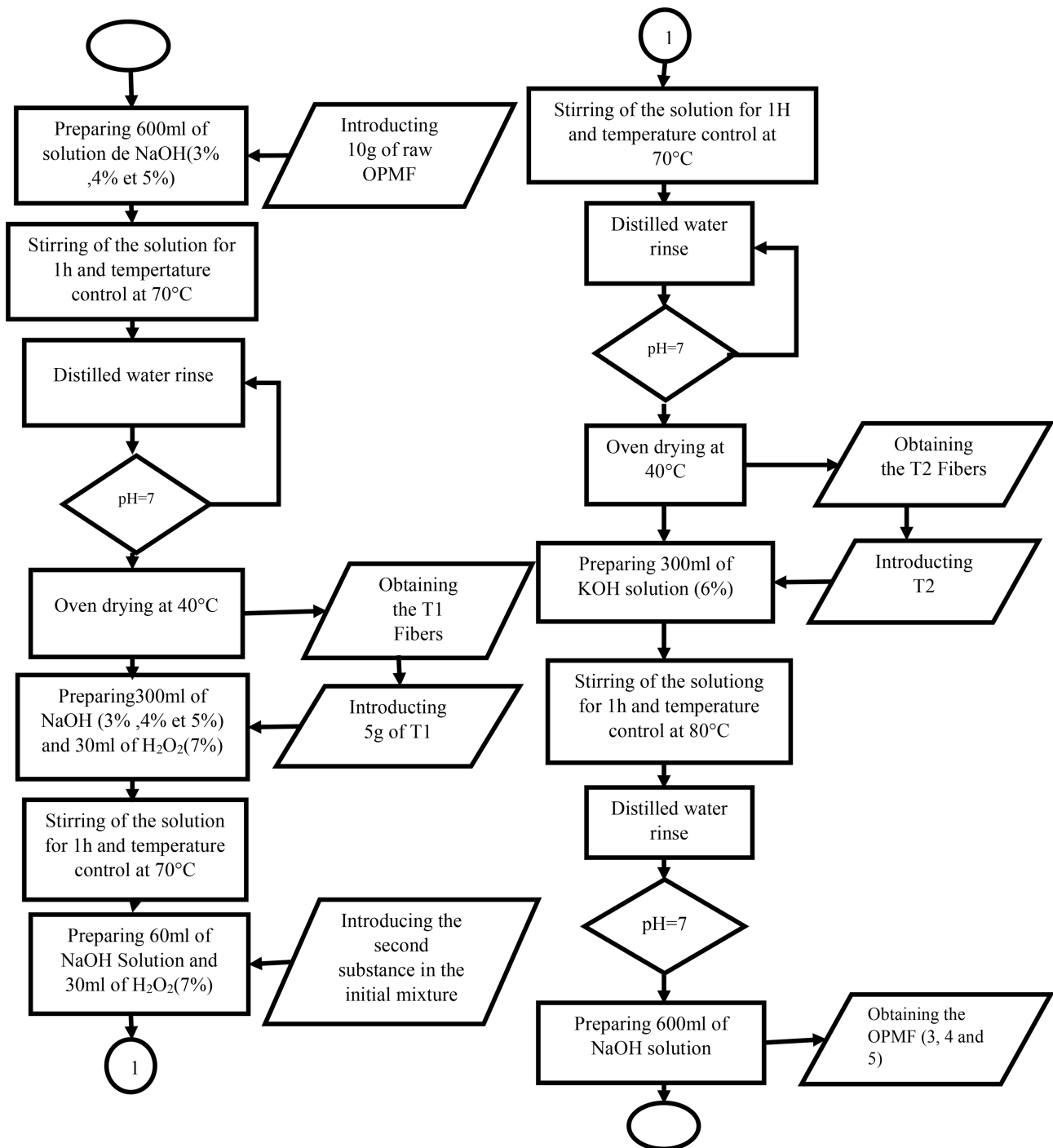
The fibers studied came from the Eseka family oil mill. The mill’s waste was first sorted manually, an operation that separated the palm fibers from the kernels still in their shells (1). They were then soaked in distilled water for 24 hours to remove the pulp and any infiltrated sand, then rinsed and oven-dried at 50 °C for 24 hours (3). The alkaline treatment was carried out according to the protocol described by Pereira *et al.* [11] applied to the pineapple crown shown in **Figure 1**. At the end of the treatment, the fibers were rinsed until a neutral pH was reached (4), then oven-dried.



**Figure 1.** Functional diagram of the fiber extraction process.

### 2.3. Extraction Process

The extraction process is described in **Figure 2** and was carried out in three stages.



**Figure 2.** OPMF extraction process.

### 2.4. Characterization

#### 2.4.1. Fourier Transform Infrared (FTIR) Spectroscopy

FTIR analyses were performed on OPMF 0, OPMF 3, OPMF 4, and OPMF 5 palm

fibers to evaluate the functional groups of the samples. FTIR spectra were obtained using a Perkin Elmer spectrophotometer (Spectrum 100 model) operating in attenuated total reflection (ATR) and transmission modes. Measurements were taken between 450 and 4000  $\text{cm}^{-1}$  with 12 accumulations.

#### 2.4.2. Scanning Electron Microscopy (SEM)

The sample surfaces were coated with a thin layer of gold by sputtering for 120 seconds to ensure their electrical conductivity before scanning electron microscopy (SEM). The longitudinal surface of the fibers was observed using a Carl Zeiss AG-supra 25 scanning electron microscope equipped with an EBSD detector. Several images were acquired under an accelerating voltage of 15 to 20 kV and at magnifications of 500 to 1500 $\times$ .

#### 2.4.3. X-Ray Diffraction (XRD) Analysis

X-ray diffraction (XRD) analysis was used to quantify the degree of crystallinity of OPMF 0, OPMF 3, OPMF 4, and OPMF 5 palm fibers after each treatment. Untreated and treated fibers exhibited similar diffractograms, with two  $2\theta$  diffraction peaks of  $16.5^\circ$  (101) and  $22.1^\circ$  (002), corresponding respectively to the amorphous and crystalline phases of the fiber [28], and characteristic of cellulose [29]. The  $16.5^\circ$  peak was broader than the  $22.1^\circ$  peak, due to its amorphous nature. The crystallinity index was calculated using the Segal method [28], which can be formulated as follows [30]:

$$I_{crl} = \frac{I_{002} - I_{am}}{I_{002}} \times 100 \quad (1)$$

where  $I_{002}$  is the maximum intensity of the diffraction peak of the (002) plane at  $2\theta \approx 22^\circ$ , and  $I_{am}$  is the minimum intensity between the main peaks.

#### 2.4.4. Thermogravimetric Analysis (TGA)

Thermogravimetric analysis (TGA) is an essential tool for determining the thermal stability of materials. In our study, TGA was performed using a thermal analyzer (TG/DTG SII Nanotechnology INC, model 6200) at a heating rate of  $10^\circ\text{C}/\text{min}$  between  $25^\circ\text{C}$  and  $600^\circ\text{C}$ , under a nitrogen flow rate of 60 mL/min, in a nitrogen atmosphere, and with approximately 10 mg of dry samples. For kinetic calculations, two additional heating rates,  $5^\circ\text{C}/\text{min}$  and  $10^\circ\text{C}/\text{min}$ , were included.

#### 2.4.5. Water Absorption

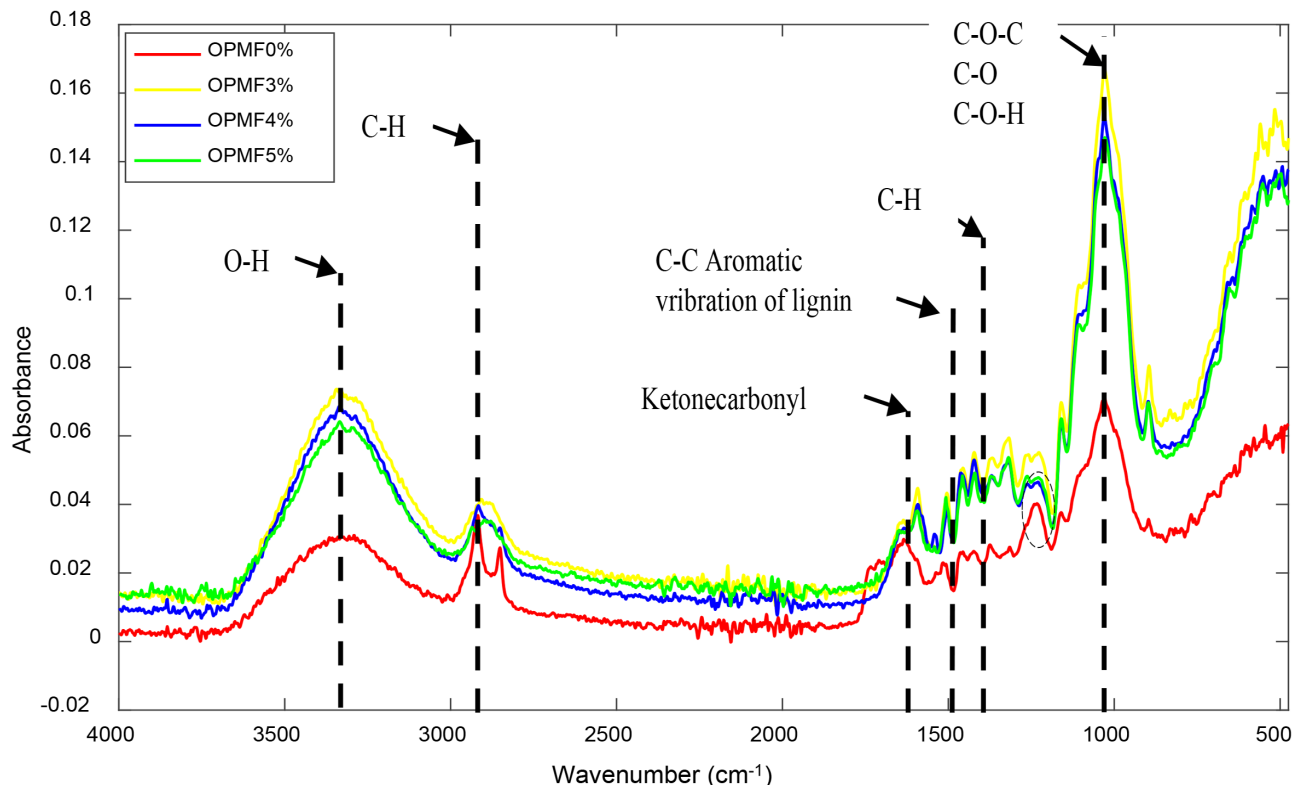
The fibers were oven-dried at  $60^\circ\text{C}$  until a constant mass was obtained before testing. The mass of the dry fibers ( $W_{0h}$ ) was measured using a microbalance with an accuracy of  $\pm 0.001$  g. The fibers were then immersed in distilled water for 24 hours at  $25^\circ\text{C}$ . After 24 hours of immersion, the fibers were removed from the distilled water and weighed immediately to obtain their wet mass ( $W_{24h}$ ). The test was performed in duplicate, and the average values were recorded. The moisture absorption of the fibers was calculated using the following formula:

$$\text{water absorption (\%)} = \frac{W_{24h} - W_{0h}}{W_{0h}} \times 100 \quad (2)$$

### 3. Results and Discussion

#### 3.1. Fourier Transform Infrared (FTIR) Spectroscopy

The FTIR spectra of various untreated (OPMF 0%) and treated (OPMF 3%, OPMF 4%, and OPMF 5%) fibers are shown in **Figure 3**. The OPMF spectrum exhibits characteristic peaks at 3300  $\text{cm}^{-1}$  (OH stretching vibration of cellulose, hemicellulose, and lignin), 2859  $\text{cm}^{-1}$  and 1434  $\text{cm}^{-1}$  (CH stretching vibration of cellulose and hemicellulose) [31], 1730  $\text{cm}^{-1}$  (C=O stretching vibration of the carbonyl groups of hemicellulose and/or lignin) [32], 1680  $\text{cm}^{-1}$  and 1514  $\text{cm}^{-1}$  (C=C stretching vibration of the aromatic ring of lignin) [33], and 1027  $\text{cm}^{-1}$  (vibration C=O of the ester, ether, and phenol groups of waxy substances or C=O stretching vibration of the acetyl groups of hemicellulose). The spectra of 3% OPMF, 4% OPMF and 5% OPMF show relatively low absorbance at the peaks located at 2859 and 1263  $\text{cm}^{-1}$ , demonstrating the partial removal of hemicellulose and waxy substances during processing. The intense peak at 1680  $\text{cm}^{-1}$  observed in the 0% OPMF spectrum disappears in the 3%, 4%, and 5% OPMF spectra. This is attributed to the removal of extractable compounds after processing [34]. Furthermore, the absorption peak at 1027  $\text{cm}^{-1}$  indicates a stretching vibration within the pyranose ring of COC, suggesting an increase in cellulose crystallinity [35].



**Figure 3.** FTIR spectrometry of OPMF 0, OPMF 3, OPMF 4 and OPMF 5.

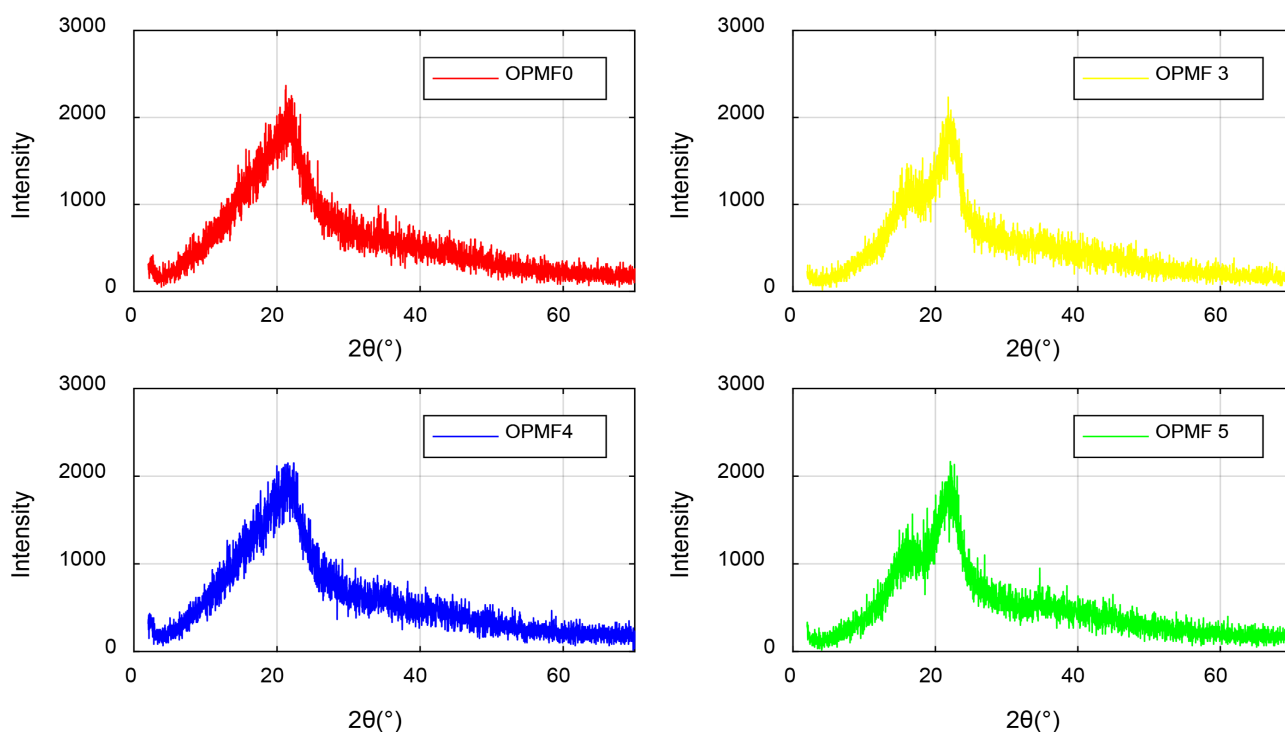
### 3.2. X-Ray Diffraction (XRD) Analysis

The crystallinity index was calculated and reported in **Table 1** from the principal peak, called the crystallographic plane, at  $2\theta = 16.5^\circ$  of reflection,  $I_{am}$  of the amorphous phase and at  $2\theta = 22.1^\circ$  of the reflection  $I_{002}$ , which corresponds to the crystalline plane.

Segal crystallinity indices are 50 and 57 for OPMF 3 and OPMF 5, respectively, increasing from 28% (before treatment) to 58%. As shown in the diffraction pattern (**Figure 4**), the highest intensity was found in the treated fibers, which ultimately affected the cellulose crystallinity. The increase in the crystallinity index after the pretreatments can be attributed to more efficient removal of non-cellulosic and amorphous materials, increasing the alpha-cellulose content and enhancing the regularity of the crystal lattice [36]. OPMF cellulose, like all plant fibers, has a high crystallinity index, implying a higher percentage of crystalline fraction relative to the amorphous fraction of the cellulosic structure [37]. This confirms the observation made in Section 2.1, which predicted an increase in crystallinity due to the

**Table 1.** Variation in fiber crystallinity rate.

Fibers	$I_{am}$ (cps)	$I_{002}$	$I_{crl}$ (%)
OPMF 0	1216	1683	28
OPMF 3	950	2300	58
OPMF 4	1250	2084	40
OPMF 5	933	2166	57

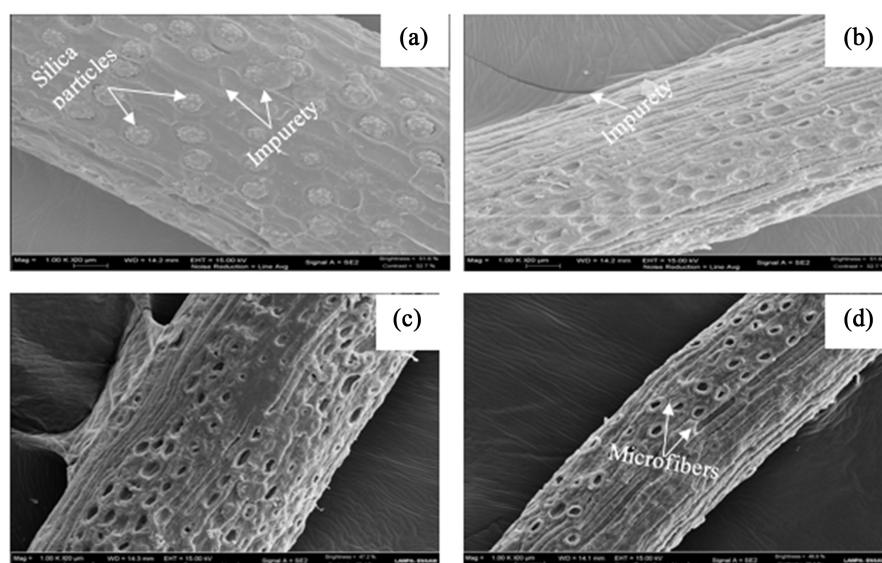


**Figure 4.** X-ray diffraction (XRD) analysis of OPMF fibers.

stretching of the absorption peak to  $1027\text{ cm}^{-1}$ . These results are consistent with those of Chieng *et al.* [38] and Souza *et al.* [1], who obtained crystallinity levels of up to 67% using alkaline treatment of palm kernel mesocarp. We note that this index is lower than that obtained by Pereira *et al.* [11] (24% to 80%) due to the fact that we performed this treatment by removing one phase in order to best preserve the lignocellulosite structure of our fiber [13].

### 3.3. Scanning Electron Microscopy (SEM)

Scanning electron microscopy (SEM) analysis was used to examine the surface morphology of the palm fiber samples. In **Figure 5(a)**, several silica aggregates are visible on the fiber surface [2] [39]. The physical structure of the raw palm fibers (OPMF) is clearly visible: the silica and aggregates are tightly bound to the pores, as are the impurities. Conversely, **Figures 5(a)-(c)** reveal perforations in the cell wall, indicating lignin degradation. The treatment facilitated lignin degradation by increasing mass transfer and inducing cavitation, where bubbles rapidly formed, expanded, and burst, generating localized hot spots that disrupted the biomass structure [35]. Huysken Mejouyo *et al.* [40] observed that the more porous the surface, the greater the hydrophilic character of the OPMF. The presence of some impurities was still noted on the surface of OPMF 3, while that of OPMF 5 was cleaner. The surfaces of the treated fibers appeared relatively clean and rough compared to those of untreated fibers, which is attributed to the partial removal of impurities and non-cellulosic substances. This observation is similar to other studies [41] [42]. In addition, the treatment eliminated silica deposits, as shown in **Figures 5(b)-(d)**. The removal of these impurities makes the fiber surface rough and promotes good contact with other surfaces; this can be an advantage for this fiber when combined with other materials for the production of composites because of the micro-adhesions that can be established between the fiber and

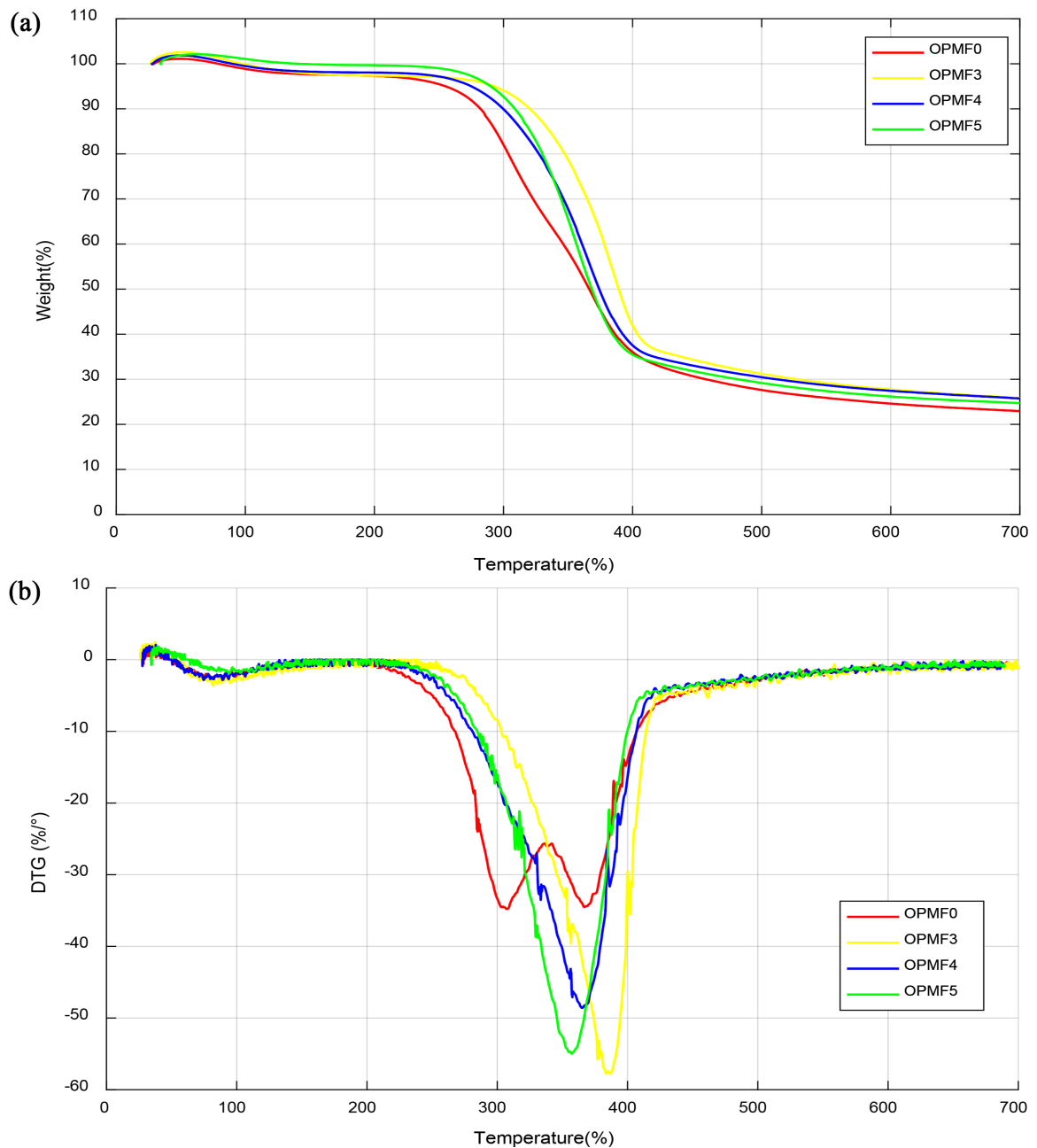


**Figure 5.** Morphology of OPMFs: (a) OPMF 0; (b) OPMF 3; (c) OPMF 4; (d) OPMF 5.

a matrix thanks to the asperities present in the fiber, creating junctions and a mixture, preventing any slippage between the two materials in case of longitudinal force on this composite.

### 3.4. Thermogravimetric Analyzer (TGA) and Derived Thermogravimetric Analyzer (DTG)

The thermogravimetric (TG) and derived thermogravimetric (DTG) curves of untreated OPMF 0% and OPMF 3% samples, as well as treated OPMF 4% and OPMF 5% samples, are shown graphically in **Figure 6(a)** and **Figure 6(b)**. The



**Figure 6.** TGA (a) and DTG (b) curves.

TG curves (**Figure 6(a)**) show several stages of degradation due to the sequential degradation of the different fiber components. This is clearly visible in the DTG curves (**Figure 6(b)**), where three distinct peaks are observed within the studied temperature range. The first peak, as shown in the DTG curves, is attributed to the evaporation of moisture absorbed at the fiber surface. The second peak corresponds to the degradation of hemicellulose, and the third to the degradation of cellulose [42].

Thermogravimetric analysis (TGA) was performed to determine the thermal degradation characteristics of the fibers. The TGA curves (**Figure 6(a)**) and differential thermogravimetric analysis (DTA, **Figure 6(b)**) illustrate the mass loss under different fiber treatment conditions (**Table 2**). The treated fibers exhibit relatively low evaporation rates of moisture or volatile compounds (0.18%) compared to untreated fibers, confirming the conclusion of Rosa *et al.* [43], who obtained a relative evaporation rate of 2.41%. Regarding the mesocarp, the largest difference is observed between 250 °C and 350 °C, with maximum values of 50.91% for OPMF 0, 65.26% for OPMF 3, 61.94% for OPMF 4, and 62.33% for OPMF 5. As the temperature increases from 160 °C to 315 °C, a slight derivative peak appears, with the maximum mass loss observed around 315 °C. This degradation step is primarily attributed to the distillation of hemicellulose to form acetic acid [44]. Thus, samples OPMF 3 (6.53%), OPMF 4 (13.48%), and OPMF 5 (12.78%) exhibit relatively low mass losses compared to OPMF (24.17%), reflecting a higher hemicellulose content in the untreated fibers. Derivative peaks are observed at an average temperature of 360 °C, corresponding to the maximum mass loss of cellulose. At this stage, the mass loss of the treated fibers (from 61.94% to 65.26%) is greater than that of OPMF (50.91%). This is explained by the increased percentage of cellulose in the treated fibers.

**Table 3** presents the thermal degradation temperatures of the fibers for mass losses of 5%, 10%, and 50%. The treated fibers exhibit a higher degradation temperature than the OPMF fibers. For example, the degradation temperature for a 10% mass loss ( $T_{10\%}$ ) is 272.03 °C for OPMF 3, SOPMF 4, and SNOPMF 5 fibers, respectively. These results are close to those obtained by Then *et al.* [42] (247.7 °C to 293.26 °C). And the degradation temperature at 50% weight loss ( $T_{50\%}$ ) of OPMF was 303.03 °C and 389.2 °C, 374.09 °C and 368.24 °C for OPMF 3, SOPMF 4 and SNOPMF 5 respectively, values slightly higher than those obtained by

**Table 2.** Fiber weight loss.

Fibers	Weight loss (%)			Residues at 500 °C (%)
	35 °C - 160 °C	160 °C - 315 °C	315 °C - 500 °C	
OPMF 0	2.41	24.17	50.91	22.51
OPMF 3	2.33	6.53	65.26	25.88
OPMF 4	1.83	13.48	61.94	22.75
OPMF 5	0.18	12.78	62.33	24.71

Pereira *et al.* [11], the evolution therefore goes from 326 °C to 351 °C.

**Table 4** presents a summary of **Figure 4** and **Figure 6**, which respectively show the crystallinity and thermal stability as a function of the percentage of sodium hydroxide used during the alkaline treatment of mesocarp fibers. It can be observed that the fibers treated with 3% exhibit a higher crystallinity and thermal stability than the other treated fibers, and even better than the untreated fibers; specifically, an increase of over 100% for the crystallinity and 28.44% for thermal stability. We also observe that the treated fibers have an average crystallinity of  $52\% \pm 10.12\%$  and a thermal stability of  $377^\circ\text{C} \pm 10.82^\circ\text{C}$ .

**Table 3.** Thermal degradation temperature of fibers.

Fibers	Temperature (°C)		
	T 5%	T 10%	T 50%
OPMF 0	255.35	282.03	303.03
OPMF 3	292.76	320.2	389.2
OPMF 4	274.88	290.54	374.09
OPMF 5	290.14	308.84	368.24

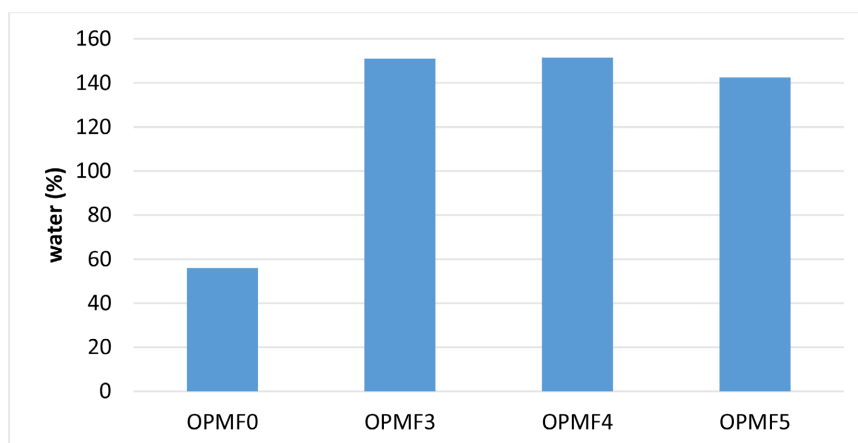
**Table 4.** Summary table of thermomechanical parameters of the mesocarp fiber.

Fibers	$I_{crit}$ (%)	$T_{onset}$ (°C)
OPMF 0	28	303.03
OPMF 3	58	389.2
OPMF 4	40	374.09
OPMF 5	57	368.24

### 3.5. Absorption of Water

Lignin is more hydrophobic than hemicellulose or cellulose, and hemicellulose is the most hydrophilic of the three fiber components and is primarily responsible for moisture absorption. Therefore, a decrease in lignin content makes the fiber more susceptible to moisture absorption. **Figure 7** illustrates the water absorption rate of different OPMF compositions with varying NaOH concentrations, showing an increase in absorption for concentrations below 3%. Above this concentration, water absorption decreases slightly, reaching a value lower than that of the initial untreated sample.

This was clearly demonstrated by the relatively high moisture content of the treated fibers compared to that of the raw OPMF. Several studies have shown that the percentage of water absorption increases with the fiber volume fraction, due to their high cellulose content [45]. Cellulose is intrinsically hydrophilic due to the presence of polar functional groups [46] (surface hydroxyl groups); this shows that the treatment effectively reduced the non-cellulosic components. However, this parameter is not advantageous for the development of a composite material



**Figure 7.** Water absorption of OPMF, OPMF 3, OPMF 4 and OPMF 5.

[47]; it will be necessary to apply a physical or chemical treatment [48] to these fibers to make them more hydrophobic.

### 3.6. Discussion

We have observed in the literature that multi-stage alkaline treatment provides strong insulation for cellulose. Since cellulose is present in the form of nanocrystals, its high percentage leads to defibrillation of the material [11] [13]. It is true, as we also noted in section 2.2, that these bleached fibers exhibited a slightly higher degree of crystallinity. In this article, we limited ourselves to three treatment phases, which allowed us to preserve the fibrous nature of our material and obtain fibers with thermal stability (Section 3.4) comparable to that of fibers described in the literature.

Furthermore, recent studies show that treatment with hydrogen peroxide (3%  $\text{H}_2\text{O}_2$ , heat) significantly improves saccharification (enzymatic hydrolysis), meaning that cellulose becomes more accessible [49] [50]. The mechanisms described show that alkaline  $\text{H}_2\text{O}_2$  generates radicals ( $\text{HOO}^-$ ,  $\text{HO}\cdot$ ,  $\text{O}_2^- \cdot$ ) capable of oxidizing lignin. The authors note that, despite this, it is not certain that the cellulose is extensively depolymerized: according to them, the main effect seems to target the lignin; this does not eliminate the risk of cellulose degradation, which could lead to alterations in the fiber's mechanical properties. The authors emphasize that optimized conditions are necessary to avoid significant polysaccharide losses [51]. Although the stepwise process limits this degradation, the possibility of some cellulose degradation must be considered. Future work should include direct measurements of tensile strength and molecular weight to fully quantify this trade-off between purification and structural integrity.

### 4. Conclusion

This work focused on improving the thermomechanical properties of palm kernel fibers for use as reinforcement in composite materials. Our approach consisted of subjecting these fibers (OPMF) to a multi-stage alkaline treatment. Our method-

ology relied on a series of physical (SEM, absorption rate), chemical and structural (FTIR), and thermomechanical (TGA, XRD) characterizations. Microscopic observation revealed a cleaner surface on the treated fibers, due to the removal of silica particles. However, traces of silica were detected on the OPMF 3 sample, which explains the significant amount of residue observed during thermogravimetric analysis. Furthermore, this porous surface promotes water absorption by the fibers, but we observed that the fibers treated with 5% sodium hydroxide (OPMF 5) absorbed less water. The multi-step pretreatment increased the crystallinity of the treated OPMF from 28% to 58%, a rate similar to that observed for OPMF 3, whose highest value proved to be the most stable (375°C), consistent with previous results. These results are corroborated by FTIR spectroscopy, which reveals the disappearance of certain specific lignin and hemicellulose peaks in the spectrum of the treated fibers. Conversely, an increase in the characteristic lignin peak to 1514 cm<sup>-1</sup> is observed in these same fibers. We were thus able to extract the mesocarp fiber while preserving its lignocellulosic structure and improving its thermomechanical properties, paving the way for its use in industrial applications. Our future research will focus on modifying cellulose by adding an additional treatment to make it less hydrophilic, and after which we will look into understanding the adhesion mechanisms between these treated fibers and different polymer matrices.

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### **Authors' Contributions**

Claire Ariane Amagnameda, Dieudonné Essola, and Fabien Ebanda Betene conceived the idea presented. Dieudonné Essola, Claire Ariane Amagnameda, and Claire Ba'Ana Melti wrote the various experimental protocols and oversaw the experimental planning in accordance with previously published studies. Claire Ariane Amagnameda established the extraction protocol and participated in sample preparation. She also contributed to writing the methodology, the formal analysis of the experimental results, and the manuscript. Pierre Marcel Noah contributed to the methodological approach, formal analysis, validation of results, and proofreading. Maurice Kontchou Vanlie studied the thermogravimetric analysis and the water absorption process. Marina Fedotova supervised part of the experimental setup, including the complete scanning electron microscopy procedure, and discussed some interpretations. Fabien Ebanda Betene supervised this work and was assisted by Dieudonné Essola, who played a key role in collecting exper-

imental data. Both participated in reviewing and validating the results. All authors accepted responsibility for the entire content of this manuscript and approved its submission.

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## Data Availability Statement

The datasets generated and/or analyzed during this study are available from the corresponding author upon reasonable request.

## Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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## Nomenclature

AIR	Residues insoluble in alcohol
DTG	Thermogravimetric derivative
FTIR	Fourier transform infrared spectroscopy
H <sub>2</sub> O <sub>2</sub>	Peroxide hydrogen
KOH	Potassium hydroxide
NaOH	Sodium hydroxide
OPMF	Oil palm mesocarp fiber
PAA	Acid acetic acid
MEB	Microscopy scanning electronics
TGA	Analysis thermogravimetric
DRX	X-ray diffraction
$I_{crl}$	Crystallinity index