Processing of undervalued dates biomass from common cultivar (Phoenix dactylifera L.) for sequential production of soluble sugars syrup and biogas

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Processing of undervalued dates biomass from common cultivar (*Phoenix dactylifera L.*) for sequential production of soluble sugars syrup and biogas

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Abstract

Date production is usually associated to a considerable loss either in common cultivars or in fruit picking and storage stages. This discarded biomass is not very well valued up to now especially in bioenergy production. The Tunisian second-grade cultivar 'Kenta' was biochemically characterized in the present study. 'Kenta' discarded flesh is rich in soluble sugars (79.5 %VS ± 0.8%VS) and fibers (7.4 %± 0.5%VS). The crude fibers were recovered after soluble sugars extraction. The biochemical composition analysis showed that this by-product contains mainly carbohydrates (33.2 % VS± 0.7%VS) and proteins (8.8% VS ± 0.1% VS) making it a suitable feedstock for biogas production. A biorefinery concept was therefore developed based on soluble sugars (date-syrup) aqueous extraction and biogas production via anaerobic digestion of the residual fibers. The proposed concept showed interesting results since it permitted the co-production of date syrup, as high-added value product, with 0.6 g sugars/gVS and biogas with maximum methane yield of 225 mL CH_4/gVS fibers. This study presents a proof of a sustainable processing approach allowing an almost bioconversion of undervalued secondary date variety and integrates the concept of circular bio-economy.

Keywords: Common date, biorefinery, crude fibers extract, biogas, date-syrup

Statements and Declarations

Conflict of interest: The authors declare no competing interests.

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1. Introduction

Dates are among the oldest fruits in the Middle East and the North Africa. In fact, date palms (*Phoenix dactylifera L*.) have been cultivated since old-time and represent an important agriculture crop in the arid and semi-arid regions (Lajnef et al., 2021, Awad et al., 2021). The world production of dates has increased from 7 428 939 tons in 2014 to 9 075 446 tons in 2019 (FAOSTAT, 2019). About 2000 date cultivars are known however less of them are valued for their performance and their fruit quality. In Tunisia, the average annual production of dates has also improved significantly from 199 000 tons in 2014 to 288 700 tons in 2019 (FAOSTAT, 2019). Unfortunately, this progress in production is associated with a considerable loss either in secondary (common) variety dates (about 30% of Tunisian dates) or in fruit picking and storage stages. Indeed, approximately 30 000 tons and 2 000 000 tons of dates are often discarded in Tunisia and worldwide, respectively, due to their unsuitable texture and deteriorated organoleptic qualities (Mrabet et al., 2015). The few studies reported on the valorization of Tunisia low-quality dates, permit to classify these fruits in two classes: dates from the coastal oasis (5 000 tons) which are rich in reducing sugars and the continental oasis cv. Deglet Nour (30 000 tons), which are rich in sucrose (Chaira et al., 2009). ‘Deglet Nour’ the most produced and appreciated date cultivar was also the most investigated one even in the bioenergy production such as biogas and biohydrogen production (Ben Yahmed et al., 2020).

Although Tunisian secondary varieties such as Garen Ghzel, Alig and Kenta cultivars have similar fiber content and composition, they are less appreciated and not very used for human consumption. They are used for limited purposes such as animal feed and fertilizers (Smaali et al., 2012). This selective orientation results in a progressive disappearance of these common cultivars and therefore a date genetic variability reduction. Annually, large amounts of secondary date waste are produced and discarded in Tunisia without any treatment or proper valorization methods which can be considered as a real economic loss. Previous studies focused mainly on the chemical characterization (Chaira et al., 2009) and technological applications of these dates by-products especially dietary fibers DF (Mrabet et al., 2012, Mrabet et al., 2015, Elleuch et al., 2008, Smaali et al., 2011, Kareche et al., 2020) however its energetic valorization has received a little attention of the scientific community up to now (Lattieff, 2016, Jaafar, 2010, Souli et al., 2018).

Bioconversion is a technology used to transform biomass into bioenergy and high added-value products (Smaali et al., 2009, Djaafri et al., 2020, Ben Yahmed et al., 2018). This technology offers several advantages: it is simple, mature, environment friendly, cheap, renewable, helping to reduce the environmental impact of waste disposal (such as agricultural residues, organic animal, vegetable and industrial waste...) and represent a good alternative to fossil fuels (Djaafri et al., 2020).

Different bioconversion techniques are used to produce energy, among them the anaerobic digestion. In fact, anaerobic digestion of organic waste has gained increased attention by means of producing energy-rich biogas, mitigating greenhouse gas emissions, destructing pathogenic organisms and reducing problems associated with the disposal of solid organic waste (Souli et al., 2018). The anaerobic digestion occurs in four steps: hydrolysis, acidogenesis, acetogenesis and methanogenesis. Depending of the substrate composition and its structure, hydrolysis or methanogenesis can be considered as limiting steps (Brémond et al., 2018).

Thus, the objective of this study is to demonstrate the feasibility of valorizing ‘Kenta’, a Tunisian common dates cultivar, by the implementation of an integrated biorefinery process aimed at the co-production of date syrup and biogas. The crude fibers of ‘Kenta’ variety were recovered after soluble sugars extraction. Chemical composition of ‘Kenta’ discarded biomass as well as of the date syrup and the crude fibers extract (CFE) were analyzed. Biochemical methane potential

(BMP) tests were assessed to evaluate the energetic potential of this undervalued by-product. A basic economic study was also performed.

2. Materials and methods

2.1. Samples

'Kenta' variety collected at the ''Tamr stage" (full ripeness) was procured from the oasis of Gabes region (south of Tunisia) during the harvest season (September-October 2019). After removing the seeds, the date fleshes were rinsed with water, dried for 24 h at 40 °C, milled and preserved at 20 °C prior to fiber extraction and bioconversion.

2.2. Chemical composition

Date fleshes and date fibers were analyzed for TS (Total Solids) and VS (Volatile Solids) in accordance with APHA standard methods (Apha, 1998). The carbohydrates and uronic acids in solid phase were measured in duplicate using the strong acid hydrolysis protocol adapted from Effland (1977) (Effland, 1977) as described by Ben Yahmed et al (2020) (Ben Yahmed et al., 2020). Total fibers were determined according to the AOAC enzymatic-gravimetric method of Prosky et al. (Prosky et al., 1988) .

The cellulose and hemicelluloses content in Kenta fibers was determined on the basis of the monomeric sugar content as described by Monlau et al. following theses equations:

$$\text{Cellulose}(\%\text{TS}) = \text{Glucose}(\%\text{TS})\ 1.11 \quad (1)$$

$$\text{Hemicelluloses}(\%\text{TS}) = [\text{Xylose}(\%\text{TS}) + \text{Arabinose}(\%\text{TS})]\ 1.13; \quad (2)$$

with 1.11 the conversion factor for glucose-based polymers (glucose) to monomers and 1.13 the conversion factor for xylose based polymers (arabinose and xylose) to monomers (Monlau et al., 2012).

Kjeldahl nitrogen (TKN) was titrated using a Buchi 370-K after mineralization of the samples. Proteins were determined by multiplying TKN by 6.25. The content of lipid was determinate using the protocol described in the standard NF V 03-713 (AFNOR, 1984). The results of different components of dates and dates fibers were expressed in percent of total solid and were presented as mean ± SD (standard deviation of triplicates).

2.3. Biorefinery concept based on the co-production of soluble sugars and biogas

Figure 1 illustrates the overall methodology followed in this work. It consisted on a cascade bioconversion intended at the co-production of soluble sugars 'dates-syrup' and biogas via hot water extraction and anaerobic digestion of the residual fibers, respectively, from 'Kenta' common date cultivar (Fig.1).

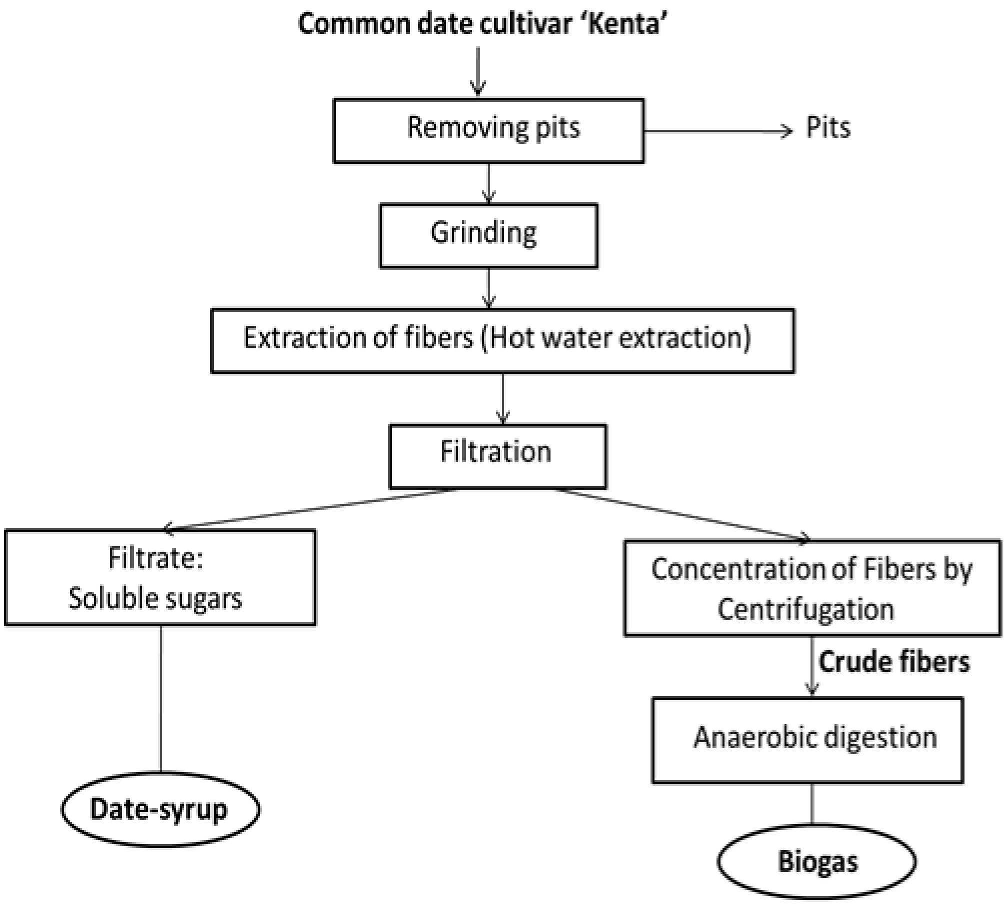

Fig.1 Biorefinery concept aimed at the co-production of date syrup and biogas from common date cultivar

The crude fiber extraction was carried out with hot water at 100 °C for 10 min. After solubilization of the sugars (sucrose, glucose and fructose), the fibers were recovered by filtration on gauze filter and centrifugation (6500g, 10 min). Five successive rinsings with water at 40 °C and of five centrifugations was released to concentrate the fibers until the residue was free of sugars. The residues obtained were dried to give the fibers concentrates then stored for biogas production.

2.4 Soluble sugars characterization

Sucrose, fructose and glucose contents were analyzed using high-performance liquid chromatography HPLC equipped with Eurospher NH_2 column (100 Å pore size, 7 mm particle size, 250 mm 4.6 mm) and RI Detector K-2301 (Knauer, Germany). The samples were filtered over a 0.45 mm membrane filter and degassed for 15 min in an ultrasonic bath Cleaner Model SM 25E-MT (Branson Ultrasonics Corporation, Danbury, CT). Acetonitrile and ultrapure water (80/20, v/v) were used as mobile phase with a flow rate of 1.0 mL/min. The integrator was calibrated with external standards consisting of glucose (2%), fructose (2%) and sucrose (1%) solutions.

2.5 Biochemical methane potential (BMP test)

To evaluate the methane production, batch BMP tests were performed in mesophilic conditions (35°C), as described by Jard et al. (Jard et al., 2013). Each bottle contained 4 gVS of inoculum (from an up flow anaerobic sludge blanket reactor of a sugar industry) and 2 gVS of ground dry dates fibers. They were filled to 400 mL with a bicarbonate buffer supplied with nutriments (Table S1 in Supplementary material). Two controls were used: sample containing ethanol to check the inoculum activity and a blank to measure endogenous methane production which was subtracted from the methane production. Bottles were rapidly sealed using butyl-rubber stoppers and held with clamped aluminum collars. Nitrogen gas was flushed into airspace in order maintain anaerobic conditions. Once prepared, bottles were shaken and incubated at 35 °C with continuous agitation.

During incubation, biogas production was regularly monitored by pressure measurement of the headspace using a manometer (Keller, LEO 2). The concentration of CH_4 in biogas was determined by gas chromatography (PerkinElmer, Clarus 480). BMP was carried out until biogas production stopped. Methane yields were calculated by dividing the corrected methane volume (standard pressure and temperature) by the weight of sample VS added to each bottle (Ben Yahmed et al., 2020). The volume of methane produced ΔV_{CH4} (mL) between the dates j and j-1 was calculated following Eq (1):

$$\Delta \boldsymbol{V_{CH4}} = \left(\left[y(j)P1(j)\frac{V}{RT}\right] - \left[y(j-1)P2(j-1)\frac{V}{RT}\right]\right)\frac{RT^{\circ}}{P^{\circ}} \qquad (1)$$

Where y(j-1) et y(j) are CH4 contents in biogas at dates j-1 and j, respectively

P1(j) (Pa) is the bottle head space pressure before sampling at the date j,

P2(j-1) (Pa) is the bottle head space pressure after gas release at the date j-1,

V (mL) is the bottle head space volume

R is the ideal gas constant (8.314 $J.(mol.K)^{-1}$),

T is the bottle temperature (K),

T° et P° are normal condition of temperature and pressure (273,15 K, 1013 hPa).

For the determination of the kinetic parameters of the methane production, a first-order exponential model was used according this equation:

$$M = M_{max} .(1 – \exp(-K.t))$$

where M (mL CH_4/g VS) is the cumulative specific methane production, M_{max} (mL CH_4/g VS) is the ultimate methane production, K ($days^{-1}$) is the specific rate constant or apparent kinetic constant and t (days) is the time. The adjustment by non-linear regression of the experimental data (M, t) using the Sigmaplot software (version 14.0) permitted the calculation of the parameters K and M_{max}.

3. Results and discussion

3.1 Chemical composition of dates fleshes

The composition of the biomass feedstocks plays an important role in the energetic bioconversion. Thus, the approximate chemical composition of dates fleshes (Table 1) as well as of the extracted crude fibers (Table 2) of 'Kenta' cultivar was studied.

Table 1. Chemical composition of date fleshes of different Tunisian cultivars

Composition	Kenta	Deglet Nour	Garn ghzal	Alig	Smiti
TS (%wet weight)	93.1 ± 0.2	76.7± 0.1	ND	ND	ND
VS (%TS)	90.2 ± 0.2	98.6 ± 0.1	ND	ND	ND
Total carbohydrates (%TS) [a]	79.5 ± 0.8	79.8 ± 0.8	61.61 ± 4.92	62.7	35,57
Glucose (%TS)	27.4 ± 0.1	15.2 ± 0.1	ND	ND	17,74
Fructose (%TS)	28.1 ± 0.2	15.8 ± 0.1	ND	ND	17,83
Sucrose (%TS)	24 ± 0.4	48.8 ± 0.5	ND	ND	-
Total fibers (%TS)	7.4 ± 0.5	12.3 ± 0.4	ND	ND	ND
Proteins (%TS)[b]	2.8± 0.1	2.6 ± 0.1	1.70 ± 0.10	3.71	ND
Lipids (%TS)	0.41 ± 0.02	0.24 ± 0.02	0.52 ± 0.01	1.79	ND
Reference	Present study	(Ben Yahmed et al., 2020)	(Mrabet et al., 2015)	(Souli et al., 2018)	(Chaira et al., 2009)

ND: not determined.

[a] Total carbohydrate content was quantified as the sum of each individual sugar (glucose, fructose and sucrose)

[b] The protein content was calculated by using a nitrogen conversion factor of 6.25

Table 1 shows that 'Kenta' flesh is rich in soluble sugars (79.5%) mainly fructose, glucose and sucrose, fibers (7.4%) and small quantities of proteins and lipids based on total solids. These results are in agreement with those reported by Chaira et al (2011) which demonstrated that 'Kenta' variety belonging to the coastal oasis cultivars is rich in reducing sugars unlike 'Deglet Nour' (continental oasis cultivar) which is rich in sucrose (Chaira et al., 2011, Ben Yahmed et al., 2020). Indeed, as shown in table 1, the biochemical composition of dates varies significantly among cultivars and it depends on the culture conditions such as the growth zone and the stage of maturity (Chaira et al., 2009, Sahari et al., 2007). This variation in biochemical components resulted in a significantly different biomethane production. In fact, Souli et al. study demonstrate that for date pulp, the carbohydrate is the most relevant parameter to evaluate the methane production kinetics of the wastes of dates (either methane potential or methane yield rate). Moreover, the results of this study showed a correlation between the lipid content and the methane production for both date pulp and seeds. An opposite observation was achieved reporting that lipids decreased the methane production (Souli et al., 2020).

3.2 Development of an integrated biorefinery concept based on soluble sugars extraction and biogas production

An integrated processing approach based on the extraction of soluble sugars and the use of issued crude fibers for biogas production was developed. This process consisted of three main stages namely the hot water extraction of sugars, the filtration of the juice and the anaerobic digestion of the residual crude fiber with two intermediate units such as the centrifugation for the concentration of the crude fiber extract and the evaporation for the date syrup preparation. The detailed flow diagram of this process with main inputs and outputs is illustrated in Fig.2. The developed concept allows to recover 0.66 g sugars/gVS flesh and 0.1 g fibers/gVS flesh.

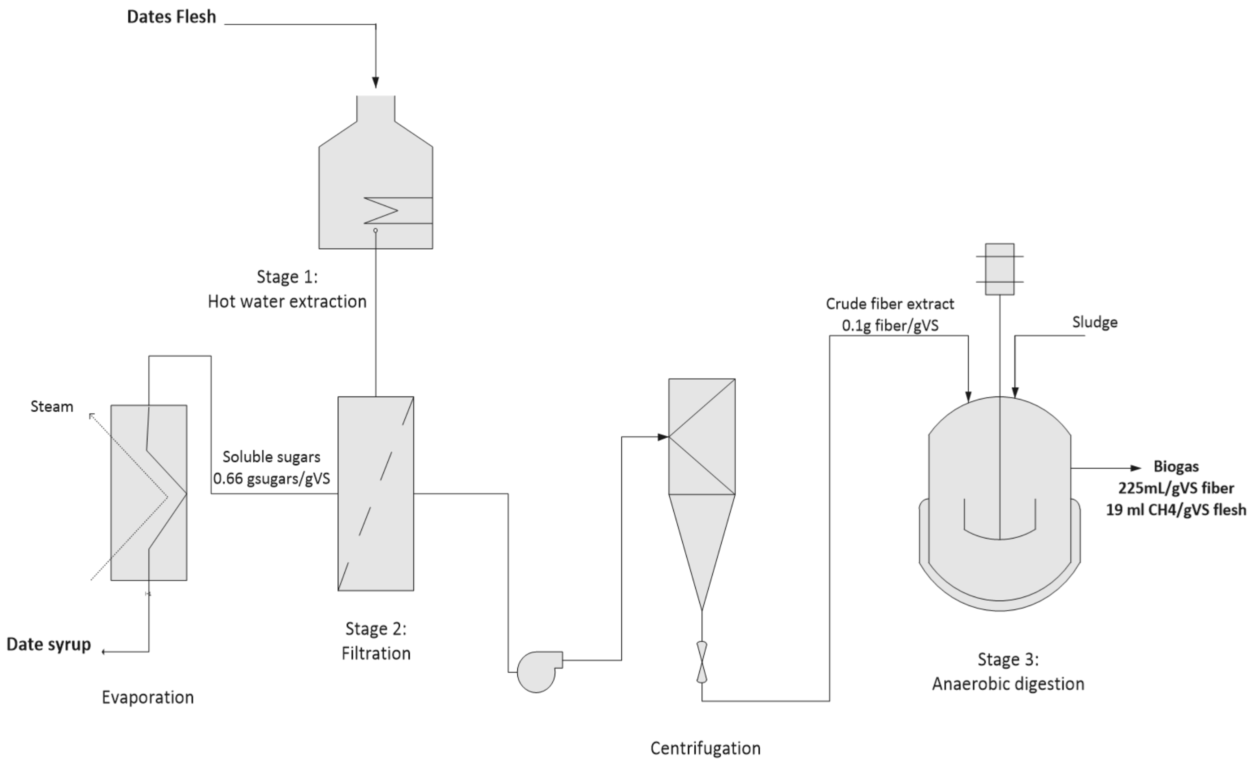

Fig.2 Diagram and main inputs/outputs of the units in the proposed biorefinery concept aimed at the co-production of date syrup and biogas from 'Kenta' dates flesh**.**

The obtained date syrup was characterized. Sugar composition was assessed by HPLC chromatography, which the chromatogram presented in Fig. 3 shows a typical composition of fructose, glucose and sucrose (Fig.3).

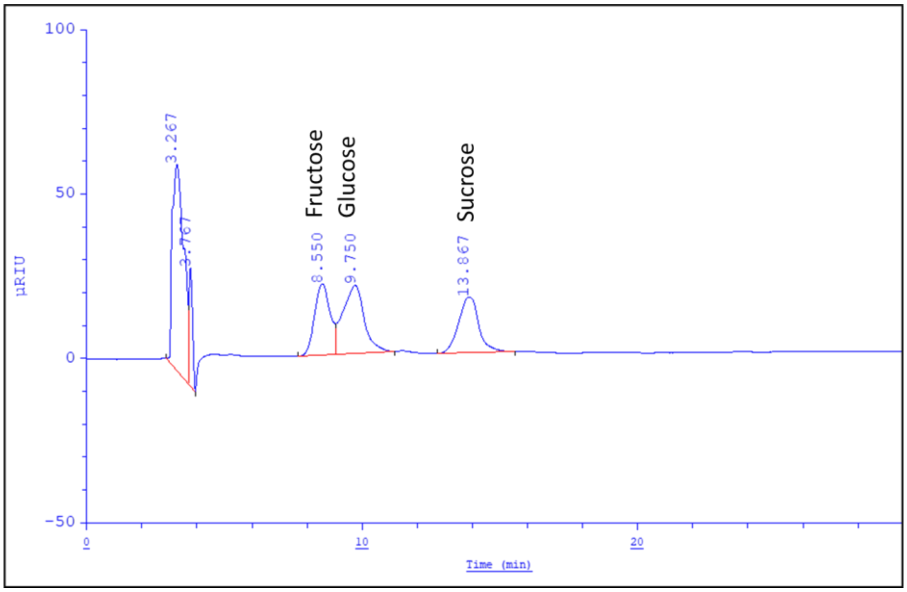

Fig.3 High performance liquid chromatography analysis (HPLC) of the ‘Kenta’ cultivar syrup

Their concentrations after calibration are given in table 2. This aqueous extract is rich in glucose (22.6 ± 0.1 %TS) and fructose (23.2 ± 0.2% TS) which is characteristic of coastal oasis dates cultivars. This sugar juice could be destined to the agro-alimentary industry such as bakery products, ice cream and in confectionery (Lajnef et al., 2021, Abbès et al., 2015). Besides, this high-added value product has recently an increased interest thanks to their potential health benefits and great biological activities (Baliga et al., 2011).

Furthermore, the date crude fibers extract (CFE) was also characterized. As shown by Table 2 the crude fiber fraction is rich in carbohydrates (33.2 ± 0.7% TS with 10.2 ± 0.5 %TS of cellulose and 19.4± 0.6 %TS of hemicellulose) and proteins (8.8 ± 0.1% TS) making it a suitable feedstock for the biogas production. This production was carried out without any pretreatment step in order to make the results of this study more applicable to the biogas plant operation and facilitate therefore the scale up of the proposed concept.

Table 2. Chemical composition of 'Kenta' syrup and crude fibers

Composition	'Kenta' syrup	'Kenta' Fibers
TS (%wet weight)	93.1 ± 0.2	90 ± 0.2
VS (%TS)	90.2 ± 0.2	70 ± 0.1
Total carbohydrates (%TS) [a]	64 ± 0.8	33.2 ± 0.7
Glucose (%TS)	22.6 ± 0.1	11.2 ± 0.5
Fructose (%TS)	23.2 ± 0.2	ND
Sucrose (%TS)	18.2 ± 0.4	ND
Xylose (%TS)	ND	16.9 ± 0.2
Arabinose (%TS)	ND	5.1 ± 0.4
Total fibers (%TS)	7.4 ± 0.5	-
Uronic acids (%TS)	ND	16.8 ± 0.4
Proteins (%TS)[b]	2.8± 0.1	8.8 ± 0.1
Lipids (%TS)	0.41 ± 0.02	ND

ND: not determined.

[a] Total carbohydrate content was quantified as the sum of each individual sugar

[b] The protein content was calculated by using a nitrogen conversion factor of 6.25

After 45 days of anaerobic digestion of the residual fibers, a maximum biomethane yield of 225 ± 11 ml CH4/gVS $_{fibers}$ corresponding to 19 ml CH4/gVS $_{flesh}$ was obtained (Fig.4). Besides, kinetic parameters were determined using a first order exponential model. The results of the modelling given by the non-linear regression of the experimental data allowed the calculation of the parameters K and Mmax for the methane production which were 0.12 ± 0.01 days^{-1} and 217 ± 12 mL CH_4/VS respectively.

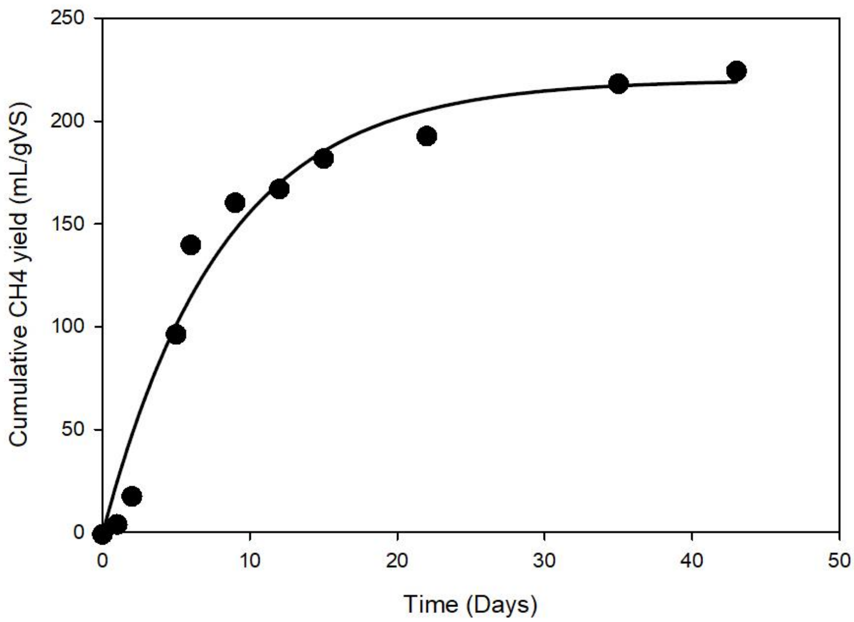

Fig. 4 Cumulative methane yield, expressed as mL CH_4/gVS added, obtained during the BMP tests performed with untreated 'Kenta' fibers extract. The standard deviation was lower than 10%. Exponential model (solid line) fitting to experimental data (solid points)

The maximum methane yield obtained is higher than the predicted one. The variations of above parameters could be attributed to factors such as the quantity and quality of the inoculums, batch digestion test parameters (e.g. digestion temperatures, substrate to inoculum ratios) and substrate characteristics (e.g. organic ingredients, volatile solid content) (Li et al., 2018).

In the same conditions (mesophilic conditions, without pretreatment), Souli et al (2018) found a BMP of 290.5 ± 1.2mL CH4/VS by the anaerobic digestion of the entire 'Kenta' date pulp not only the extracted fibers (Souli et al., 2018). However, the proposed biorefinery approach based on crude fibers digestion after soluble sugars extraction allows both biogas production as source of energy and date syrup recovery as high added value product. It permits therefore an almost complete utilization of the dates byproducts and represents therefore a good alternative for the management of this discarded biomass waste. The digestate is the only co-product of the entire process remaining after methane production. It could be used as fertilizer since it represents the unconverted biomass without any chemical addition.

To better evaluate the proposed biorefinery concept, a preliminary economic study must be carried out. Typically, an economic approach of a biorefinery concept can be focused on two key cost contributors namely the raw material (including its transportation) and the operating costs (energy, enzymes, reagents…). For the glucose syrup production, the raw material cost is between 4% and 8% approximately (Dávila et al., 2014). However, in our case, undervalued common cultivar which represent a discarded biomass is used as raw material. So, the proposed process will save about 4% of the biorefinery total cost if this later will be installed locally. Besides, it will allow financial benefits to farmers and meet the local fuel consumption of rural population.

The operating costs, especially the energy requirement, are the main factors that contribute to a high total production cost of 80% (Dávila et al., 2014). Generally, the pretreatment is among the costliest steps in the bioconversion of lignocellulosic biomass in particular dilute acid hydrolysis which is more expensive than other physicochemical pretreatments methods (Agbor et al., 2011). By using a hot water extraction, as simple and eco-friendly method, we can decrease the pretreatment cost step (no reagents or enzymes used). Besides, in order to reduce the energy cost, a heat integration strategy is suggested (Sánchez and Cardona, 2012).

An economic evaluation based on Tunisian market conditions permitted to estimate the cost and the revenue of the date syrup which are 0.45 USD/kg and 0.64 USD/kg, respectively (Lajnef et al., 2021). Nevertheless, the date syrup is not the only output of the proposed concept, the biogas issued from the anaerobic digestion of the residual crude fibers is also produced. This sequential production improves the energetic efficiency of the biobased process and represents an excellent example of circular bioeconomy.

Conclusion

This study presents a proof of an innovative concept demonstrating the feasibility of the co-production of both biogas and date syrup from the undervalued common cultivar ‘Kenta ‘using an eco-friendly process. Throughout the proposed integrated biorefinery approach, the date syrup was extracted with a 0.6 g/g yield and the residual crude fibers extract was submitted to anaerobic digestion. The methane potential reached 225 mL CH_4/gVS fibers. Thus, it allows an efficient bioconversion of the discarded dates biomass. Furthermore, throughout the valorization of the date’s secondary varieties, the biodiversity of coastal oases could be conserved. This concept leads therefore not only to the bioenergy production but also to sustainable ecological effects. Some stages of this integrated process may be optimized to improve the production yields. A detailed techno-economic analysis is also necessary to envisage the scale-up of this biorefinery concept.

Authors' contributions

Ben Yahmed N. carried out the most of the experiments, participated to the interpretation of data and the redaction-correction of the manuscript. Carrere H. planned and supervised the BMP experiments at the LBE, participated to the interpretation of results and the correction of the manuscript. Chaira N. provided the dates cultivar and participated to the interpretation of results. Smaali I. conceptualized the work, supervised the soluble sugars extraction and characterization experiments at the LIP-MB Laboratory and participated to the redaction-correction of the manuscript.

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Supplementary material

Macro nutriments		
NH4Cl	26.2	g/L
KH2PO4	10	g/L
MgCl2, 6H2O	6	g/L
CaCl2, 2H2O	3	g/L

Micro nutriments		
$FeCl_2, 4H_2O$	2	g/L
$CoCl_2, 6H_2O$	0.5	g/L
$MnCl_2, 4H_2O$	0.1	g/L
$NiCl_2, 6H_2O$	0.1	g/L
$ZnCl_2$	0,05	g/L
H_3BO_3	0,05	g/L
Na_2SeO_3	0,05	g/L
$CuCl_2, 2H_2O$	0,04	g/L
$Na_2MoO_4, 2H_2O$	0,01	g/L
Bicarbonate buffer		
$NaHCO_3$	50	g/L

Table S1. Composition of BMP nutriments