

Characterization of the chemical composition of extracted oil palm frond (OPF) fibres using TAPPI standard methods and Wise's method

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ABSTRACT

Malaysia is the second largest exporter of oil palm, with over 5 million hectares of plantations that produce a large amount of waste materials like palm oil mill waste, empty fruit bunches, fronds, trunks, shells and fibres. Extracting cellulose fibres (CFs) from oil palm fronds (OPF) waste is a smart strategy in managing oil palm plantation waste. Therefore, this study was aimed to extract OPF fibres and determine the actual percentage weight content of the cellulose, hemicellulose and lignin from the extracted OPF fibres. The first part of the study is to extract OPF fibres from Malaysia OPF using direct chemical method. A mixture of 3.2 mol/L nitric acid and 60 mmol/g hydrogen peroxide was used to break down the complex structure of OPF at temperature of 50 °C for 48 h. Meanwhile, in the second part of the study, the percentage weight of the lignin, hemicellulose and cellulose in the extracted OPF were determined using the TAPPI 222, Wise's Method and TAPPI 203 respectively. Additionally, Fourier Transform Infrared Spectroscopy (FTIR) analysis was conducted to characterize and identify the functional groups present in the extracted OPF fibres. The yield of successful extracted OPF fibres using direct chemical treatment is $51.3 \pm 0.7\%$. Meanwhile, the analysis result shows that the extracted OPF fibres contained 7 ± 0.5 wt% of lignin, 32 ± 4.1 wt% of hemicellulose and 37 ± 4.5 wt% of cellulose. The actual content of lignin, hemicellulose and cellulose in the extracted OPF fibres is useful to guide for suitable commercial sustainable applications based on the required strength.

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1. INTRODUCTION

Oil palm or also known as *Elaeis Guineensis Jacq* originated from West Africa. Today, the cultivation of the oil palm has been spread throughout the tropical regions and become one of the important crops, especially in the Southeast Asia (Ooi et al., 2017). Originally, it was grown as the decorative plant but over time, it has become one of the leading sources of the vegetable oil worldwide. According to the Statista (2024), oil palm has produces about 77.99 million metric tons of edible oil or cooking oil for the global consumption. In line with this, a study by Ahmad et al. (2019), shows that oil palm is the highest yielding oil crop in the world which producing nearly eight times more oil than rapeseed and six times more than soybean. This high productivity contributes significantly to Malaysia's economy, generating billions of ringgits for the oil palm industry (Ooi et al., 2017). As the second largest producer and exporter of palm oil globally, Malaysia has made oil palm one of its most dominant industrial crops which creating a million of jobs across the country (Harun & Laksito, 2022).

However, the cultivation of oil palm results in a significant amount of the agricultural waste which are also known as the oil palm biomass. Basically, the biomass refers to the by-product produced during activities such as replanting, pruning and milling in the oil palm industry. In many cases, this biomass just left to decompose in the fields (Onoja et al., 2019). Recent studies by Yow et al. (2024) report that Malaysia produces about 168 million tonnes of biomass which including palm oil waste, sugarcane waste and rice husk waste and others. Basically, the oil palm extraction is only around 10% of the palm oil production while the remaining 90% is left as the biomass (Jafri et al., 2021). OPF is the most abundant type of biomass generated in the palm oil industry which accounts for over 50% of the total biomass produced (Ooi et al., 2017). According to Pulunggono et al. (2019), the largest contribution to biomass availability comes from oil palm frond and trunks compared to other types of the oil palm waste. These fronds which are produced during harvesting and pruning are typically left on the ground to decompose naturally and replenish the nutrients to the soil. However, during replanting, OPF production is estimated to reach around 56 million tonnes annually. This highlights its value as a major byproduct of the oil palm industry (Rusli et al., 2021). This large amount mainly results from the routine pruning activities throughout the tree's lifespan.

In a typical replanting cycle, which occurs every 25 to 30 years which about 75 tonnes per hectare of oil palm trunk (OPT) and 15 tonnes per hectare of OPF are generated. Due to its high availability and potential, OPF is considered a promising raw material in Malaysia (Ahmad et al., 2019). However, the full utilization of the OPF still remains limited due to challenges in accurately extracting and quantifying these component (Ooi et al., 2017). The extraction and measurement of the percentage composition of cellulose, hemicellulose and lignin and lignin from the lignocellulosic biomass such as OPF require a reliable method to obtain an accurate and consistent results for further study or analysis. Generally, direct chemical treatment method (DCM) is the method that is commonly used to break down the complex plant structure and for isolating these components for further analysis.

However, there is limited research on the analysis of actual composition of cellulose, hemicellulose and lignin in the extracted OPF fibres. Moreover, the Technical Association of the Pulp and Paper Industry (TAPPI) standard method is a recognised standard for analysing the lignocellulosic content but has not been widely and extensively applied. Hence, the first focus of this research is to extract CFs from OPF fibres using DCM. Chemical treatment such as acid and alkali methods is widely used to weaken or remove lignin from the OPF fibres (Kumar & Sharma, 2019). Meanwhile, the second focus of this research is to determine

the actual percentage weight content of the cellulose, hemicellulose and lignin in the extracted OPF fibres by using TAPPI standards methods (TAPPI 222; TAPPI 203) and Wise's method (Ali et al., 2023). The outcome analysis will help to establish an efficient extraction method and support suitable commercial sustainable applications that meet the required strength

2. METHODOLOGY

2.1 Direct chemical treatment method (DCM)

Materials

The OPF fibre was supplied by Persatuan Peladang Hulu Langat, Selangor. OPF was chopped into small parts after being cleaned. After that, it was dried in the sun (Azani et al., 2020; Abdul Khalil et al., 2025). Then, the initial and intervals weight of the OPF were recorded until the moisture content stabilized at less than 10%. The dried OPF fibre was ground using a grinding machine (SR 300, Retsch, Germany) and then sieved using a sieve machine to obtain OPF fibres in powder form. The resulting powder has a finer distribution with particle size ranging from 40 to 60 mesh (250–425 μm) as suggested by (Abdul Khalil et al., 2025; Indriani Widiputri et al., 2022). Fig. 1(a) and (b) show the image of raw OPF and grinded OPF respectively. The OPF fibres were then stored in a glass desiccator until the experiment was conducted. In this direct chemical treatment method, nitric acid (HNO_3) (Merck, Germany) with 60% purity and hydrogen peroxide (H_2O_2) (Merck, Germany) with 30% purity were utilized as chemical reagents (Abdul Khalil et al., 2025).

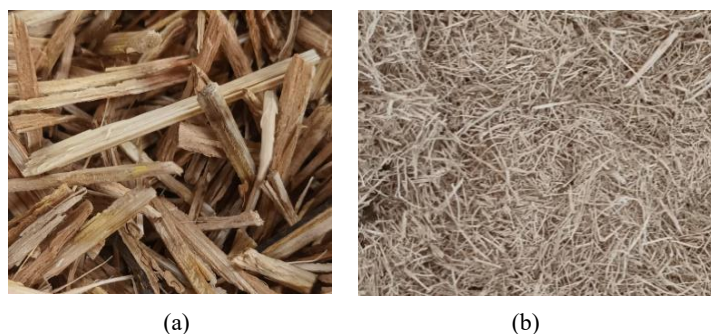


Fig. 1. (a) Raw OPF and (b) ground OPF used in the DCM

Source: Authors' own data

Methods

Five (5) grams of OPF powder was treated in a flask consist of hydrogen peroxide (H_2O_2 , 30% purity) (Merck, Germany) at 60 mmol/g and nitric acid (HNO_3 , 60% purity) (Merck, Germany) at a concentration of 3.2 mol/L as suggested by our previous study (Abdul Khalil et al., 2025). The mixture was agitated with magnetic stirring at 1000 rpm at 50 °C for 48 h and conducted in fume hood (refer Fig. 2(a)). The treatment procedure was halted by introducing deionized water which is five times the solution's volume. Subsequently, the solution was then washed with deionized water under vacuum suction until the filtrate reached neutrality to obtain the OPF cellulose fibre. The apparatus used were shown in Fig. 2(b). Then the

extracted OPF cellulose fibre was allowed to dry at room temperature for one day. The drying process was continued in a vacuum oven (IVO25, Taisite, United States of America) at a temperature lower than 50 °C (Abdul Khalil et al., 2025) until constant weight was recorded (refer Fig. 2(c)). The yield of extracted OPF fibre was calculated using Eq. (1).

$$\text{Yield of extraction} = \frac{\text{Dried OPF weight (g)}}{\text{Initial OPF weight (g)}} \times 100 \quad (1)$$

This extracted OPF fibre was quantitatively analysed to meet the second objective of the study. The actual composition of cellulose, hemicellulose, and lignin in the extracted OPF fibre was determined via TAPPI standard methods and Wise's method. Meanwhile, the chemical composition of the initial raw OPF fibre was not analyzed.

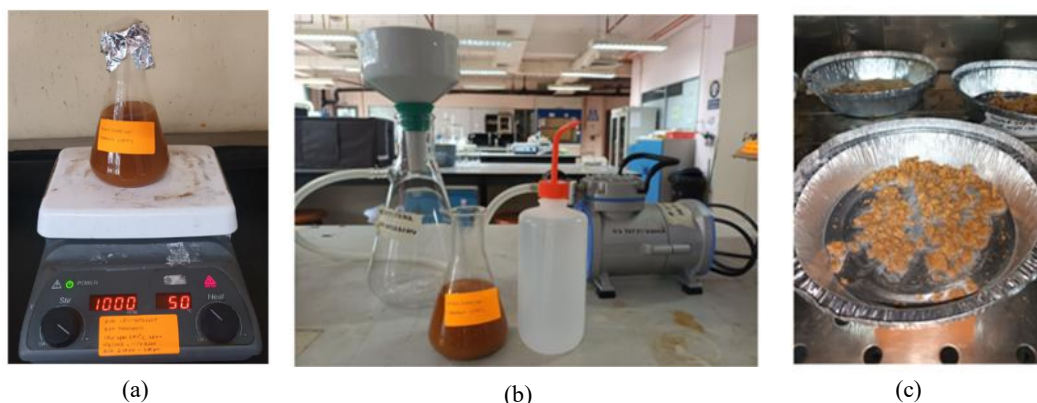


Fig. 2. (a) Heating and stirring process, (b) filtration setup, (c) drying process during the DCM

Source: Authors' own data

2.2 TAPPI 222: Determination of Lignin Content

Materials

The materials used for this process included one gram of extracted OPF fibre, 15 mL of 72% (w/w) sulfuric acid (H_2SO_4) (Chemiz, Malaysia) and distilled water. The equipment required were beakers for mixing, stirring rods, a filter for separating solids, an analytical balance (MA204, Mettler Toledo, Switzerland) for accurate weight measurement, a vacuum oven (IVO25, Taisite, United States of America) for drying and pH meter to test acidity levels during washing.

Methods

To measure lignin, one gram of extracted OPF fibre was mixed with 15 mL of sulfuric acid (H_2SO_4 , 72% (w/w)) (Chemiz, Malaysia) in a beaker. This strong acid helps break down the other components of the fibre, leaving behind the lignin. The mixture was stirred gently and allowed to react at room temperature for 2 h. After this, distilled water was added to dilute the acid, and the mixture was left undisturbed for 4 h to allow the lignin to settle. The solid lignin was separated from the liquid by filtering the mixture. The

solid residue was washed multiple times with hot water until the pH was neutral to ensure there is no acid remained in the sample. The washed residue was dried in a vacuum oven (IVO25, Taisite, United States of America) at 105 °C to remove moisture. The weight of the dried lignin was then recorded until constant weight obtained (Borrero-López et al., 2022). The lignin content was calculated using Eq. (2).

$$\text{Lignin content} = \frac{\text{Dried weight of lignin (g)}}{\text{Initial extracted OPF fibre weight (g)}} \times 100 \quad (2)$$

2.3 Wise's method: Determination of holocellulose content

Materials

The materials used in this method were extracted OPF fibres, distilled water, 10% (v/v) acetic acid (CH₃COOH) (Merck, Germany), sodium chlorite (NaClO₂) with 99% purity (Sigma Aldrich, Germany) and acetone (Chemiz, Malaysia). Other apparatus used included a 50 mL beaker, a small petri dish, a water bath, an ice bath, a vacuum oven (IVO25, Taisite, United States of America), filter papers, and a filter funnel.

Methods

The holocellulose content in the extracted OPF fibre was determined using Wise's method (Ali et al., 2023). The experiment was carried out in a fume hood for safety. Two grams of dried extracted OPF fibre was placed into a 250 mL beaker. Then, 100 mL of distilled water, 1.5 g of sodium chlorite, and 5 mL of 10% (v/v) acetic acid were added. The beaker was covered with a small petri dish and placed in a water bath at 70 °C. After 30 min, 5 mL of 10% (v/v) acetic acid was added, followed by the addition of 1.5 g of sodium chlorite after another 30 min. These additions of acetic acid and sodium chlorite were repeated every 30 min until the total amount of sodium chlorite added reached six grams. After the final addition, the mixture was heated for another 30 min and then cooled in an ice bath. The sample was then filtered using a filter funnel with filter paper and rinsed several times with distilled water at room temperature. A final rinse was done using acetone. The residue was left to air dry until a constant weight was achieved. The final dried sample was used to calculate the holocellulose content (Ali et al., 2023). The holocellulose content was calculated using Eq. (3).

$$\text{Holocellulose content (\%)} = \frac{\text{Dried OPF weight (g)}}{\text{Initial extracted OPF fibre weight (g)}} \times 100 \quad (3)$$

2.4 TAPPI 203: Determination of α-cellulose content

Materials

The materials used in this method included holocellulose sample, 17.5% (w/v) sodium hydroxide (NaOH) solution (Chemiz, Malaysia), 8.3% (w/v) NaOH solution (Chemiz, Malaysia), 2N acetic acid (CH₃COOH) (Merck, Germany) and distilled water. The apparatus involved were a 250 mL beaker, a burette, a stirring rod, a filter funnel with filter paper and a drying vacuum oven (IVO25, Taisite, United States of America).

Methods

The alpha-cellulose content was determined based on TAPPI Standard, T203 procedure. First, the holocellulose sample was weighed and placed into a 250 mL beaker. A burette was filled with 75 mL of 17.5% (w/v) NaOH solution. Then, 15 mL of the 17.5% (w/v) NaOH solution was gently added into the beaker and stirred for one minute. After that, 10 mL was added and stirred for 45 s, followed by another 10 mL which was stirred for 15 s. The mixture was then stirred again and allowed to stand for 3 min. Next, 10 mL of 17.5% (w/v) NaOH was added and stirred for 2.5 min. This step was repeated for four times.

The mixture was then allowed to stand for 30 min. After that, 100 mL of distilled water was added and stirred for another 30 min. The mixture was then filtered using a filter funnel with filter paper. The remaining residue was rinsed with 25 mL of 8.3% (w/v) NaOH solution, and all fibres were carefully transferred into the funnel. The sample was then rinsed with 650 mL of distilled water. After rinsing, 2N acetic acid was gently poured over the residue and allowed to stand for 5 min. The liquid was then drained, and the residue was rinsed again with 150 mL of distilled water. Finally, the residue was carefully placed on the tray and dried in an oven until a constant weight was reached (Ali et al., 2023). The α -cellulose content was calculated using the Eq. (4).

$$\alpha\text{-cellulose Content (\%)} = \frac{\text{Dried holocellulose weight (g)}}{\text{Initial holocellulose weight (g)}} \times 100 \quad (4)$$

2.5 Wise's method: Determination of holocellulose content

The hemicellulose content was not measured directly but was calculated from the results of holocellulose and α -cellulose. The formula to calculate the hemicellulose content is shown in Eq. (5).

$$\text{Hemicellulose Content} = \text{Holocellulose content} - \alpha\text{-cellulose content} \quad (5)$$

3. RESULTS AND DISCUSSION

3.1 Extraction yield of OPF fibre

The findings from this study indicate that OPF fibres can be effectively treated using a direct chemical treatment method to remove lignin while retaining the cellulose content. The yield of extraction obtained for the three OPF samples were 51.2 wt%, 50.6 wt% and 52 wt% respectively as shown in Table 1 below. The average extraction yield is 51.3 ± 0.7 wt%. This value is consistent and does not differ significantly from the findings reported by Abdul Khalil et al. (2025), who achieved a 57.2 wt% extractive yield under similar chemical treatment conditions. The consistent trend of result suggests that the chemical treatment method used combining nitric acid and hydrogen peroxide at controlled temperature and time which is at 50 °C and 48 h respectively was effective and reliable in breaking down the lignocellulosic matrix of OPF fibre.

The small variation in yield across the samples which ranges from 50.6 wt% to 52 wt% may be due to natural differences in the fibre structure or moisture content of each OPF sample. However, the overall

consistency supports the reliability of the treatment process. The relatively high extractive yield confirms that a significant portion of non-cellulosic and partially degraded hemicellulose or lignin content was removed which is necessary to isolate pure cellulose for further analysis (Ali et al., 2023; Abdul Khalil et al., 2025). These results are in line with previous findings that highlight the effectiveness of oxidative chemical treatments in degrading complex biomass structures. By removing unwanted components, the chemical treatment helps to enhance the purity of the cellulose. This enhances the potential of the OPF as a feedstock for application such as bioplastic production.

Table 1. Extractive yield of OPF samples after direct chemical treatment

No. of Sample	OPF Weight (g)		Yield of Extraction (wt%)
	Before Treatment (g)	After Treatment (g)	
1	5.00	Day 1	14.00
		Day 2	2.56
		Day 3	2.56
2	5.00	Day 1	15.10
		Day 2	2.53
		Day 3	2.53
3	5.00	Day 1	15.85
		Day 2	2.60
		Day 3	2.60
Average			51.3 ± 0.7

Source: Author's own data

3.2 Compositional analysis of oil palm frond (OPF)

The quantitative analysis found that the extracted OPF fibre contains 7 ± 0.50 wt% of unremoved lignin during direct chemical treatment and 69 ± 0.90 wt% of holocellulose. The holocellulose content possess 32 ± 4.10 wt% of cellulose and 37 ± 4.54 wt% of hemicellulose as shown in Table 2. The unremoved lignin and holocellulose components accounted for approximately 76 wt% of the total sample, leaving about 24 wt% unaccounted for in the overall composition. This remaining fraction is likely attributable to ash, crude protein, lipids, phenolics, and other extractive substances that were not quantified by the TAPPI or Wise methods employed in this analysis (Hassan et al., 2019; Rahim et al., 2022). These extractives are typically present in plant biomass and are often removed during standard procedure. However, in some cases, methods such as direct chemical treatment may not completely eliminate non-structural components like ash and crude protein. As a result, part of these extractives may still remain in the treated sample and contribute to the unaccounted fraction observed in the final composition. According to a study by Chanjula et al., (2018), OPF fibre contains around 6.12 wt% of ash and 15.30 wt% of crude protein, which together explain a significant portion of the missing percentage in this study. This supports the idea that extractive substances may still be present in the extracted OPF fibre and contribute to the unmeasured portion of the sample. Fig. 3 shows the raw OPF fibre, extracted OPF fibre, lignin, holocellulose and cellulose in the extracted OPF fibre.

Table 2. Percentage composition of lignocellulosic components in OPF

Analysis Composition in Extracted OPF Fibre	
Component	Percentage Composition (%)
Lignin	7 ± 0.50
Holocellulose	69 ± 0.90
Cellulose	32 ± 4.10
Hemicellulose	37 ± 4.54

Source: Author's own data

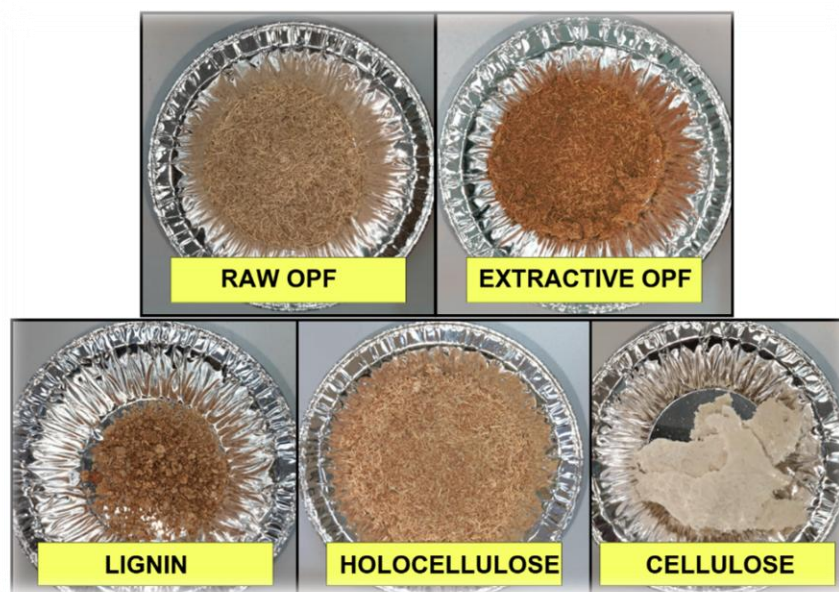


Fig. 3. Raw OPF fibre before treatment, extracted OPF fibre obtained after DCM, lignin obtained using TAPPI 222, holocellulose obtained using Wise's method and cellulose obtained using TAPPI 203

Source: Authors' own data

Although the chemical treatment used in this study was effective for isolating lignocellulosic components such as cellulose and lignin, it may not have completely eliminated all extractives. In some cases, even strong oxidative treatments like the one applied here may leave behind residues of inorganic matter or proteins. Studies from Abolore et al. (2024); Horhammer et al. (2018) have also reported that standard chemical methods sometimes fail to fully remove these components especially if the treatment is not specifically targeted at them.

The presence of extractives like ash and proteins is important to consider because they can affect downstream processing. For example, ash can interfere with enzymatic reactions and reduce the efficiency of bioconversion (Zhang et al., 2019), while proteins and lipids may influence the structural or mechanical

properties of bio-based materials (Lehr et al., 2021). If these substances are not fully removed, they could lower the quality or performance of the final product.

3.3 FTIR analysis

Fourier Transform Infrared (FTIR) spectroscopy (Spectrum 100 FTIR, Perkin Elmer, United States of America) was used to analyse the chemical changes in OPF samples before and after direct chemical treatment. The FTIR spectra, as shown in Fig. 4 demonstrates clear differences in functional group intensities and peak positions which indicate effective direct chemical treatment method.

In the raw OPF fibre (black spectrum) as shown in Fig. 4, a broad absorption peak at 3264.54 cm^{-1} corresponds to O–H stretching vibrations commonly associated with hydroxyl groups in cellulose, hemicellulose, and lignin. After treatment, this band shifted to 3322.01 cm^{-1} with reduced intensity, suggesting partial removal of hemicellulose and lignin components, which contain abundant hydroxyl groups. This observation aligns with findings by Azizan et al. (2022); Abdul Khalil et al. (2025), who reported similar shifts in hydroxyl bands after oxidative chemical treatment of OPF fibre and other biomass. The C–H stretching vibrations are evident at 2911.19 cm^{-1} (raw OPF fibre) and 2893.39 cm^{-1} (extracted OPF fibre) where a reduction in intensity post treatment indicates a breakdown of aliphatic chains from lignin and waxy substances (Azizan et al., 2022).

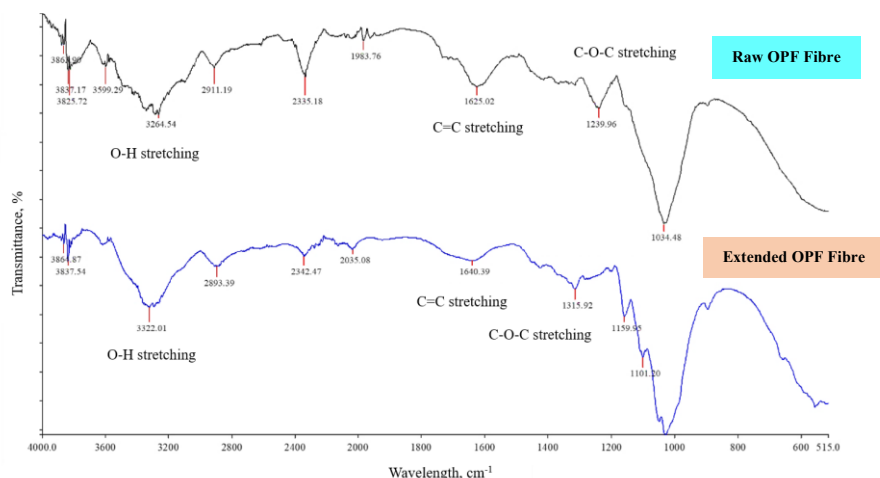


Fig. 4. FTIR spectra of raw OPF fibre before treatment and extracted OPF fibre after DCM

Source: Authors' own data

One of the most notable changes occurs at 1625.02 cm^{-1} in the raw OPF fibre, which is attributed to aromatic C=C stretching in lignin. After treatment, the corresponding peak is weakened and shifted to 1640.39 cm^{-1} , confirming partial degradation or removal of lignin (Apaydin Varol & Mutlu, 2023). This supports the goal of reducing lignin content to isolate pure cellulose for further applications. In the fingerprint region, raw OPF fibre displays strong peaks at 1239.96 cm^{-1} and 1034.48 cm^{-1} , representing C–O–C stretching in cellulose and hemicellulose. After treatment, these peaks decrease in intensity and shift to 1159.95 cm^{-1} and 1101.20 cm^{-1} , reflecting cleavage or alteration of glycosidic bonds in

hemicellulose and exposure of cellulose structure (Azizan et al., 2022). An additional new band was found at 1315.92 cm^{-1} in the extracted OPF fibre spectrum may relate to enhanced deformation vibrations of cellulose, which becomes more dominant after hemicellulose and lignin are removed (Abdul Khalil et al., 2025).

These results confirm that the combined use of nitric acid and hydrogen peroxide at $50\text{ }^{\circ}\text{C}$ for 48 h effectively disrupts the lignocellulosic matrix. The FTIR results are consistent with the extractive yield and compositional analysis which demonstrating the successful removal of amorphous hemicellulose and lignin fractions. While treatment was effective in enriching cellulose, some residual non-structural extractives such as proteins and ash may remain as supported by Azizan et al. (2022) who noted that chemical pretreatments may not completely remove all inorganic components from biomass.

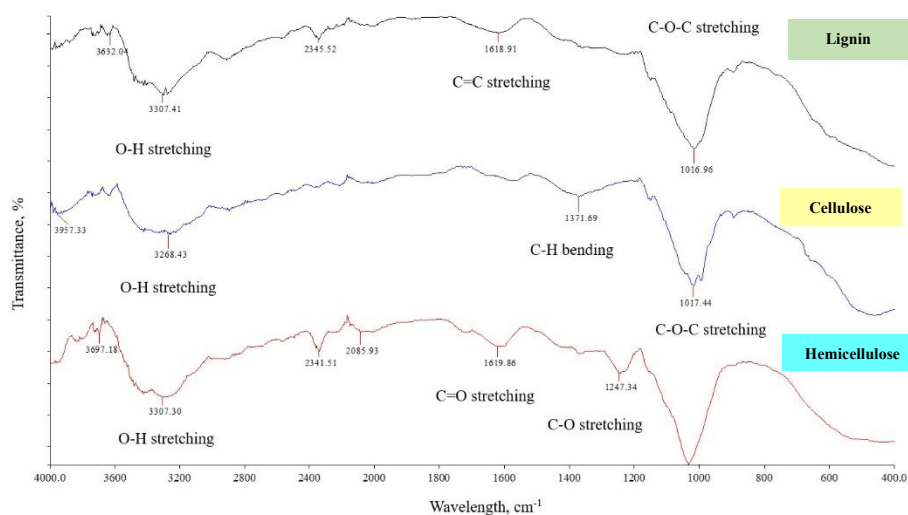


Fig. 5. FTIR spectra of lignin obtained using TAPPI 222, cellulose obtained using TAPPI 203 and holocellulose obtained using Wise's method

Source: Authors' own data

Furthermore, Fig. 5 shows the FTIR spectrum of extracted OPF composition, which includes lignin, cellulose, and holocellulose. Each spectrum displays unique peaks that represent the specific chemical bonds present in the three major lignocellulosic components. These differences in peaks confirm that the treatment process successfully separated the components and retained their individual structures. For the lignin sample, a broad peak around 3307.41 cm^{-1} was observed which corresponds to the O–H stretching vibration. This peak is common in phenolic and alcohol groups found in lignin. A strong band at 1618.91 cm^{-1} is related to the C=C stretching of aromatic rings, confirming that the extracted lignin still contains its aromatic structure. In addition, the peak at 1016.96 cm^{-1} is assigned to the C–O stretching in ether or phenolic groups, which are also typical features of lignin (Azizan et al., 2022; Kaur et al., 2022).

The cellulose spectrum shows a sharp O–H stretching peak at 3268.43 cm^{-1} , which indicates strong hydrogen bonding between cellulose chains. This type of bonding is important for maintaining cellulose's crystalline structure. A smaller peak at 1371.69 cm^{-1} corresponds to C–H bending, a signature of cellulose.

The key peak appears at 1017.44 cm^{-1} , which represents C–O–C stretching in the β -(1 \rightarrow 4)-glycosidic linkages of the cellulose backbone. These peaks suggest that the cellulose structure was preserved during extraction (Md Salim et al., 2021). The spectrum for holocellulose which includes both cellulose and hemicellulose, also shows an O–H stretching peak at 3307.30 cm^{-1} . However, this peak is broader than in cellulose, likely due to the more amorphous nature of hemicellulose. A peak at 1619.86 cm^{-1} may indicate C=O stretching from acetyl or ester groups in hemicellulose or traces of remaining lignin. A noticeable band at 1247.34 cm^{-1} is linked to C–O stretching in acetylated sugars found in hemicellulose (Javier-Astete et al., 2021).

4. CONCLUSION

The yield of extraction obtained is $51.3 \pm 0.7\text{ wt\%}$ using a direct chemical treatment. FTIR analysis further supported this finding by showing clear changes in chemical bonds. The shifts in peak positions confirmed the removal of hemicellulose and lignin, while the presence of cellulose was clearly identified. This proves that the treatment method was effective in extracting the cellulose fibre from OPF. The quantitative analysis of the actual cellulose, hemicellulose, and lignin content in the extracted OPF fibre was successfully conducted using the TAPPI standard methods and Wise's method. The extracted OPF fibre contains approximately $32 \pm 4.10\text{ wt\%}$ of cellulose, $37 \pm 4.54\text{ wt\%}$ of hemicellulose, and $7 \pm 0.50\text{ wt\%}$ of lignin. These findings provide valuable insight and guidance for the appropriate application of the material as a reinforcing agent.

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CONFLICT OF INTEREST STATEMENT

The authors declare that this research was conducted in the absence of any commercial or financial relationships that could be construed as a potential conflict of interest. No conflicting interests exist with the funders.

AUTHORS' CONTRIBUTIONS

Nur Ain Razak: Conceptualisation, lab work execution, formal analysis, investigation and writing-original draft; **Suffiyana Akhbar:** Conceptualisation, formal analysis, review-editing and validation; **Sakinah Mohd Alauddin:** Conceptualisation and validation.

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