Stabilized Biochar from Anaerobic Digestion as a Sustainable Strategy for Global Warming Mitigation

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ABSTRACT

The Sultanate of Oman grapples with mounting agricultural waste, particularly palm tree residue, necessitating intervention. This study proposes converting palm waste, specifically Date Palm Empty Fruit Bunches (DPEFB), into eco-friendly products, such as biochar, fertilizers, and methane, to mitigate environmental hazards associated with illegal disposal methods, such as burning. The objectives include extracting lignin, cellulose, and hemicellulose from DPEFB, followed by carbonization to produce biochar at varying temperatures. Analytical techniques, including X-Ray Diffraction (XRD), Thermogravimetric Analysis (TGA), Fourier Transform Infrared Spectroscopy (FTIR), Raman spectroscopy, and Nuclear Magnetic Resonance (NMR) spectroscopy, are used to characterize the samples. The results indicate optimal cellulose, lignin, and hemicellulose extraction at particle sizes of 250–500 µm, with biochar conversion showing promising results under specific conditions. The study's significance lies in being the first in Oman to demonstrate the partial anaerobic decomposition of biochar with wastewater and

elucidate the roles of extracted samples and biochar. However, the absence of predictive models for biogas production from biochar remains a challenge. Future implementation entails integrating findings with the One Million Palm Tree Project in state of Ibri, utilizing palm residues for fertilizer and clean methane gas production, supplemented by solar energy for sustainability.

Keywords-agricultural waste; carbon fraction; DPEFB; lignocellulosic, biochar; bio-methane gas

I. INTRODUCTION

Oman has witnessed rapid growth in municipal solid waste generation due to increasing urbanization and population. This growth has burdened the existing waste management infrastructure and increased the sector's carbon footprint. Thus, an integrated waste management system is required along with adopting waste-to-energy technologies [1]. In the Sultanate of Oman, the annual volume of agricultural waste generated solely from date palms amounts to approximately 163 million kg. Additionally, sewage waste, specifically the anaerobic sludge produced by Haya, remains underutilized without a defined economic purpose. The prevailing waste management practices in Oman mainly involve disposal, with approximately 90% of waste sent to landfill sites. Only a marginal proportion, constituting 10%, undergoes treatment following the principles of the 3R approach reuse, reduce, and recycle [2].

Hence, the principal objective of this investigation is to produce biochar and biomethane gas from DPEFB through the extraction and characterization of cellulose, hemicellulose, and lignin constituents, alongside the determination of their biomethane potential [3]. Furthermore, this study aims to evaluate the economic viability of incineration, gasification, and Anaerobic Digestion (AD) technologies in Oman. By examining the labile carbon pool and the impact of slow pyrolysis temperatures on labile biochar carbon formation derived from date and oil palm empty fruit bunches and their constituents, this research also proposes mechanisms to enhance methane yield before utilizing the recalcitrant residue for soil enrichment post-anaerobic digestion. This holistic approach is anticipated to provide greater efficacy than direct biochar application in soil, as it facilitates carbon sequestration, thereby mitigating greenhouse gas emissions from labile carbon components [4]. Additionally, investigating the correlation between pyrolysis conditions and the labile carbon pool in methane production enhances the mechanistic comprehension of biochar degradation during anaerobic digestion, thereby contributing to the conversion of organic fractions of date palm waste and municipal solid waste into renewable energy sources. This initiative addresses significant challenges, including tool availability, data scarcity, and the management of novel materials. Its potential environmental and societal impacts encompass advancements in waste management, renewable energy generation, and agricultural sustainability, all of which align with the United Nations Sustainable Development Goals (UNSDG) [5]. This study provides a comprehensive, multidisciplinary exploration of the extraction of lignocellulosic components from DPEFB and their subsequent conversion into biochar for environmental applications. It integrates biomass valorization, biochar engineering, and wastewater biochar collaboration analysis, offering valuable insights for researchers, policymakers, and industries.

The work supports global priorities on circular economy, renewable bioresources, and sustainable waste management, making the findings relevant to environmental science, chemical engineering, and bioenergy. Accurate experimental characterization, using XRD, TGA, FTIR, Raman spectroscopy, and NMR spectroscopy, ensures scientific integrity and reproducibility. The outcomes also specify rational guidelines for enhancing particle size, extraction efficiency, and thermal conversion conditions, which are crucial for scaling up biochar production. The study's real-world relevance, particularly for Oman and the broader GCC region, further strengthens its importance.

II. METHODOLOGY

A. Sample Collection and Feedstock Pretreatment

Drying, size reduction, and particle size analysis by sieve analysis are shown in Figure 1. The initial experiment involves preparing samples through several sequential procedures, from sample collection to grinding and sieving, to achieve the desired sample size for the subsequent extraction processes. Sieve analysis determines the particle size distribution of both fine and coarse aggregates in samples. Before extraction, specific pre-treatment procedures are implemented, including drying and size reduction, which are crucial for ensuring uniformity and facilitating subsequent processing. Two distinct types of Empty Fruit Bunches (EFB) undergo these pre-treatment steps. The subsequent Soxhlet extraction procedure, intended for cellulose, hemicellulose, and lignin, requires a fine powder, underscoring the importance of achieving an appropriate particle size through grinding and sieving. The detailed systematic sample preparation is presented in Figure 1.

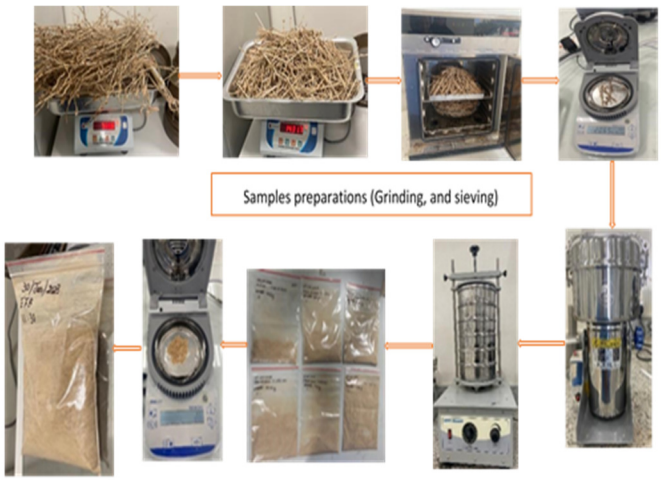

Fig. 1. Flowchart of sample preparation.

B. Removal of Extractives

In this experiment, the standard test procedure outlined in ASTM D1105-96 is employed to produce extractive-free wood from the provided samples. Extractives refer to nonstructural components of wood that are susceptible to removal using natural solvents. These extractives encompass a diverse array of substances, including but not limited to fats, waxes, proteins, phenolics, gums, resins, simple sugars, essential oils, pectins, mucilages, glycosides, saponins, fatty acids, sterols, and flavonoids, all of which are considered undesirable in the context of the experiment. The extraction of extractives is achieved through a three-step process, involving the successive washing of the sample with toluene-ethanol for 4 h, followed by washing with ethanol for 3.7 h, and finally washing with hot water for 3 h. The process is repeated three times, aiming to eliminate extractive components from the wood samples, thereby ensuring their suitability for subsequent analyses and experimentation. Figures 2-4 depict the sequential processes of toluene-ethanol wash, ethanol wash, and hot water wash, respectively.

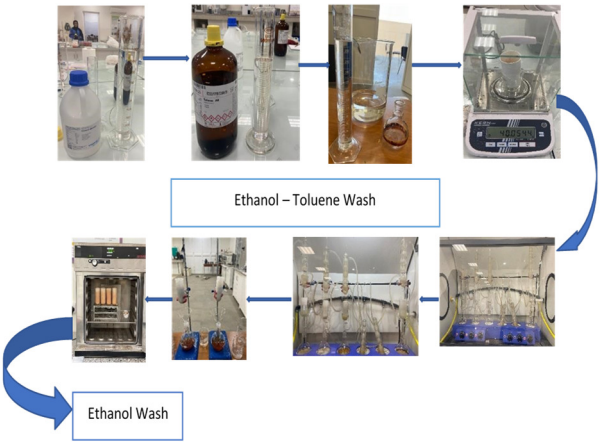

Fig. 2. Flowchart of ethanol-toluene wash.

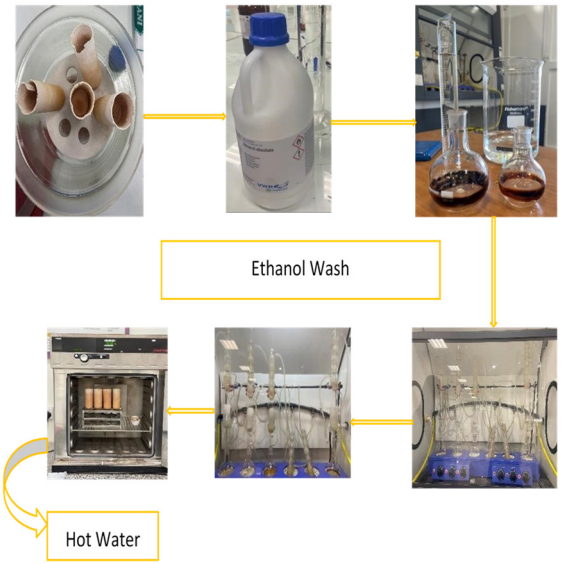

Fig. 3. Flowchart of ethanol wash.

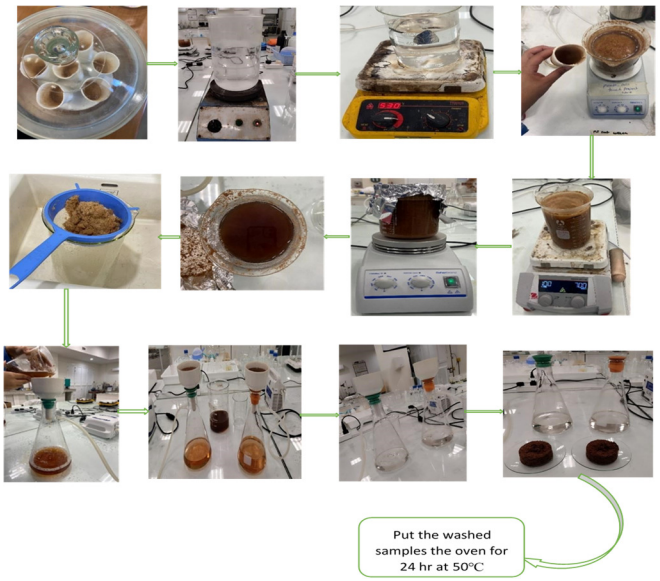

Fig. 4. Flowchart of hotwater wash.

C. Extraction of Cellulose, Hemicellulose, and Lignin

The extraction of cellulose, hemicellulose, and lignin was conducted following established protocols. Cellulose extraction was performed according to the ASTM D1103 protocol, which involves extraction of holocellulose and cellulose with acetic acid, followed by bleaching with NaClO2. The ASTM D1106 protocol was followed for lignin extraction, using 72% H_2SO_4. Additionally, hemicellulose A and B were extracted with KOH, following the methodology outlined in [6]. These standardized procedures ensure consistency and reproducibility in the extraction process, thereby facilitating accurate characterization and analysis of the biomass constituents. Figures 5-7 illustrate the extraction processes of holocellulose, cellulose, and lignin, respectively.

The extraction of holocellulose, cellulose, and lignin separates the main structural constituents of biomass through successive chemical behaviors. These processes help investigators understand material properties and develop value-added products. Their influence lies in facilitating advances in biofuels, biopolymers, and green sustainable materials.

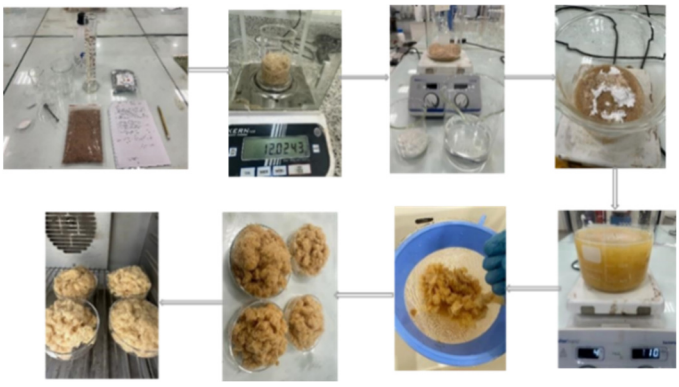

Fig. 5. Extraction of holocellulose.

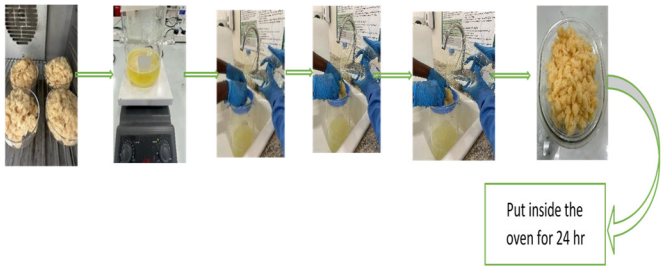

Fig. 6. Extraction of cellulose.

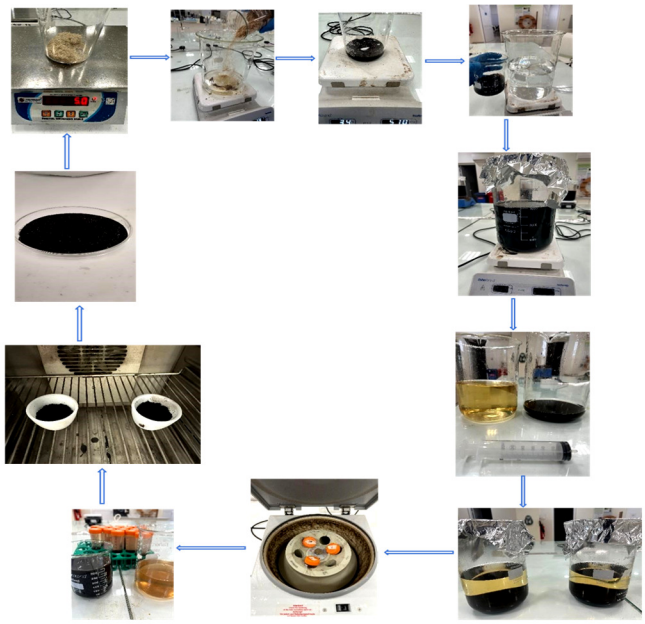

Fig. 7. Extraction of lignin.

D. Carbonization and Characterization of Biomass Components

The DPEFB and its constituent components will be carbonized at 250°C (for 3 h), 400°C (for 1 h), and 750°C (for 0.5 h) using a furnace and silica crucibles. These selected temperatures correspond to the approximate carbonization temperatures of hemicellulose, cellulose, and lignin, respectively. Furthermore, the impact of varying holding times (0.5, 1, and 3 h) was investigated at a constant temperature of 400°C. After carbonization, the resulting materials will undergo characterization using various analytical techniques. Proximate analysis will be conducted to determine the fundamental properties of the carbonized material. Additionally, XRD, TGA, FTIR, Raman spectroscopy, and NMR spectroscopy will be employed to elucidate the structural and chemical properties of the carbonized material. These analytical methods provide insights into the composition, crystallinity, thermal stability, functional groups, and molecular structures of the carbonized biomass constituents. Biochar was incorporated into the AD process and mixed with anaerobic sludge according to [7]. The experimental procedure involves mixing 2 gm of biochar and 2 gm of sludge with 30 mL of OBED chemicals and maintaining the mixture at 37 °C. Subsequently, the syringe gas percentage is monitored daily throughout the experimental duration.

III. RESULTS AND DISCUSSION

A. Analysis and Discussion of the Results Determine the Size of the Particles

The particle size was measured by sieve analysis using a set stack of sieves ranging from 1mm to 125 µm to determine the ideal particle size of the DPEFB sample. The best particle size range for DPEFB used for extractive removal was found to be 250-500 µm. Table I shows the percentage of samples obtained from various particle sizes.

TABLE I. PARTICLE SIZE

Size and weight of sieve set stack and sample	Weigh of sample only (g) after shaker	Percentage (%)
Sieve set stack (1mm)	0	0
Sieve set stack (500µm)	2.08	1.25
Sieve set stack (250µm)	85.32	51.5
Sieve set stack (125µm)	60.75	36.5
Less than (125µm)	17.85	10.75

B. Analysis and Discussion of the Removal of Extractive Results

Ethanol-Toluene, Ethanol, and hot water, with an overall extraction time of 600 min, for extractive removal, and Two-way ANOVA revealed a statistically significant difference between typical (direct) and alternative (indirect) extraction methods for determining extractives. Figures 8-11 show the extractive yields achieved with various solvents and the corresponding number of siphon cycles required to eliminate impurities [8].

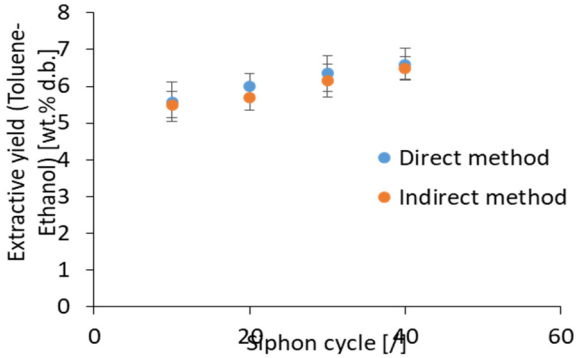

Fig. 8. Extractive yield of Toluene-ethanol and number of siphon cycles.

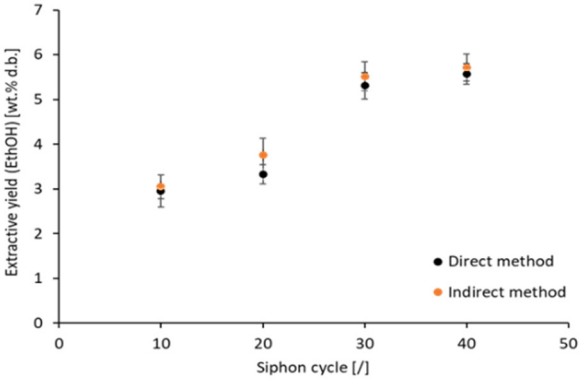

Fig. 9. Extractive yield of ethanol and number of siphon cycles.

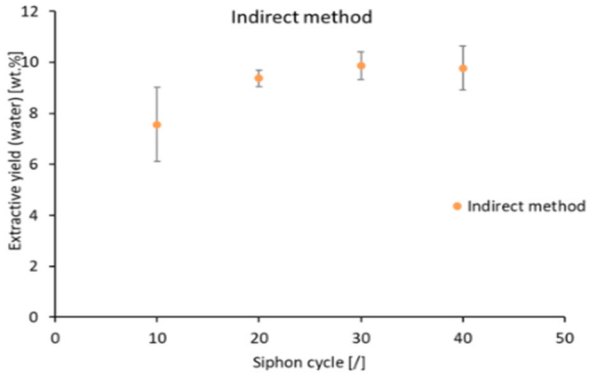

Fig. 10. Extractive yield of hot water and number of siphon cycles.

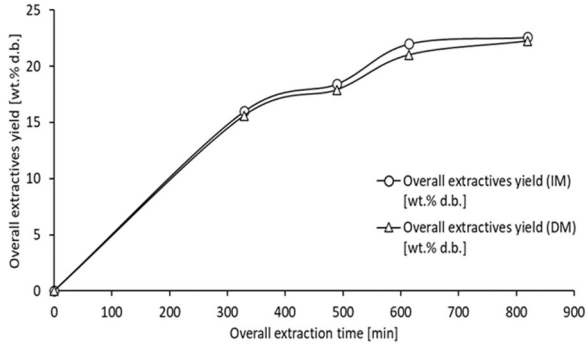

Fig. 11. Relation between overall extractive removal and overall time.

C. Analysis and Discussion of the Extraction Result and Characterization of Cellulose, Hemicellulose, and Lignin

The ash content was determined by TGA according to the Mayoral protocol. The applied protocol yielded the highest extraction rate, with ash content below 2%. This indicates that all elements consist predominantly of aliphatic and labile carbon. Table II displays the cellulose, hemicellulose A, B, and lignin after the extraction process. Figure 12 provides a comparison of the extracted materials with commercial compounds and samples, and Figure 13 shows the % FTIR of cellulose after extraction. Table III demonstrates identical results obtained with a furnace [9]. Table IV presents the ultimate analysis (C, H, and N content) of cellulose, HA, HB, lignin, and the corresponding DPEFB-derived samples after extraction.

TABLE II. CELLULOSE, HEMICELLULOSE A, HEMICELLULOSE B, AND LIGNIN AFTER EXTRACTION

#	Lignocellulosic	Yield (%) (this work)	Other references (%)	Ash (%)
1	Cellulose	87.2	Max.86.9	0.07
2	Lignin	90	83	1.98
3	Hemicellulose A	37	27	2.04
4	Hemicellulose B	49.2	41.8	1.02

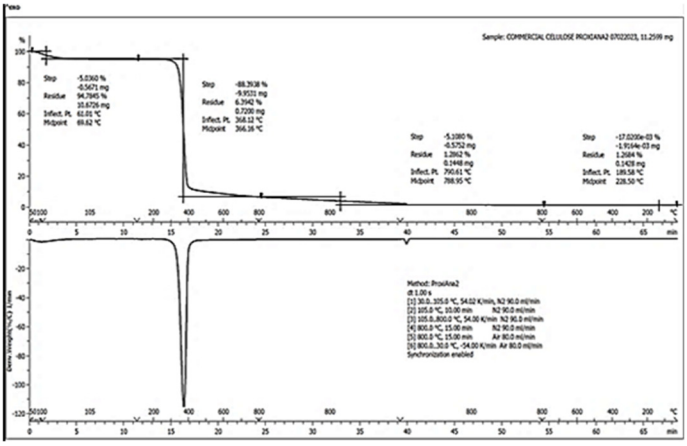

Fig. 12. Comparison of extracted materials.

TABLE III. PROXIMATE ANALYSIS OF CELLULOSE, HA, HB, AND LIGNIN AFTER EXTRACTION

ASTM D1762 (furnace)	Sample	MC [wt.% a.r.]	VM [wt.% d.b.]	AC [wt.% d.b.]	FC [wt.% d.b.]	QC (VM+AC+FC)
	Original DPEFB(R1)	2.51	79.20	4.40	16.40	99.90
	DPEFB.EF.R1	8.60	80.30	3.10	16.60	100.00
	Commercial cellulose R1	4.26	83.30	1.20	15.50	100.00
	Cellulose DPEFB (R1)	6.20	87.90	2.00	10.10	100.00
	Commercial lignin R1	2.11	62.80	0.20	37.00	100.00
	DPEFB- LIGNIN (R1)	24.40	39.10	21.20	39.70	100.00
	Commercial hemicellulose (R1)	13.00	82.00	5.00	13.00	100.00
	DPEFB- hemicellulose-A (R1)	15.80	47.80	39.30	12.90	100.00
	DPEFB- hemicellulose- B (R1)	22.70	59.80	28.10	12.10	100.00
TGA	**Sample**	**MC [wt.% a.r.]**	**VM [wt.% d.b.]**	**AC [wt.% d.b.]**	**FC [wt.% d.b.]**	**QC (VM+AC+FC)**
	Original DPEFB(R1)	6.82	80.70	3.85	15.45	100.00
	DPEFB.EF.R1	5.23	83.56	1.59	14.85	100.00
	Commercial cellulose R1 (cellulose)	3.45	90.63	0.60	8.77	100.00
	Cellulose DPEFB (R1)	4.29	88.07	1.18	10.75	100.00
	Commercial lignin R1	2.17	64.40	2.43	33.17	100.00
	DPEFB- LIGNIN (R1)	16.17	27.76	27.72	44.52	100.00
	Commercial hemicellulose (R1)	9.74	79.57	7.08	13.35	100.00
	DPEFB- hemicellulose-A (R1)	7.32	73.45	18.74	7.81	100.00
	DPEFB- hemicellulose- B (R1)	16.25	70.43	28.73	0.85	100.00

TABLE IV. ULTIMATE ANALYSIS OF CELLULOSE, HA,HB, AND LIGNIN AFTER EXTRACTION

Sample	Carbon (% ± SD)	Hydrogen (% ± SD)	Nitrogen (% ± SD)
DPEFB ORIGINAL	43.48 ±0.07	5.86 ±0.19	0.53 ±0.01
DPEFB EF	45.53 ±0.15	6.30 ±0.01	0.43 ±0.03
CC	42.09 ±0.06	6.51 ±0.01	0.025 ±0.004
DPEFB CELLULOSE	42.12 ±0.21	6.40 ±0.01	0.11±0.01
CL	46.40 ±0.09	4.94 ±0.10	0.04 ±0.03
DPEFB LIGNIN	42.14 ±0.73	5.86 ±0.01	0.01±0.03
HC	40.45 ±0.02	6.63 ±0.11	0.16 ±0.05
HA	26.09 ±0.39	3.83 ±0.10	0.15±0.09
HB	28.12 ±0.42	5.64 ±0.06	0.02 ±0.04

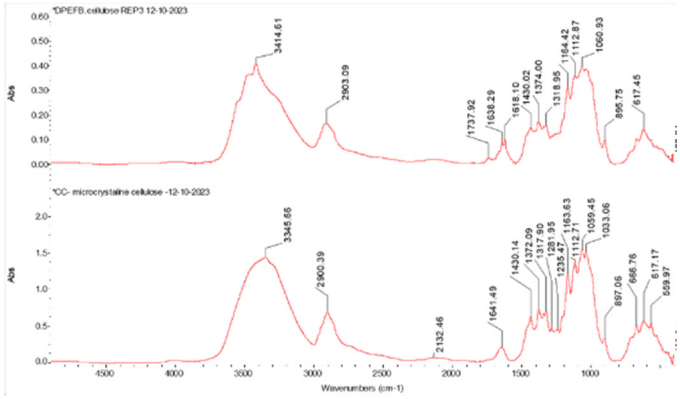

Fig. 13. FTIR of cellulose after extraction.

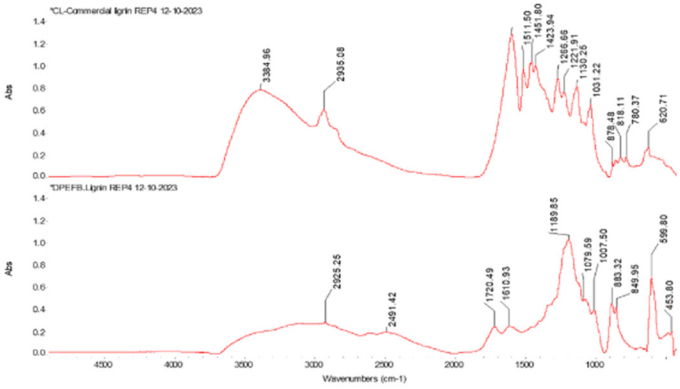

Fig. 14. FTIR of lignin after extraction.

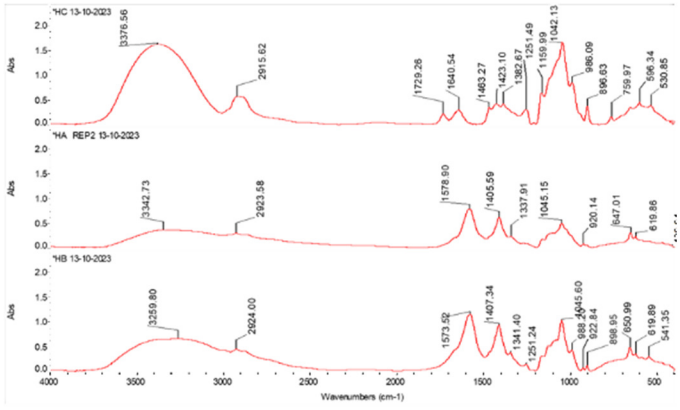

Fig. 15. FTIR of HA and HB after extraction.

Figures 13-15 illustrate a comparison of the extracted materials with commercial compounds and samples, demonstrating a high percentage of agreement, as nitrogen gas and an oven drying process were used before starting the analysis [10].

D. Analysis and Discussion of the Result Production/Results and Characterization of Biochar

Based on the TGA results, the time and temperature conditions required to convert each sample into biochar were determined. This conversion process is achieved by employing a furnace under a nitrogen atmosphere [11]. Table V presents the quantified yield of biochar obtained from individual samples, while Figure 16 visualizes the size and morphology of the biochar particles obtained through the processes of sieving and grinding.

TABLE V. AMOUNT OF BIOCHAR FROM CELLULOSE, HA, HB, AND LIGNIN AFTER EXTRACTION

#	Lignocellulosic material	Temperature (°C)	Time (min)	Amount (g)
1	Cellulose	250	30	4g from 40g
2	Lignin	400	60	9g from 40g
3	Hemicellulose A	750	180	6.4g from 40g
4	Hemicellulose B	742	187	5.8g from 40g

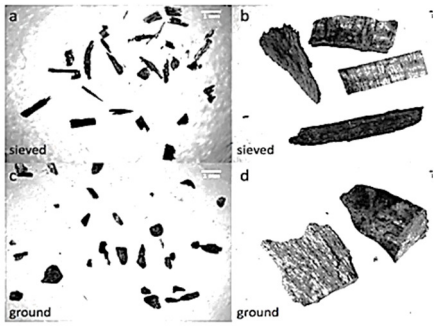

Fig. 16. Size and shapes of biochar obtained from sieving and grinding.

The NMR analysis conducted on the biochar revealed the presence of carbon-13, indicating the presence of both labile and stable carbon within the biochar composition, as depicted in Figure 17.

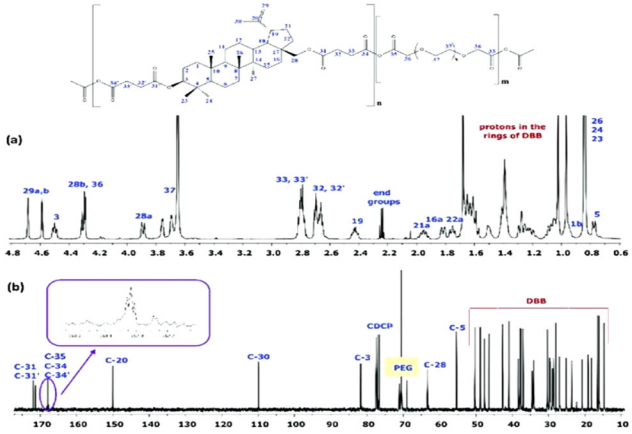

Fig. 17. 13C NMR of biochar.

E. Analysis and Discussion of the Results for Biochar Application

The analysis and discussion of the results for biochar application revealed that the highest methane production was achieved with cellulose, reaching up to 800 ml over 40 days. Figure 18 illustrates the quantity of bio methane gas generated from the lignocellulosic materials over successive days [12].

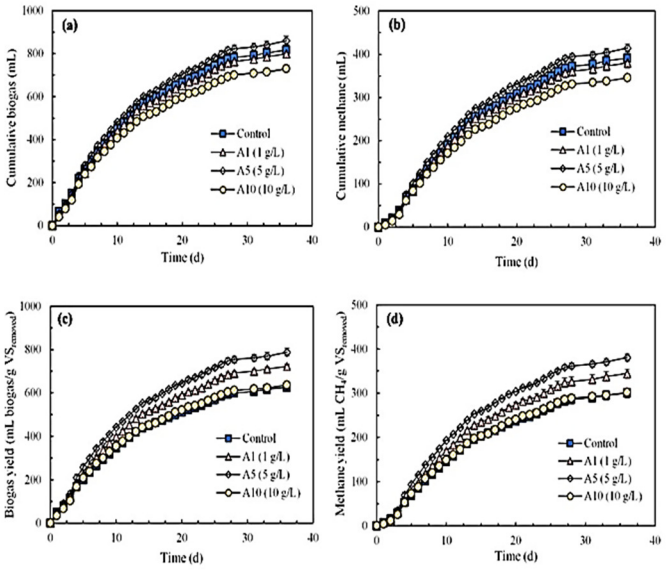

Fig. 18. Methane yields versus time.

IV. CONCLUSIONS

The present research was effectively carried out through strategic integration with the one million palm trees program in Ibri, using the residual biomass produced by date palm farming. It is specifically interesting in the available palm-derived residues, which are normally left out of traditional value-added uses, including wicker and handicraft production. The research will result in better use of agricultural waste in general by making the process a resource recovery process rather than a way of disposal. The proposed structure allows producing treated organic fertilizer while simultaneously recovering clean biomethane to generate localized energy in Ibri. To enhance sustainability, solar energy is also integrated into major operational phases, including lighting systems, cutting and size-reduction devices, evaporation facilities, and drying. This energy-saving strategy will significantly reduce reliance on traditional energy sources and lower the project's environmental impact. Based on an estimated annual availability of about 21,000 tons of agricultural palm waste, the system can generate close to 8,400 tons of processed fertilizer, demonstrating significant potential for large-scale resource optimization. By integrating these strategies, the project aims to maximize resource utilization, enhance sustainability, and encourage the effectiveness of the One Million Palm Trees initiative in Ibri. Moreover, developing a predictive mathematical model to assess the relationship between holding time and biomethane gas production further enhances the project's scientific rigor and operational efficiency.

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