



SPECTRAL ANALYSIS OF IRAQI AMBER RICE HUSK COMPONENTS PRODUCER OF ECO-FRIENDLY BIOPLASTIC

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Received 29/ 9/ 2024, Accepted 11/ 12/ 2024, Published 30/ 9/ 2026

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ABSTRACT

Reducing agricultural waste is necessary to prepare environmentally friendly materials at a low cost and economic feasibility. In this research, cellulose was extracted from Amber rice husks (ARH) using three types of extractions: 12% sodium hydroxide, an autoclave for 30 min, treatment with 25% food waste fermentation extract for 7 d, then bleaching the products for one hour at 80 °C by 2.5% sodium hypochlorite. The percentage of extracted cellulose was 45.62, 61.05, and 45.06% for the chemical, physical, and biological extractions, respectively, with the best rate for the physical treatment based on the remaining concentrations of lignin being 5.17, 3.16, and 3.33% respectively. Fourier transform infrared spectroscopy (FTIR) spectra of pure cellulose and cellulose that was extracted using the chemical, physical, and biological methods from Amber rice husks observed stretching vibration of O-H and C-H bonds in polysaccharides is characterized by wave number 3500-3000 cm⁻¹, with peaks at 3400.50, 3433.29, 3415.93, and 3437.15 cm⁻¹ respectively, broad peaks 2908.65, 2926.01, 2922.16, and 2924.09 cm⁻¹ respectively, is thought to be caused by the CH stretching vibration of all hydrocarbon constituents in polysaccharides. In cellulose, the vibrations of absorbed water molecules are represented by the peaks at 1633 cm⁻¹ correspond with bands at 1637.56, 1641.42, 1647.21, and 1637.56 cm⁻¹ respectively, on the other hand, absorbance at 1431.18, 1377.17, 1382.96, and 1431.18 cm⁻¹ respectively, is associated with the stretching and bending vibrations of the cellulose (-CH₂), (CH), (OH), and (C-O) bonds. At the same time, the stretch vibration of the (C-O-C) bonds in the 1,4-glycosidic linkage that connects the d-glucose units to cellulose is therefore referred to as absorbance at 1161.15, 1095.57, 1097.50, and 1103.28 cm⁻¹ respectively. Thus, the amorphous region of cellulose is responsible for the peaks located at 898.83, 910.40, 796.60, and 794.67 cm⁻¹ respectively.

Keywords: Environmental waste, Environmental Sustainability, biological treatment, chemical treatment, FTIR, physical treatment.



التحليل الطيفي لمكونات قشور الرز العنبر العراقي المنتجة للبلاستيك الحيوي الصديق للبيئة

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الخلاصة

أصبح التخلص من المخلفات الزراعية ضرورة لتحضير مواد صديقة للبيئة بتكلفة منخفضة وجدوى اقتصادية. في هذا البحث، تم استخلاص السليلوز من قشور الرز العنبر (ARH) باستخدام ثلاثة أنواع من طرق الاستخلاص: 12% هيدروكسيد الصوديوم، الأوتوكلاف لمدة 30 دقيقة، المعالجة بمستخلص تخمير بقايا الطعام 25% لمدة 7 أيام، ثم قصر المنتجات لمدة ساعة واحدة بمعاملتها مع هيبوكلوريت الصوديوم 2.5% عند 80 درجة مئوية. وكانت نسبة السليلوز المستخرج 45.62، 61.05، و 45.06% للاستخلاص الكيميائي، الفيزيائي، والبيولوجي على التوالي، مع أفضل نسبة للمعالجة الفيزيائية على أساس التراكيز المتبقية من اللجنين هي 5.17، 3.16، و 3.33%. تم مقارنة أطياف التحليل بالأشعة تحت الحمراء (FTIR) من السليلوز النقي والسليلوز الذي تم استخلاصه باستخدام الطرق الكيميائية والفيزيائية والبيولوجية من قشور الرز العنبر، واهتزاز روابط OH و CH في السكريات الممتدة برقم موجة 3000-3500 سم⁻¹، بالقمم 3400.50، 3433.29، 3415.93، و 3437.15 سم⁻¹ على التوالي، يُعتقد أن القمم العريضة 2908.65، 2926.01، 2922.16، و 2924.09 سم⁻¹ على التوالي، ناجمة عن اهتزاز تمدد CH لجميع مكونات الهيدروكربون في السكريات. في السليلوز، يتم تمثيل اهتزازات جزيئات الماء الممتصة بالقمم عند 1633 سم⁻¹ والتي تتوافق مع الحزم 1637.56، 1641.42، 1647.21، و 1637.56 سم⁻¹ على التوالي، من ناحية أخرى، الامتصاصية عند 1431.18، 1377.17، 1382.96، و 1431.81 سم⁻¹ على التوالي يرتبط باهتزازات التمدد والانحناء لأواصر السليلوز (CH₂-)، (CH)، (-OH)، (CO-O)، في حين أن اهتزاز التمدد لروابط C-O-C في الروابط 4،1- يشار إلى الارتباط الجليكوسيدي الذي يربط وحدات الجلوكوز d- بالسليلوز على أنه امتصاص عند 1161.15، 1095.57، 1097.50، و 1103.28 سم⁻¹ على التوالي، وبالتالي، فإن المنطقة غير المتبلورة من السليلوز هي المسؤولة عن القمم الواقعة عند 898.83، 910.40، 796.60، و 794.67 سم⁻¹ على التوالي.

الكلمات المفتاحية: المخلفات البيئية، الاستدامة البيئية، المعالجة البيولوجية، المعالجة الكيميائية، FTIR، المعالجة الفيزيائية.

INTRODUCTION

Around half of the world's population depends on rice as a food source. Approximately 21 percent of human energy is needed, and up to 76% of Southeast Asians' caloric intake comes from rice, making it one of the world's most important crops (Zhao *et al.*, 2020). Rice husks are essentially burned as agricultural waste or routinely disposed of in landfills, contaminating the environment. Growing environmental concerns have limited scientists' ability to extract materials from plants. As cellulose, hemicellulose, and lignin are present in the organic portion of rice husk in literature, this provides the basis for cellulose extraction from rice husk. Plant cell walls are a renewable source of the polysaccharides hemicellulose (12-29%), cellulose (23-50%), and the aromatic polymer lignin (13-31%) (Sharma *et al.*, 2019). The plant's cell walls are rigid because cellulose is β -d-glucose repeating units connected by β -1, 4-glycosidic bonds (Adewuyi, 2022).



As a polymeric substance with numerous applications and no environmental effects, cellulose from natural fibers such as rice husks is a carbon source (**Abbas et al., 2023**). These applications present commercial and industrial technological opportunities in automotive, electronics, and packaging materials (**Norrrahim et al., 2021**). As opposed to synthetic fibers, which not only have a detrimental effect on human health but also air pollution, which exacerbates the world energy crisis, produces respiratory illnesses, and emits greenhouse gases, which are one of the factors contributing to global warming, during production (**Al-Rubaie & Abdulhay, 2022**).

However, in environmental degradation and waste management, plastic waste like polyethylene terephthalate (PET) or polyvinyl chloride (PVC) is exerted in the environment, not degrading rapidly, resulting in pollution, and increased tensile and compressive strength, commonly known as the so-called white pollutants (**Hameed & Talib, 2018**). Microorganisms, such as bacteria and fungi extracted from soil, artificial clay, compost, sludge, and water, have been utilized to degrade hydrocarbon waste and control plastic accumulation while used as a carbon source (**Nafal & Abdulhay, 2020; Sabei et al., 2024**). Also, artificial plastic, polystyrene, polyethylene, and ethylene vinyl acetate, were ingested by some insects, such as the flour beetle and the larvae of *Galleria mellonella* (**Abdulhay, 2020; Khlaif & Abdulhay, 2024**).

Bioplastics from agro-polymers are derived from natural polymers such as polysaccharides (starch, cellulose, pectin, hemicellulose) and proteins (casein, zein, gluten, and gelatin) that generally involve intra-and intermolecular interactions and cross-links (crosslinking) between polymeric constituents, forming a semi-rigid three-dimensional polymeric network that retains the solvent (**Tharanathan, 2003**).

Polysaccharides, the most abundant macromolecules in flora and fauna, are one of the most suitable raw materials for bioplastics in the form of starch, which is renewable and sustainable but plentiful and cheap. Starch also has favorable thermoplastic properties and is biodegradable (**Thakur & Thakur, 2016**).

Based on the study of the abundance of plastic waste, bioplastics derived from starch, pectin, and cellulose are biodegradable by microbes (**Chodijah et al., 2019**). First-preferred rice husk cellulose is a thermoplastic material, more water-resistant than starch, and can package films (**Asgher et al., 2020**).

According to **Tata et al. (2023)**, FTIR spectra characterized the unknown samples and determined the chemicals present in the sample. For instance, the technique is employed in measuring the improvement of rice husk structure that undergoes physio-chemical treatment (**El-Azazy et al., 2021**). Depending on the proposition that the infrared frequency of light corresponds with the vibrational frequencies of the chemical bonding carried by molecules triggering vibration in the molecular bonds (**Weber et al., 2023**). The peaks of the



electromagnetic spectrum in the infrared portion involve the use of distinct absorption bands for identified inorganic and organic compounds, and the detection of functional groups of organic compounds (El-Azazy *et al.*, 2021).

The objectives of this research are as follows: a) Isolation of cellulose from ARH using chemical, physical, and biological approaches b) Characterization of the functional groups of chemical, physical, and biological extraction products using FTIR.

MATERIALS AND METHODS

Collection of ARH

At the Scientific Research Commission for Advanced Materials Center, ARH was washed, dried for an hour at 60 °C in an oven, ground into a fine powder with a laboratory disc grinder, and sieved using 80 meshes, then the cellulose extracted by using chemical, physical, and biological methods (Yunus *et al.*, 2019).

Chemical extraction

Chemical extraction of cellulose from ARH was done by employing sodium hydroxide (NaOH) to remove lignin and sodium hypochlorite (NaOCl) for bleaching. This method involved, dissolving 50 g of ARH in the powdery form in 500 ml of 12% NaOH solution, and heating the resultant solution to 80 °C for 3 hours. After that, the precipitate was treated with a 2.5 percent NaOCl solution that was heat-activated at 80 °C for an hour, and then the samples were washed with distilled water until the pH became neutral. The precipitated cellulose was successfully washed with distilled water until an end-point was reached and dried at 60 °C to a constant weight (Yunus *et al.*, 2019; Karan *et al.*, 2019).

Physical extraction

Fifty grams of ARH powder and 500 ml of distilled water were combined, and the mixture was autoclaved for 30 min at 121 °C to carry out the physical extraction. Following the mixture's filtering, a 2.5% NaOCl solution was used to bleach the ARH for one hour at 80 °C. It was washed several times to get the precipitate's pH to be neutral. The solid residue was dried at 60 °C until it reached a constant weight, according to Yunus *et al.* (2019).

Biological extraction

For the biological extraction, 500 ml of 25% crude garbage enzyme that was extracted from the fermentation of household food waste was added to 50 g of dried ARH, which was then treated and incubated for 7 d at room temperature in a shaker incubator. Precipitate the ARH and washed twice by distilled water. Next, it was heated for an hour at 80 °C to bleach it using 500 ml of NaOCl (2.5%) solution agent. The precipitation was cleaned with distilled water until it reached a neutral pH value, and then it was dried in an oven at 60 °C to a constant weight, according to Andalia *et al.* (2020).

Estimating the proportions of ARH components

The proportion of fibers, including cellulose and hemicellulose, extracted from ARH through chemical, physical, and biological extraction methods was estimated at the Fiber Analyzer Device Consulting Office (College of Agricultural Engineering Sciences, University of Baghdad).

Analysis of the extraction products by FTIR

Cellulose extracted from all types of extraction and pure cellulose were analyzed for the functional group using an FTIR device (Shimadza / Japan) at wavelengths between 4000 and 400 cm^{-1} , the spectrum is captured in the absorbance mode, because of the differences in their atom counts and bond strengths, functional groups and their classes have particular vibrations (Kassem *et al.*, 2023).

RESULT AND DISCUSSION

Cellulose extraction

The results of the cellulose extraction process from Amber rice husks, using the chemical, physical, and biological extraction methods demonstrated that the percentage of cellulose concentrations extracted with all the above when using ARH is 45.62, 61.05, and 45.06%, respectively, as shown in Figure (1).

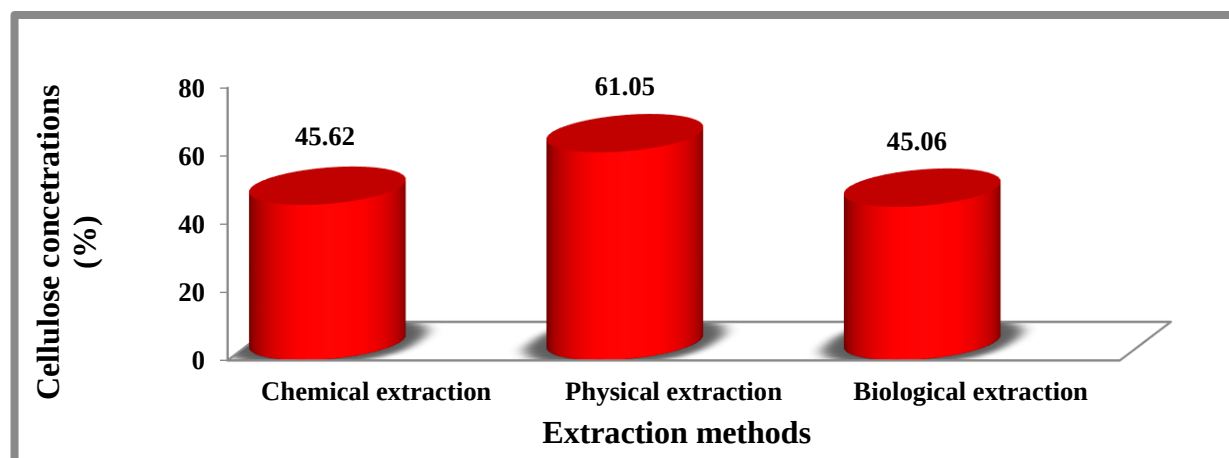


Figure (1): Percentage of cellulose extracted from ARH by chemical, physical and biological extractions.

Chemical extraction

Sodium hydroxide solution increases the yield of cellulose fiber during chemical extraction by breaking hydrogen bonds between the lignocellulosic components, which dissolve in alkali solutions to form black liquor (Kucharska *et al.*, 2018). After the alkali extraction, the lignin was removed using NaOCl as a bleaching agent because NaOH alone was



unable to remove all of the lignin. NaOCl contains a hypochlorite ion, a potent oxidant that can dissolve the ether bond in the lignin structure and improve the pulp's white brightness, according to **Aridi et al.(2021)** .

Strong hydrogen bonds in cellulose's structure prevented it from dissolving at a temperature below its degradation point (**Keshk, 2015**). Nevertheless, it was discovered that raising the NaOH concentration reduced the yield since some cellulose chains would break down during the extraction process at greater concentrations. Elevated concentrations of NaOH disrupt certain crystalline areas of cellulose, facilitating its easy dissolution in the solution treatment and consequently decreasing the cellulose fiber yield (**Utoro et al., 2022**).

Physical extraction

Hydrothermal recovery is by far an uncomplicated and inexpensive method of extraction. In the case of hydrothermal pretreatment without chemical addition, the following physical changes occurred, reduction of lignin on the cellulose surface, dissolution of hemicellulose, and enhanced cellulose structure permeability (**Martin et al., 2022**). When cellulose is wet, the water forms hydrogen bonds of the -OH groups in the cellulose structure; hence, the cellulose is least packed, and the chain becomes less mobile because of the increasing inter-chain bonds due to the withdrawal of water from the cellulose and the degree of crystallinity increases.

Biological extraction

Biological extraction, which involves soaking ARH in a 25% extract of raw garbage enzymes for a week, is thought to be an environmentally friendly method because it doesn't require the use of alkali or acidic materials like chemical treatment, even though the percentage of cellulose extracted by chemical and biological methods is similar as 45.62 and 45.06% respectively.

When lignin concentration in the ARH after chemical, physical, and biological extractions is measured in a fiber estimation device, the results are identical to the cellulose extraction results because the physical method was found to be the most effective in removing lignin and extracting cellulose and hemicellulose when compared to the percentage of lignin in the ARH before treatments, as shown in Figure (2).

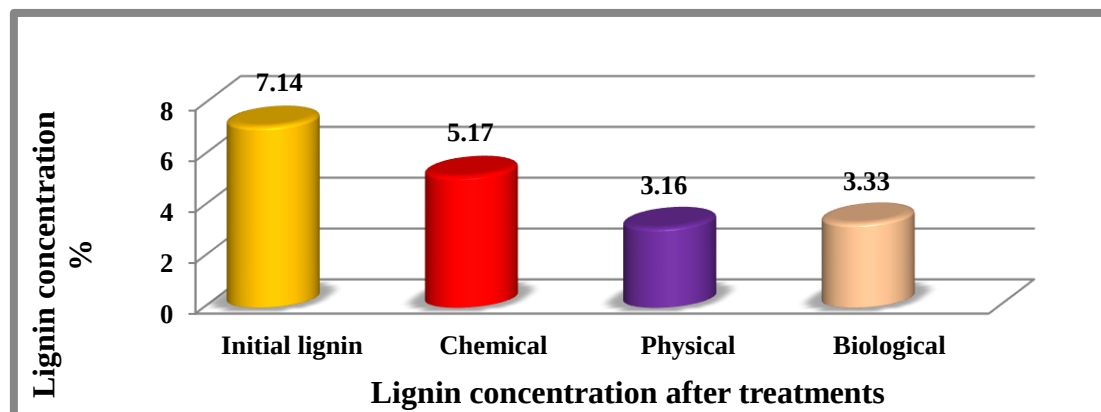


Figure (2): Lignin percentage of ARH after the chemical, physical, and biological treatment methods

The delignification and cellulose pulp formation processes may impact the chemical bonds that bind cellulose, hemicellulose, and lignin together conversely, the solvent eliminated or dissolved hemicellulose and lignin (**Smink et al., 2019**). Experiments reported by **Bhatia et al. (2020)** & **Pramasari et al. (2021)**, are an alkaline pretreatment employing 3% NaOH coupled autoclaving at 121 °C. Alkaline agents (NaOH) combine high-temperature work on efficient pretreatment for delignification by rupturing hydroxyl ion (OH⁻) linkages between lignin and hemicellulose and dissolving the lignin polymer into its aromatic derivatives (**Bhatia et al., 2020**).

FTIR analysis

FTIR spectra of cellulose-containing materials display the absorbance spectra at various peaks, between 4000-400 cm⁻¹ wave numbers (**Smink et al., 2019**), there was a broad spectrum of spectral scales where the absorbance peaks were found. Within Using FTIR spectra, the functional group of cellulose is identified in (Fig. 3, Fig. 4, Fig. 5, and Fig. 6). Other compounds that contain nitrogen and are important in biochemistry and pharmaceuticals made from plants include alcohol, phenols, and primary, secondary, and amide amines, these compounds can undergo a wide range of reactions that lead to complete ionization. Alkenes and alkynes are unsaturated hydrocarbons ready to polymerize to saturation, aromatics and aliphatic amines can combine with halogen to form alkyl halides. Based on the analysis, cellulose extracted from chemical, physical, and biological extraction methods for ARH contains all the functional groups in pure cellulose.

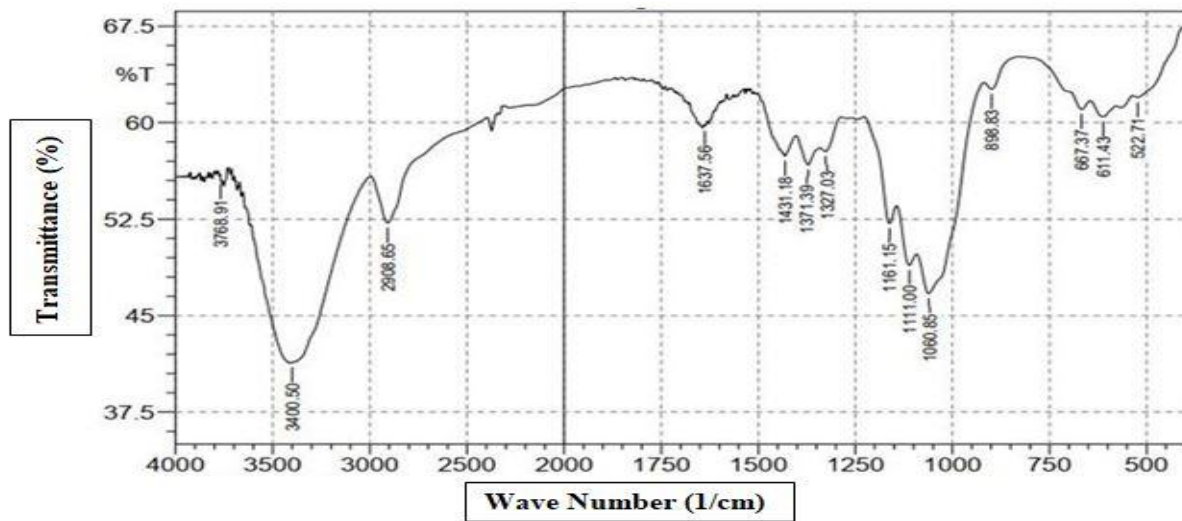


Figure (3): The FTIR absorption spectra of pure cellulose.

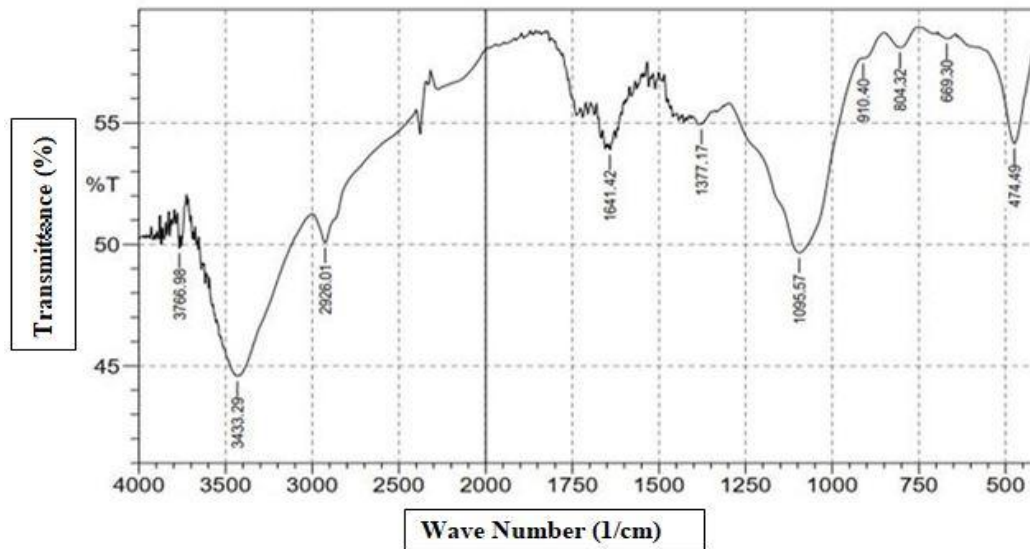


Figure (4): ARH cellulose's FTIR absorption spectra after chemical treatment.

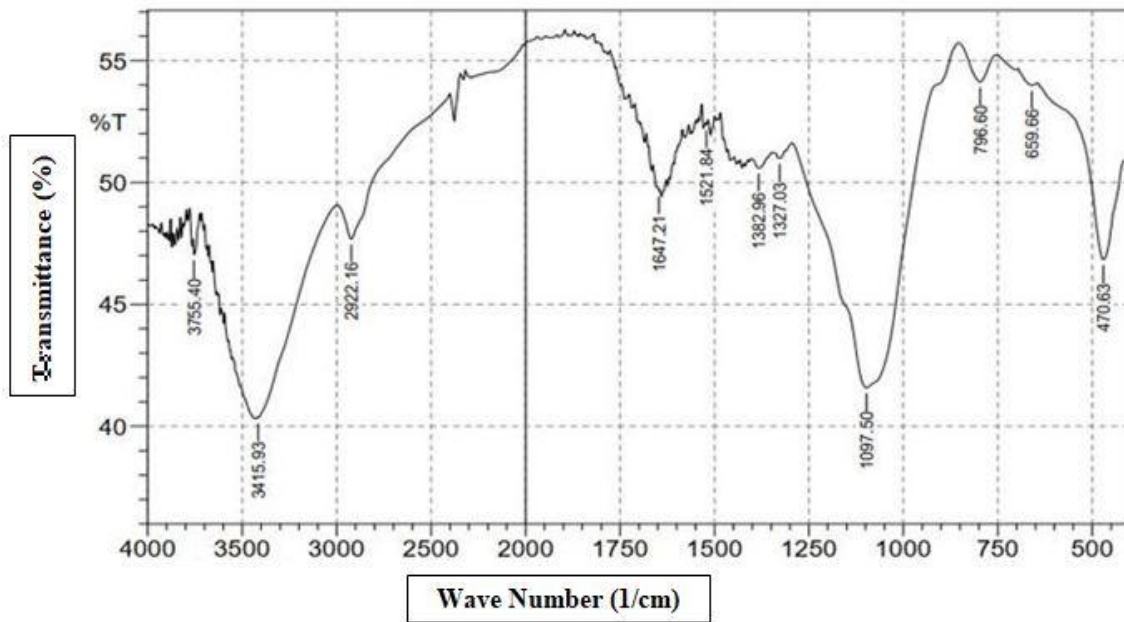


Figure (5): ARH cellulose's FTIR absorption spectra after physical treatment.

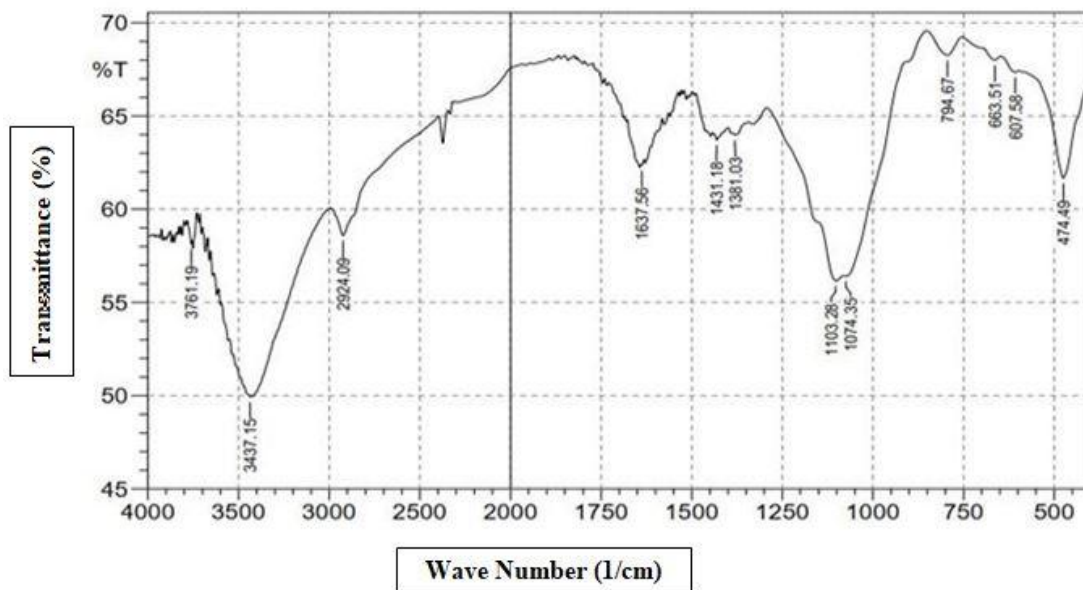


Figure (6): ARH cellulose's FTIR absorption spectra after biological treatment.



Numerous peaks can be observed in the FTIR spectra of pure cellulose and cellulose that was extracted using the chemical, physical, and biological methods from ARH (Fig. 3, Fig. 4, Fig. 5, and Fig. 6). Stretching vibration of O-H and C-H bonds in polysaccharides is characterized by wave number 3500-3000 cm^{-1} , with peaks at 3400.50, 3433.29, 3415.93, and 3437.15 cm^{-1} for pure cellulose, chemical, physical, and biological extractions, respectively. The wide peak at 3331 cm^{-1} is hydroxyl group's stretching vibration in polysaccharides, which also includes hydrogen bond vibrations between and within molecules in cellulose (**Popescu et al., 2011**).

Broad peaks 2908.65, 2926.01, 2922.16, and 2924.09 cm^{-1} for pure cellulose, chemical, physical, and biological extraction methods, respectively, are similar to the band at 2894 cm^{-1} , which is thought to be caused by the CH stretching vibration of all hydrocarbon constituents in polysaccharides, at wave number 3000-2500 cm^{-1} (**Poletto et al., 2011**),

Usually, bands ranging from 1630 to 900 cm^{-1} contain cellulose. In cellulose, the vibrations of absorbed water molecules are represented by the peaks at 1633 cm^{-1} . At 1637.56, 1641.42, 1647.21, and 1637.56 cm^{-1} , these peaks were measured in pure cellulose and cellulose extracted by chemical, physical, and biological methods, respectively. Otherwise, absorbance at 1431.18, 1377.17, 1382.96, and 1431.18 cm^{-1} is associated with the stretching and bending vibrations of the cellulose (-CH₂), (-CH), (-OH), and (C-O) bonds (**Xu et al., 2014**).

The absorbance at 1161.15, 1095.57, 1097.50, and 1103.28 cm^{-1} for pure cellulose and cellulose extracted by chemical, physical, and biological methods, respectively, is referred to the stretch vibration of the C-O-C bonds in the 1,4-glycosidic linkage that connects d-glucose units of cellulose molecules. Accordingly, the peaks at 898.83, 910.40, 796.60, and 794.67 cm^{-1} , respectively, are caused by the amorphous region of cellulose (**Song et al., 2019**).

CONCLUSION

Extracted cellulose in the current study was applied to chemical, physical, and biological processes involving agricultural waste (ARH). The physical and biological treatment has proven to be efficient in removing lignin from cellulose without the use of chemicals and is, therefore, a green process. The results also show that for FTIR modeling cellulose obtained from ARH gives similar results as compared to pure cellulose.

Authors' Contribution Statement

The Author E.H. contributed to implementing the manuscript plan steps, conducting some work-related transactions, analyzing the data, and contributing to the interpretation of the results and provided critical revisions, references of the manuscript preparation, and writing of the manuscript with support from Author A. H. The Author H.S. contributed to the review and proofreading of the manuscript (supervisor). The Author A.H. contributed to developing the plan, practically supervising the research, preparing and providing research supplies of materials and laboratories, and reviewing and auditing the research plan as a co-supervisor,

Declaration of competing interest



The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgment

The researchers extend their sincere thanks to the employees of the Pollutants Treatment Department and Food Research Department laboratories in the Research and Technology Center of Environment, Water and Renewable Energy in the Scientific Research Commission, for conducting experiments on cellulose extraction and FTIR analyses.

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