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Anaerobic digestion of dry palms from five cultivars of Algerian date palm (*Phoenix dactylifera* L.) namely *H'mira, Teggaza, Tinacer, Aghamou* and *Takarbouchet:* A new comparative study

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ABSTRACT

The lignocellulosic properties of date palm waste (dry palm) differ significantly from one cultivar to another, which affects the anaerobic digestion (AD) process. This study is believed to be amongst the first to evaluate the influence of date palm cultivars on the biomethane yield in order to offer an annual, continuous and cost-effective biogas production model. In this work, 5 cultivars from date palm waste namely; *H'mira* (H), *Teggaza* (Tg), *Tinacer* (Ti), *Aghamou* (Ag) and *Takarbouchet* (Tk) were evaluated for biogas production. All experiments were performed for 45 days with 5 reactors in triplicate under mesophilic conditions (37 °C). The highest methane yield of 231.87 ml of CH_4/g of Volatile Solid (VS) was obtained with the Ag cultivars with a difference that varied between 37% and 62% depending on the cultivar type. These results indicate that the date palm cultivars massively influence the biomethane yield, it may give an opportunity for researchers to select the most suitable cultivars for methane production and provide opportunities to valorize other cultivars on other beneficial uses, such as adsorption, thermal insulation, or charcoal production etc.

1. Introduction

Most of the world's energy needs are currently met by the non-renewable fossil fuels which are believed to have limited and declining reserves. In addition to the risk of their anticipated depletion, the negative environmental impacts caused by burning fossil fuels such as global warming, climate change and depletion of the ozone layer have become serious threats day by day [1]. Consequently, the growing interest in the production of eco-friendly and renewable fuels has become an important issue for many countries. On the other hand, the global awareness on the risk of the accumulation of the biogenic waste in the landfills without proper recycling is also rising with many policies aimed at turning these wastes into useful energy or green products through the integrated biorefineries. The anaerobic digestion (AD) process, which is one of the most established biofuels production technologies, is an effective method to convert the biogenic waste into useful energy [2]. Biogas which is produced by the AD process is one of the most promising alternatives to fossil fuels due to its versatile use in electricity and heat generation besides the vehicles. Biogas is rich in biomethane (CH_4) and therefore it can be used as a substitute for natural gas in many different applications. Biogas is one of the cheapest and harmless renewable energy sources. The production of biogas can be done from various biogenic wastes, residues and energy crops [3–5]. For instance, food wastes have a high biomethane production potential thanks to their high organic matter contents [6]. Cow dung, municipal sewage sludge, and

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Nomenclature

AD	Anaerobic digestion
Ag	*Aghamou* cultivar
BMP	Biochemical methane potential
C_I%	Percentage of Carbon in inoculum
CMP	Cumulative Methane Production
COD	chemical oxygen demand
C_S%	Percentage of Carbon in substrate
C/N	Carbone/Nitrogen ratio
FTIR	Fourier transform infrared spectroscopy
Gj	Gigajoules
H	*H'mira* cultivar
I_{add} (VS)	The amount of inoculum added (in VS)
MGM	Modified Gompertz Model
N_I%	Percentage of Nitrogen in inoculum
RURESM	Research unit in Renewable energies in Saharan Medium
N_S%	Percentage of Nitrogen in substrate
S_{add} (VS)	The amount of substrate added (in VS)
TA	Total Alkalinity
TC	Total Carbon
Ti	*Tinacer* cultivar
Tg	*Teggaza* cultivar
Tk	*Takarbouchet* cultivar
TKN	Total Kjeldahl Nitrogen
TN	Total Nitrogen
TS	Total Solid
VFA	Volatile Fatty Acids
VS	Volatile Solid

fruit/vegetable waste are also among the popular raw materials for the biogas production. It has been reported that more CH_4 can be produced from food and fruit/vegetable waste compared to animal manure and municipal sewage sludge due to their rich nutrition content and their combinations [7,8].

The AD process is a biological pathway that is grouped into four main phases namely; hydrolysis, acidogenesis, acetogenesis and methanogenesis [9]. Each phase is crucial and acts as a determining factor of the biochemical reactions. Additionally, each phase is dependent on the feedstock combinations [10]. The methanogenesis phase can be accepted to be the rate determination step when the feedstock is easily broken down into small components, otherwise, the hydrolysis phase acts as a limitation step for the biochemical reactions [11]. The time of the hydrolysis phase is generally short for the carbohydrates, proteins and lipids whereas it takes a longer time for the lignocellulosic feedstocks such as the agricultural wastes. The lignocellulosic feedstocks contain cellulose, hemicellulose and lignin. Cellulose and hemicellulose are biodegradable and easily digestible in the AD process; however, lignin-containing materials have a lower biodegradability [12,13].

Agricultural wastes are generally regional and their characteristics and structural composition vary depending on many variables such as climate conditions, soil characteristics, agricultural practices and water quality [14]. According to the climatic conditions of Africa, one of the most common agricultural wastes is the date palm waste. Date trees produce about 40 kg of combustible waste per year, including leaves, bark, sheaths and petiole [15]. Some previous studies aimed at valorizing the date palm waste for energy, catalyst, building materials and the environment applications [16–18]. Makkawi et al. [19] experimentally conducted the fast pyrolysis of three anatomical parts of the date palm (*Phoenix dactylifera* L.) which included leaves, petioles and hollow fruit clusters in a bubbling fluidized bed reactor. The main findings showed that the overall products yield (in a mass percentage) were 38.8% bio-oil (including 10.4% reaction water), 37.2% biochar and 24.0% non-condensable gas obtained at 525 °C. Giwa et al. [20] conducted economic and environmental assessments of date palm waste pyrolysis for biochar production using concentrated solar thermal energy and compared the results with the traditional process (electric heating-based pyrolysis). The study indicated that the concentrated solar energy pyrolysis is more environmentally friendly and more cost-effective than the traditional pyrolysis. The economical aspect indicated that the payback time and gross margin of the described system were found to be approximately 4.4 years and 35.5%, respectively. Moreover, the CO_2 emission of the described system can be reduced by 62% in comparison with a conventional pyrolysis system. Daoud et al. [21] investigated the energy conversion of some agricultural wastes grown in Algeria such as date palm rachis and jujube stones. The study revealed that the energy conversion ratio via pyrolysis pathway can reach 70% when the produced gases are used for the next pyrolysis reaction. Other useful studies on the pyrolysis of date palm waste can be found at [22–24]. Bassyouni et al. [25] studied the simulation of date palm waste gasification using ASPEN HYSYS in which the developed model was able to predict the syngas composition. Ben Hnich et al. [26] evaluated the life-cycle sustainability performance of synthetic diesel and gasoline produced from Tunisian palm waste by gasification method and Fischer-Tropsch process and compared with fossil fuels. It has been found that biofuels from Tunisian palm waste provided a slight increase in carbon footprint compared to their fossil homologues, as well as being renewable. However, when discussing the economic aspect, the situation changes, as the biofuels cost is nearly double the fossil fuels cost (diesel and gasoline) for the year 2020 in Tunisia, where it reached 31 dollars in 2020/Gigajoules (Gj), versus 14.72 and 21.56 dollars/Gj, for diesel and gasoline, respectively (International Petroleum price, 2021). This situation can be partly improved by optimizing the biofuel production system through increasing the plant size and improving the electricity consumption by introducing renewable systems.

Ahmad et al. [27] produced bioethanol by fermentation with *Saccharomyces cerevisiae* strain using date palm waste. The fermentation process resulted in a production of 15% (v/v) ethanol under the optimum condition of an incubation period of 72 h. Moreover, Martis et al. [28] investigated biological and thermochemical ways such as pyrolysis, gasification and fermentation to produce biofuel from date palm waste. Their analyses revealed that fermentation, as a biochemical pathway, had a higher production rate than the thermochemical processes. However, the fermentation process is less economically feasible than pyrolysis and gasification processes. For 1 ton of date palm waste, fermentation required an investment of USD 763, while gasification generated USD 166 and pyrolysis generated a return of USD 768. To balance this difference, it is suggested that the spent hemicellulose and lignin from the fermentation process could be re-used for subsequent fuel production in a thermochemical process.

The AD process is another biological pathway to produce energy from the date palm waste. Subsequently, it is being valued as a renewable process for biogas production. In the literature, few studies related to the AD process of this waste are available. Chandrasekhar et al. [29] experimentally investigated the biochemical methane potential (BMP) of different date palm waste, including dry palm. The higher CH_4 production of 72.60 mL CH_4/g VS/days was obtained from the dry palm compared to the other date palm waste such as Pedicels, Fibrilium, Petiole, Fruit bunch, Spath, Palm, and their mixture. It has been concluded that dry palms provided more CH_4 production because it has less lignin component than the other date palm waste. Consequently, the higher lignin content has an influence on the insoluble fraction of substrates. The same authors also researched influences of acid, alkali and hydrothermal pretreatments on the CH_4 production from date palm

biomass namely Pedicels, Fibrilium, Petiole, Fruit bunch, Spath, Palm, and their mixture. The study revealed that the alkaline pretreatment showed a better performance than other pretreatment methods due to delignification of date biomass. Moreover, the highest CH_4 production of 295.6 mL CH_4/g Total Solid (TS) was recorded with Palm feedstock (dry palm) [30]. Kerrou et al. [31], on the other hand, conducted an experimental study on the production of CH_4 from date palm leaflets by the AD in Morocco. The study concluded that 133 mL CH4/g VS was obtained from date palm leaflets and its biodegradability was reported to be 33%. Moreover, three different kinetic models namely Gompertz, Richards and Logistic to determine the experimental CH_4 production were used. The modified Gompertz model showed the best fit with the experimental data ($R^2 = 0.998$). Al-Juhaimi et al. [32] studied the CH_4 production by the AD from the date palm tree waste. 342.2 mL biogas/g VS was obtained from this waste and a 58% reduction of organic content was observed after the AD process. Moreover, the observed reduction was the combination of cellulose, hemicellulose and soluble organic components, which were recorded as 68.7, 73.4, and 71.9%, respectively whereas lignin contents of the waste were almost constant after the AD process.

The combinations of lignocellulosic feedstocks and their percentages have a strong influence on the AD process. Polysaccharide is the combination of cellulose and hemicellulose in lignocellulosic feedstocks and polysaccharide is convertible into a simple sugar via hydrolyzing [11]. On the other hand, lignin, which is another component of the lignocellulosic feedstocks, negatively affects CH_4 production. The AD process may not be completed when the combinations of feedstocks have a high percentage of lignin [33]. Agricultural wastes such as date palm waste can show differences between the ratios of cellulose, hemicellulose and lignin depending on the species even though they are in the same family and are grown in similar conditions. Despite the several previous studies addressing CH_4 production from date palm waste, as mentioned in the above paragraphs, rare information about the effects of date palm waste (dry palms) from different cultivars on the AD process is available.

This study aims to evaluate the difference in the CH_4 production when the same waste type (dry palms) coming from different cultivars of date palm is used as substrate for the AD process, as well as to give more details on the scientific basis for the future methanation of such wastes. These differences, if not considered, could negatively affect the techno-economic studies carried out by researchers in the biogas production from such wastes. Therefore, the date palms cultivars from which the samples (dry palms) were taken are namely *H'mira, Teggaza, Tinacer, Aghamou, and Takarbouchet*. The samples were collected in the Adrar region (south-west) Algeria and they were used as feedstocks for the AD process in a batch reactor under mesophilic condition. The kinetic modeling was performed with the modified Gompertz model. This would offer an opportunity to provide annual, continuous and cost-effective biogas production model from the selected source of the dry palms. Meanwhile, this study will allow researchers to better perform techno-economic studies (reducing the error margin) and to easily select the appropriate cultivars for biogas production to provide opportunity to valorize other cultivars on other appropriate uses, such as thermal insulation, adsorption or charcoal production, and etc.

2. Materials and methods

2.1. Substrate sampling

In this study, the substrates used for the biogas production are composed of the most dominant date palm cultivars (*Phoenix dactylifera* L.) in the Adrar region, Algeria namely; *H'mira, Teggaza, Tinacer, Aghamou* and *Takarbouchet*. In order to ensure consistent research, the different cultivars were collected from 5 different sites in the Adrar city (Tsabit, Tammaset, Sahla, Tiouririne and Benitamer) and then mixed before being crushed. After mixing, the substrates were first introduced into a large grinder to minimize their particle sizes. This was followed by passing through a microgrinder to further minimize their sizes to fine particles (≤0.5 mm) in order to facilitate their degradation during the AD process.

2.2. Inoculum preparation

The inoculum is taken from a 20-L batch digester at the laboratory of the Research unit in Renewable Energies in Saharan Medium (RURESM) fed with sludge obtained from the lagoon of Adrar city, working in mesophilic conditions (37 °C).

2.3. Anaerobic digestion (AD)

Each digester was fed with 100 ml inoculum which corresponds to 4 g VS and compared to 2 g VS of substrate so that the inoculum/substrate ratio is always fixed at 2/1. The reactors are always maintained at mesophilic conditions (37 °C ± 1 °C) in a heated water bath and monitored by a thermostat. The reactors are stirred manually once a day throughout the experiment. The medium pH is maintained between 7 and 8 due to the alkaline pH of the inoculum.

2.4. Analytical methods

The total solids (TS%), volatile solids (VS%), BMP, Total alkalinity (TA), volatile fatty acids (VFA) and chemical oxygen demand (COD) were measured as prescribed in Ref. [34]. The pH was measured using a pH-meter model (Mettler-Toledo AG, Analytical CH-8603 Schwerzenbach, Switzerland).

The Total Kjeldahl Nitrogen (TKN) and C/N ratio were measured as prescribed by Ref. [35] using Eq (1).

$$C \Big/ N = \frac{VS(\%)/1.76}{TKN(\%)} \tag{1}$$

where VS(%) is volatile solids per cent relative to the TS.

TKN content was determined using Eq. (2) [35].

$$TKN(\%) = \frac{\text{Protein}}{6.25} \tag{2}$$

The total Carbon Nitrogen ratio of the mixture inoculum substrate was calculated using Eq. (3)

$$\frac{\text{Total C}}{\text{Total N}} = \frac{((S_{add}(VS) \times C_S\% + I_{add}(VS) \times C_I\%)/(S_{add}(VS) + I_{add}(VS)))}{((S_{add}(VS) \times N_S\% + I_{add}(VS) \times N_I\%)/(S_{add}(VS) + I_{add}(VS)))} \tag{3}$$

where $S_{add}(VS)$ and $I_{add}(VS)$ are The amount of substrate and inoculum added (in VS) respectively. $C_S\%$ and $C_I\%$ are the percentage of Carbon in substrate and inoculum respectively. $N_S\%$ and $N_I\%$ are the percentage of Nitrogen in substrate and inoculum respectively.

Methane yield is usually expressed as Eq (4) as reported by Refs. [36, 37].

$$\text{Methane } (CH_4) \text{ yield} = \frac{VCH_4}{mVS_i} \left(\frac{ml}{gVs}\right) \tag{4}$$

where VCH_4 and mVS_i are CH_4 volume and mass of the initial volatile solid, respectively.

To determine the functional group composition of date palm waste biomass, Fourier transform infrared spectroscopy (FTIR) (Agilent technologies model Cary 660) was used. The powdered samples were dried to avoid moisture and placed on a diamond (FTIR) at room temperature for recording transmittance spectra. The spectra have been recorded at 4 cm^{-1} spectral resolution covering a wavenumber range of 400–4000 cm^{-1}.

3. Results and discussion

3.1. Substrate characterization

3.1.1. TS and VS

The results of the TS and VS content and the VS/TS of the studied substrates before the AD process is represented in Table 1.

The characteristic results of the dry leaflets from date palm (*Phoenix dactylifera* L.) of cultivars *H'mira, Teggaza, Tinacer, Aghamou and Takarbouchet* which are represented in Table 1 show that the VS rates in substrates ''*H'mira, Teggaza, Tinacer, Aghamou, Takarbouchet* are estimated at 85.71%, 83.96%, 86.87%, 89.60%, 87.30% respectively. These results are close to those obtained by Djaafri et al. [38] who studied the dry leaflets of Algerian date palm (*Phoenix dactylifera* L.) where the VS of the *H'mira* cultivar was 86.48%. The same findings have been reported by other researchers [31,35].

The substrates studied are characterized by the low moisture content which is estimated between 4.83% and 8.86% because they are generally dry palms (dead). The results of the VS/TS ratio are estimated between 89.99 (lowest) and 98.31 (highest). These results show that the organic matter is predominant over the solid components, which is a good indication for the CH_4 production and indicates that these substrates are highly biodegradable [39].

3.1.2. C/N ratio

The carbon and nitrogen contents are presented in Table 2. These contents were calculated according to **Eq (1)** and **(2)**. The C/N ratios of all mixtures (samples + inoculum) were in the range of the optimum level for methanogenesis phases which is reported as between 20 and 30 [9]. No correlation was observed between C/N and the cumulative methane production (CMP); however, the highest CMP yield was obtained from the highest C/N sample, *Aghamou*.

3.2. Substrate's chemical structure

To identify the substrate chemical structure, the FTIR analysis was used. This technique allows to determine the functional groups present in a molecule. This method measures the decrease in radiation intensity through the sample as the wavelength decreases [40].

To identify the different functional groups as well as the different intensities by FTIR technique, Fig. 1 (a) and (b) identifies in detail the chemical changes between the five cultivars. This technique has been adopted in several previous studies such as Akhtar et al. [41], Abid et al. [42] and Abdullah et al. [43].

According to Fig. 1 (a) and (b), the substrate transmittance before the AD process (20–90%) is lower than that after the AD process (55–97%). This phenomenon is explained by the substrate's degradation during the AD process by their decrease in the absorbance and increase in transmittance. Among the 5 cultivars, the *Aghamou* cultivar has the highest transmittance before the AD process (between 70 and 90%). The difference is very noticeable between *Aghamou* and the other 4 cultivars that have low transmittance (between 20 and 83%). The lowest transmittance is recorded with the Tk cultivar followed by Ti, H and Tg respectively. This difference is due to the fragility of the *Aghamou* cultivar and the rigidity of the other cultivars. After 43 days of the AD process, the transmittance of the 5 cultivars became higher with no difference between the *Aghamou* cultivar and the other 4 cultivars. This is explained by the effect of the AD process on the degradation of the studied substrates. Likewise, it was noticed that the succession of peaks according to the percentage of transmittance before the AD process is different from that after the AD process. The Ag cultivar has always the highest transmittance, while the position of the other 4 cultivars has been changed to become higher and closer to the Ag cultivar with the following succession: H, Tk, Tg, Ti from highest to lowest transmittance.

Table 1
Rate of the TS, VS and the VS/TS before the AD process.

Substrate	TS%	VS%	VS/TS
H'mira	95.17	85.71	90.05
Teggaza	93.29	83.96	89.99
Tinacer	93.74	86.87	92.67
Aghamou	91.14	89.60	98.31
Takarbouchet	94.64	87.30	92.24
[31]	92.25	86.08	93.31
[35]	96.37	92.89	96.38
[38]	95.10	86.48	90.93

Table 2
C/N ratio.

Substrate	C%	N%	Substrate	TC%	TN %	C/N
Inoculum	30.11	2.23	Inoculum	30.11	2.23	13.5
H'mira	49.59	1.05	*H'mira* + Inoculum	36.6	1.65	22.18
Teggaza	48.86	0.36	*Teggaza* + Inoculum	36.36	1.67	21.73
Tinacer	50.53	0.22	*Tinacer* + Inoculum	36.92	1.57	23.54
Aghamou	52.06	0.15	*Aghamou* + Inoculum	37.43	1.57	23.86
Takarbouchet	50.5	0.56	*Takarbouchet* + Inoculum	36.91	1.63	22.69

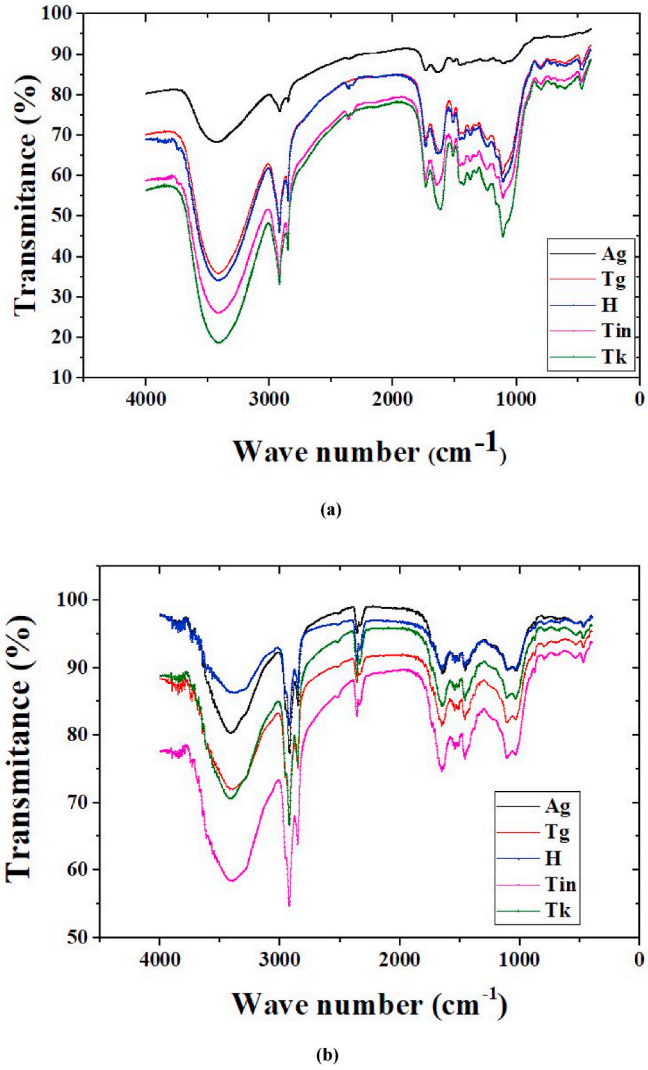


Fig. 1. FTIR spectra before **(a)** (initial) and after digestion **(b)** (final) of cultivars (H, Tg, Ti, Ag, Tk).

This indicates that the substrates with high transmittance are the most exposed to the degradation.

As per Fig. 1, the changes affecting the FTIR spectra of lignocellulosic substrates are localized in 2 important regions, that of the region between 4000 and 1500 cm^{-1} and that of the fingerprint region in the range between 1500 and 400 cm^{-1}.

3.2.1. FTIR spectra interpretation of the region between 4000 and 1500 cm^{-1}

Spectrometric peaks near to 3400 cm^{-1} were found in all spectra (all 5 cultivars) with a broad band. This band represents the hydroxyl group (O–H). It is found in the phenolic and aliphatic structures of the lignin as well as in the structure of cellulose and hemicellulose [44,45]. The width and intensity of the bands vary from one cultivar to another. The *Aghamou* cultivar has the least intense band than the others, which explains the low content of lignin and cellulose and hemicellulose. The same results were found at the end of the AD process.

Looking at the peaks recorded at 2920 and 2850 cm^{-1}, previous studies on the date palm seeds, demonstrate that the bands observed at 2924 and 2855 cm^{-1} are attributed to the asymmetric and symmetric elongation of the C–H bands in the methyl and methylene groups, respectively. These absorption bands generally include contributions from cellulose, hemicellulose and lignin [46]. The intensities of all the substrate peaks are large except for the peak of the Ag cultivar which has less intensity than the others. This is due to its low lignin and hemicellulose contents compared to the high content of these two elements in the other 4 cultivars. The same results were obtained at the end of the AD process.

The peaks recorded at 1700 and 1630 cm^{-1} can be attributed to the carbonyl group of the hemicelluloses corresponding to the bond C═O [47]. As per the same observation with the previous bands, the intensities of all the peaks of the substrates are different. The Ag cultivar has a lower intensity than the others which explains the low hemicellulose content. The Tk cultivar has the highest intensity than the other cultivars. This is due to the high content of hemicelluloses. The other three remaining cultivars (H, Tg, Ti) have an intermediate intensity. At the end of the AD process, the intensity of the peaks for the cultivars Ag and H are less than the other three cultivars. This is explained by the effect of the AD which degrades this element (hemicelluloses). The peaks in the 1522 cm^{-1} region can be attributed to the C═C stretching of the aromatic vibration of lignin [48]. The 4 cultivars (Tg, H, Ti, Tk) have low intensity which explains the low lignin content. In contrast, this peak is almost absent for the Ag cultivar. This shows the negligible content of lignin in this cultivar. The same is observed in the peaks at the end of the AD process. This absence is explained by the removal of lignin during the AD process.

3.2.2. FTIR spectra interpretation of the fingerprint region (1500-400 cm^{-1})

The peaks at 1445, 1430 and 1360 cm^{-1} attribute to stretching of the aromatic C–H vibration of lignin [41]. These peaks have low intensities and remain as such at the beginning and end of the AD process. The low intensity in the 4 cultivars (Tg, H, Ti, Tk) is explained by the low lignin content. In contrast, this peak is absent for the Ag cultivar. This is due to the insignificant lignin content in this cultivar. The peaks at 1090,1100 and 1233 cm^{-1} correspond to C–O cellulose and hemicellulose [46]. All 4 cultivars have high intensity for the 1090 cm-1 peak and low intensity for the 1100 and 1233 cm^{-1} peaks at the beginning of the AD process. At the end of the AD process, the 1090 cm^{-1} peak became weaker and shifted to 1100 cm^{-1}, while the other two peaks 1100 and 1233 cm^{-1} disappeared altogether, which can be explained by the structural change of cellulose and hemicellulose at the end of digestion.

Concerning the Ag cultivar, the peaks at 1090, 1100 and 1233 cm^{-1} are absent at the beginning and at the end of the AD process which implies the absence of amorphous cellulose and the low content of hemicellulose. The absence of the H cultivar peak is due to the partial de-crystallization of cellulose.

Das et al. [49], attribute the 800 cm^{-1} peaks to C–H deformation, which is a property of the β-glycosidic bond of cellulose. At the beginning of the AD process, all the 4 cultivars mark low intensity which explains the low crystalline cellulose content. The absence of these peaks at the end of the AD process is justified by the de-crystallization of cellulose.

3.3. Operating parameters at the beginning and end of anaerobic digestion

3.3.1. pH evolution

The hydrogen potential is a very important parameter for the AD process. This is because the methanogenic organisms are very sensitive to the pH variation. The optimal pH value for a good AD process is close to neutrality with a pH between 6.5 and 7.5 [50]. Whereas some other researchers indicated that the ideal pH range could be between 6.5 and 8.78 [51].

Fig. 2 shows that the initial pH values (8.54–8.75) are moderately higher compared to the final pH values (8.13–8.42). The increase in the initial pH is due to the fact that the amount of inoculum is too high compared to the substrate amount, so that the buffer effect dominates, bringing the pH to higher levels. This can be explained by the degradation of the proteins contained in the inoculum which causes the formation of ammonia responsible for the pH increase as reported in several works [52,53]. Despite the inhibitory effect of ammoniacal nitrogen responsible for the pH increase, the values of VFA/TA ratio (Fig. 6) below 0.2 are good signs that the digesters are functioning well. Researchers reported that it is better to adjust the pH using an acid or base to avoid digester instability, but when the VFA/TA values are below 0.4, no pH adjustment is needed [51]. A recent study by Palakodeti et al. [54] indicated that the increase in pH values from 8, 8.5 to 9.5 showed no effect on digester stability. The slight decrease in the pH value recorded at the end of the AD process for the 5 cultivars involves the degradation of the organic matter and the formation of VFA. Xiaoshan et al. [55] indicated that the CH_4 production is always accompanied by the formation of VFA and ammonia simultaneously.

3.3.2. COD biodegradability

COD represents the amount of oxygen required to oxidize the organic and inorganic materials in the digesting solution [56]. Fig. 3 shows the COD at the beginning (initial) and at the end (final) of the AD process for the 5 cultivars studied.

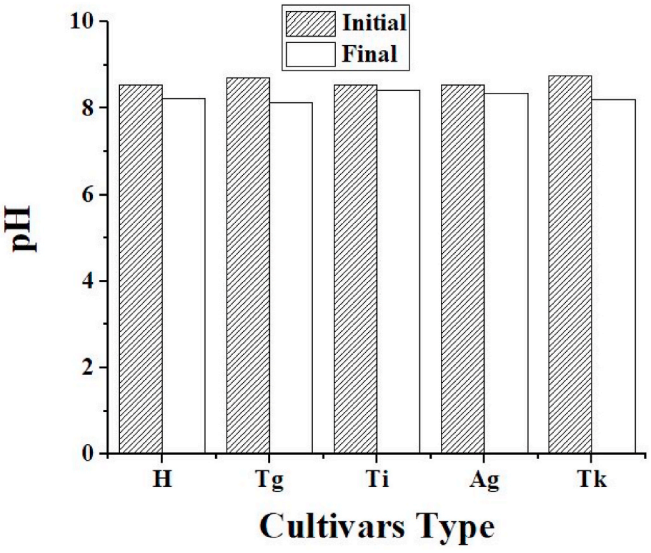


Fig. 2. Initial and final pH at the beginning and end of digestion for the five cultivars.

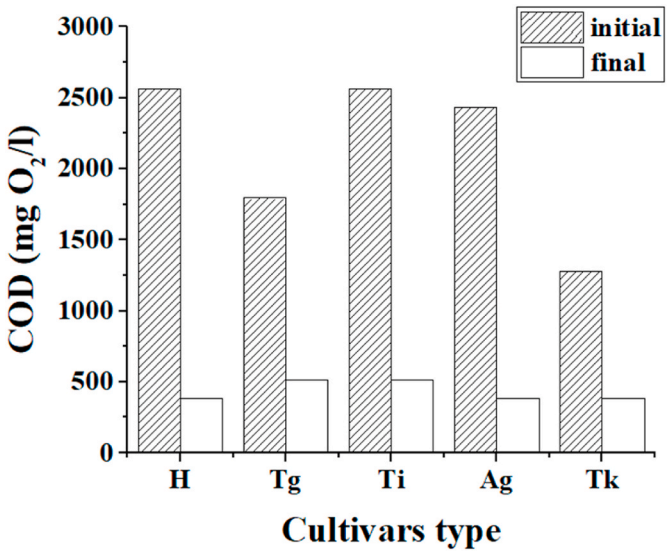


Fig. 3. Initial and final COD at the beginning and end of digestion for the 5 cultivars.

A significant increase in COD at the beginning of the AD process is due to the high substrate degradation due to the fine particle size as well as the large number of microorganisms that easily degrade these substrates. The same findings were reported by Kaidi et al. [57]. The decrease of the final COD is explained by the transformation of the latter into biogas. The same results were reported by Takdastan et al. [58]. Alvarez et al. [59] explain that the decrease in the COD is caused by the majority substrate degradation and the latter's bioconversion into biogas which is well evident by the volume of the produced biogas. The biodegradability rates for the 5 cultivars are (84.99%, 71.42%, 80%, 84.42%, 69.99%) for H, Tg, Ti, Ag and Tk respectively. The 3 cultivars of H, Ag and Ti record a high biodegradability rate which exceeds 80%. On the other hand, Tg and Tk record a biodegradability inferior to the first one (<70%) due to the rigid structure (difficult to biodegrade) of these two cultivars.

3.3.3. VFA

The VFA concentration is one of the most important parameters during the 1st stages of the AD process.

From Fig. 4, the initial VFA values are (0.14, 0.149, 0.16, 0.18, 0.13) and the final values are (0.096, 0.108, 0.108, 0.096, 0.12) for the 5 cultivars (H, Tg, Ti, Ag and Tk) respectively. The high VFA values at the beginning of the AD process are explained by the high VFA production resulting from the presence of the easily biodegradable substrates available initially for the microorganisms. The same remark was reported by Kalloum et al. [60]. The Ag cultivar recorded the highest rate. These results confirm the results of FTIR which shows that the palms of the Ag cultivar are the most fragile and easiest biodegradable cultivar. At the end of the AD process, the VFA are reduced due to their consumption and transformation into acetate and methane. This operation is part of the balance between the different acetogenic and methanogenic bacteria and the other bacteria in the reaction medium [61].

3.3.4. TA

Fig. 5 represents the values of the TA at the beginning and at the end of the AD process for the 5 cultivars studied. The decrease in the TA values is recorded at the beginning of the AD process in parallel with the VFA production (hydrolysis and acidogenic phase). Despite the high pH values (>8), the TA is still low due to some VFA production that does not affect the pH values. Then a sharp increase in the TA values is recorded at the end of the AD process. This increase indicates the VFA consumption. The same results were reported by Tahri [62].

3.3.5. VFA/TA ratio

The amount of the formed VFA in relation to the reaction medium buffering capacity is determined by measuring the VFA/TA ratio. Several researchers reported that the optimal VFA/TA ratio is less than 0.4. Higher values indicate the instability of the AD process [63].

Fig. 6 shows the values of the VFA/TA ratio at the beginning and end of the AD process for the 5 cultivars. These results indicate that the initial and final values of these ratios are less than 0.15 for all reactors. These values are very good and reflect the good functioning of the AD process according to the work of Kalloum et al. [60]. Additionally, the best VFA/TA ratio is recorded for the Ag cultivar in accordance with the previous results (FTIR, COD and VFA).

3.4. Biochemical methane potential (BMP) and kinetics study

Fig. 7 presents the experimental and expected CH_4 yield produced from the 5 cultivars versus time. A significant and continuous CH_4

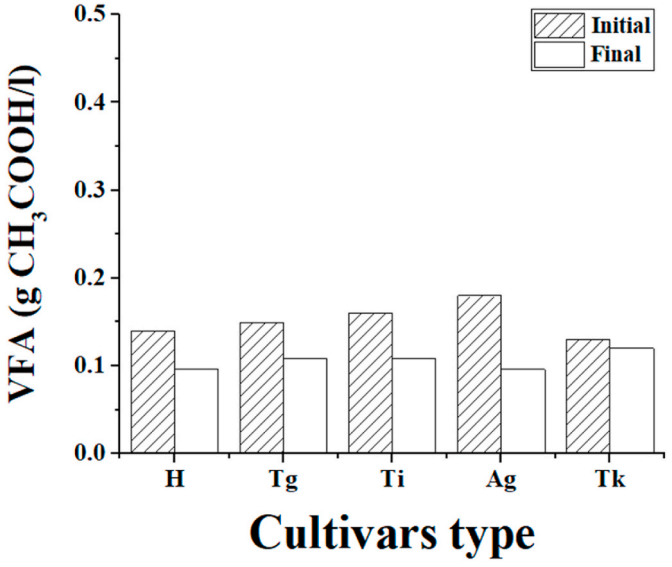


Fig. 4. Initial and final VFA at the beginning and end of digestion for the 5 cultivars.

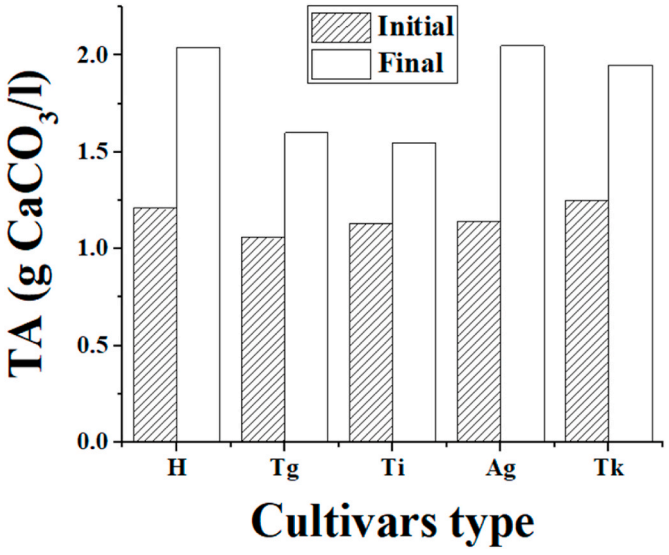


Fig. 5. Initial and final TA at the beginning and end of digestion for the 5 cultivars.

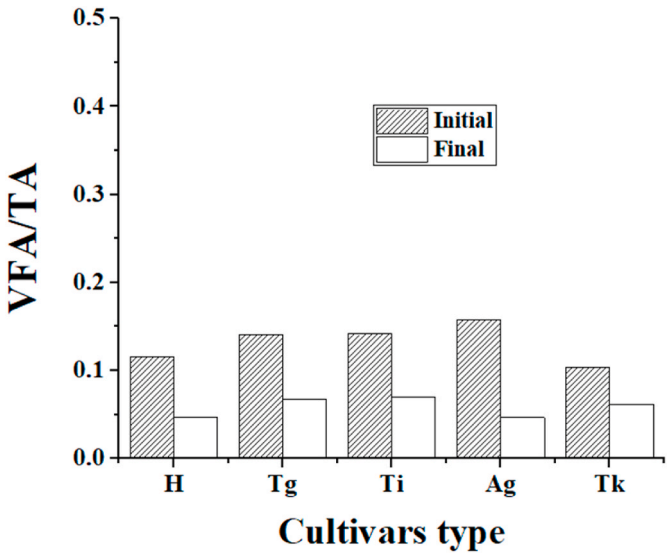


Fig. 6. Initial and final VFA/TA at the beginning and end of digestion.

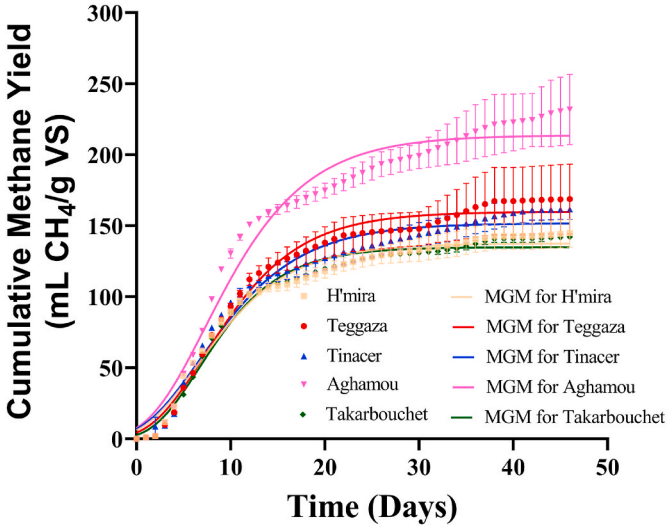


Fig. 7. Experimental and expected methane yield produced from dry palms of the 5 cultivars, *MGM is the modified Gompertz Model, **Error bars indicate standard deviation of triple measurements.

production is recorded from the 1st day until the 9th day for the 4 cultivars H, Tg, Ti and Tk with values that range between 100 and 112 ml of CH_4/g VS. During the same period, the Ag cultivar recorded the highest production than the other 4 cultivars with a value that reached 150 ml of CH_4/g VS. As discussed earlier in this paper, the initial high accumulated CH_4 production for the 5 cultivars is attributed to their easily biodegradable natures [57,64]. The most remarkable difference between the 4 cultivars and that of Ag is due to the fragile structure of the latter as indicated by the results of the FTIR which facilitates their biodegradability [29,30].

Starting from the 10th day, the accumulated production of CH_4 started to decrease and almost showed a stable pattern till the end of the AD process. For the 4 cultivars (H, Tg, Ti and Tk), the amount produced during this period is estimated between 40 and 55 ml of CH_4/g VS. This decrease is due to the VFA consumption besides the existence of the inhibitors that prevent the microbial activity and consequently the degradation of substrates becomes difficult [29,30]. Regarding the Ag cultivar, the amount produced during this period is the highest compared to that produced by the other 4 cultivars. It is estimated to be $\approx$ 80 ml of CH_4/g VS. This variation is reflected by the late degradation of the substrate that is difficult to biodegrade due to its lignocellulosic nature [31,35].

The final CH_4 yield is estimated to be (144.68, 168.75, 161.42, 231.87 and 142.04 ml CH_4/g VS) for the 5 cultivars (H, Tg, Ti, Ag and Tk) respectively.

In the kinetical study, the modified Gompertz model (MGM) has been chosen because it is a widely accepted model and takes into account the bacteria growth and their productions [65–67]. The non-linear data, which was obtained from the CMP, was further processed using Eq. (5).

$$y(t) = a \times epx\left(- exp\left[\frac{b \times e}{a}(c - t) + 1\right]\right) \quad (5)$$

where (y) states the CMP (mL CH_4/g VS), (a) presents the estimation of methane production (mL CH_4/g VS), (b) refers to the maximum CH_4 production rate (mL CH_4/g VS/day), (c) is the lag phase (day), (t) states the time (day) and (e) is Euler's number (~2.718) [65,68]. *a*, *b* and *c*, are the important parameters affecting the characteristics of methane production [69].

The CH_4 production during the AD process showed a "S" shape. This is because a low production rate was observed at the beginning stage followed by a rapid increase. This finding is similar to that reported by Ref. [66]. The model parameters are given in Table 3. The best fit between the experimental data and model was obtained from *Takarbouchet* in which the R^2 was (0.98). This value is similar to the result obtained by Ref. [66] for vegetable crop residues. The lowest R^2 obtained is 0.85 for *Teggaza* where the weakest fit between experimental and model data has been observed.

The CMP results for the 5 cultivars *H'mira, Teggaza, Tinacer, Aghamou,* and *Takarbouchet* were recorded to be 137.5, 159.8, 151.7, 213.7, and 134.9 mL CH_4/g VS, respectively. According to the model, the highest daily CH_4 production rate is obtained from *Aghamou*. This result is similar to that obtained from the experiments. The same remark is noted for *Takarbouchet* whose daily CH_4 production is the lowest according to the model and the experiment.

The technical digestion time (T_{80}) is the time required to reach 80% of total gas production. This parameter can be used to indicate the biodegradability of the substrate [70]. The values of T_{80} obtained in this study are shown in Table 3, the results demonstrated that the T_{80} values varied from 14 days for *Takarboucht* to 20 days for *Aghamou*, these values are lower than those obtained by Ref. [71] and higher than the result obtained by Ref. [66] for different substrate. The values of T_{80} indicate that the biodegradability of *Takarbouchet* is higher than *Aghamou*, it can be noticed that *H'mira* and *Teggaza* show similar T_{80} values. These results confirm that the T_{80} strongly depends on the type of substrate as mentioned by Refs. [68,72].

4. Conclusion

The present work discusses the anaerobic digestion (AD) potential of dry palms from the most dominant 5 cultivars of date palms (*Phoenix dactylifera* L.) in the Adrar region, Algeria namely; *H'mira*, *Teggaza, Tinacer, Aghamou* and *Takarbouchet*.

Table 3
The kinetic parameters based on modified Gompertz Model (MGM).

Parameters	*H'mira*	*Teggaza*	*Tinacer*	*Aghamou*	*Takarbouchet*
a	137.5	159.8	151.7	213.7	134.9
b	9.11	10.42	9.33	12.95	10.19
c	0.80	1.60	0.76	1.24	1.77
R^2	0.9364	0.8506	0.9323	0.9517	0.9822
T_{80}	16	16	18	20	14

*T_{80} indicates time required in days to reach 80% of total cumulative methane yield.
*MGM indicates modified Gompertz Model.

The experimental work lasts for 45 days. The analyses of the substrates showed that they have a high organic matter content with 85.71%, 83.96%, 86.87%, 89.60%, 87.30% for the cultivars (H, Tg, Ti, Ag, Tk) respectively. Moreover, the results of the FTIR analyses showed the rigidity and fragility of the studied substrates. The Ag cultivar had the highest transmittance which indicates their fragility translated by their low absorbance compared to the other 4 cultivars.

The control parameters (pH, COD, VFA and VFA/TA) showed a good kinetics of AD process (with VFA/TA less than 0.2) for all substrates. The Ag cultivar had the best biodegradability characteristics. The results of the accumulative yield of methane (CMP) further confirmed these findings. The Ag cultivar had the highest yield (231.87 ml of CH_4/g of VS) while the other 4 cultivars (Tg, Ti, H, Tk) had a yield of (168.75, 161.42, 144.68, 142.04 ml of CH_4/g of VS respectively.

This study concludes that the cultivar type of the date palms influences the CH_4 yield due to their different characteristics. This could help the scientists to carry out a real techno-economic study (minimizing errors) and eventually help decision makers to provide a model for annual, continuous and economically efficient production of biogas from the selected source of dry palms. Simultaneously, the other cultivars can be directed to other uses.

5. Future recommendations

As a future perspective, it is recommended to conduct another study aimed at evaluating the biogas production capacity from the other waste types that remain in each variety (petiole, fruit bunch, pedicel, spathe and fibrilium), followed by another study aimed to evaluate the capacity of biogas production from the mixture of these wastes and finally a study aimed at evaluating the biogas production capacity from the mixture of wastes of all dominant varieties. These studies will allow to exploit well the numerous and renewable phoenicicole biomass in the Adrar region (south west of Algeria) for energetic and environmental purposes. The objective is to carry out a complete techno-economic study and to evaluate their economic feasibility. This study helps the decision makers to properly exploit this abundant and renewable biomass.

Credit author statement

Mohammed Djaafri: Conceptualization, Methodology, Validation, Formal analysis, Investigation, Resources, Writing – original draft, Visualization. Aicha Drissi: Conceptualization, Investigation, Resources, Writing – original draft. Sabrina Mehdaouib: Conceptualization, Investigation, Resources, Writing – original draft. Slimane Kalloum: Validation, Writing – review & editing, Supervision, Project administration, Funding acquisition. M.R. Atelge: Methodology, Formal analysis, Data curation, Writing – review & editing, Visualization. Mostefa Khelafi: Methodology, Validation, Project administration, Funding acquisition. Kamel Kaidi: Investigation, Resources. Fethya Salem: Investigation, Resources. Ahmed Tahri: Investigation, Resources. A.E. Atabani: Validation, Writing – review & editing, Visualization, Supervision. Libor Štěpanec: Writing – review & editing, Visualization.

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.

Data availability

Data will be made available on request.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.energy.2023.126774.

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