Chemical Composition of Three Types of Low-grade Dates in Upper Egypt

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Abstract

The aim of the study was to determine the chemical composition of low-grade dates for three types of palm cultivated in Egypt (Phoenixdactylifera L.). in order To assess their sugars, crude protein, crude fat, ash and the moisture. The dates were rich in sugar (71.9–81.5% dry weight), while ash represented (2–2.31%). they contained low concentration of protein and very low concentration of fat (3.37–3.86% and 0.19 –0.26%, respectively),and moisture content (9-11%). Although the mineral contents varied widely, all varieties could be an important source of potassium. These results show that dates are nutritious and can play a major role in human nutrition and health. The high content of sugars found in low-grade dates makes significant chance of use it to produce of bio-alcohol.

Keywords: Chemical composition; Low-grade dates; Dates in Egypt; Bio-alcohol.

Introduction

The date palm, known scientifically as *Phoenix dactylifera* of the monocot's family, is a dioecious species with several hundred varieties. It grows best between 10 and 39 degrees north latitude in the desert and arid regions. This is because of its resistance to extreme weather conditions including soil lacking organic matter, salinity of about 18g L^{-1}, excessively cold nights and hot days***(Kacem-Chaouche et al., 2013)***. In Egypt, date palms are distributed in Nile valley, oases and desert, Include soft dates (Zagloul, Samani, Hayani, Bent-Aicha, Amhat, etc) semi–dry dates (Al-Amri, Saidy, Al-Aglani, etc) and dry dates (Barakawi, Ebrimi,

Sakouti, etc). Besides, there is a great number of seedling date palms (Manthour), as a result of sexual reproduction, some of them are highly desirable for fruit qualities and propagation of their off-shoots as well ***(Ragab, 2011)***. The global production of date fruits exceeds 7,600,315 tons at 2014 annually in the world ***(FAOSTAT, 2017)***. Egypt is the largest producer of dates in the world, with more than 1,465,030 tons of dates ***(FAOSTAT, 2017)***, collected from 12,000,000 of date palm trees **(caae-eg, 2016)**,distributed in 44,037 Hectare at 2014 ***(FAOSTAT, 2017)***.The date palm is rich in mineral salts and vitamins and is an excellent material for producing refined sugar, concentrated juice, confectionery pastes and fermentation products ***(Assirey, 2015)***. Dates contain small amounts of vitamins C, B1,thiamine, B2,riboflavin and nicotinic acid , and studies have shown that dates have strong antioxidant , anticancer and antiviral activities ***(Assirey, 2015)***.Botanically date fruit is one-seeded berry consisting of fleshy mesocarp covered by a thin epicarp, a hard endocarp surrounding the seed***(Hasnaoui et al., 2010)***,The present study aims to throw chemical composition in fruits of low graded Siwi, Saidi and Aswani dates palm grown in south valley, Egypt.

Materials and methods

Chemicals :3,5-Dinitrosalicylic acid, Rochelle salt (sodium potassium tartrate), phenol, sodium bisulfate, sodium hydroxide, sulfuric acid (98%), D-(+)- glucose, petroleum ether (40-60 ^{0}C).

Instrumentals:

Muffle furnace (Isuzu), drying oven (DRU600TA),Soxelet unit, Analytical balance (at least 0.1 mg =100µg sensitivity), spectrophotometer, ES-ICP unit (inductive coupled plasma emission spectrometer) (ICAP 6200); distillation system.

Preparation of date flour : Dates(dates that using in this study in tamr stage) were pitted and freeze by drying using liquid nitrogen , crashed to small sizes using hand Hun ceramics ,dried at 55-60°C in an oven for 24h. They were then milled and sifted by Mixer Home or hand Hun. The prepared flour was stored in 4°C to minimize hydrolysis enzymatic reactions.

Chemical composition of dates.

Determination of moisture:

The moisture content was measured by drying the sample in an air oven at 70◦c ***(Ragab, 2011)***, until it reached to a constant weight. The difference between the weight of the sample before and after drying is the moisture content ,it can be calculate using the following formula: (E - m).100 / E, where E is the original mass in grams, and m is the mass in grams of the dry sample ***(Kacem-Chaouche et al., 2013)***.

Determination of ashes:

The ash content was determined from the difference after heating samples of dates in muffle oven for 8 h at 600^{o}C. ***(Assirey, 2015)***. In this work the 3h at 600^{0}C was found to be enough to determine of ash, this saves time and energy.

Procedures: 5 g of dried date, dried and weighted crucible and put in muffles furnace at 600^{0}C for 3h then the crucible contained ashes of sample placed in desiccated to reach to room temperature, ashes percent calculated from the following equation %ashes = ($m_2 - m_1$) / wt. of sample(5g) × 100, where m_1 is the weight of the empty crucible and m_2 is weight of crucible + ashes.

Note: Balance with 100µg sensitive was used to get more accurate estimation of ashes.

Extraction and determination of total sugars:

Total Sugars were determined by phenol-sulfuric acid method***(DuBois, Gilles, Hamilton, Rebers, & Smith, 1956)***, in this method all non-reducing sugar converted to reducing sugar and detects mono-, di, oligo- and polysaccharides, so that this method used to estimate the total sugar present in fruits. It is the most common, effective and simple method have been used in a broad range of fields ***(Would& Tsigie, 2015)***. Sensitivity of this method range from 10 to 100µg of total sugar and the quantification is made from calibration curve using glucose or pentose as standard ***(de Toledo, Ruvolo-Takasusuki, de Oliveira, Chambó, & Lopes, 2012)***.

Procedures:

1- Extraction of sugar:

0.1 g (100 mg) of sample (powder) hydrolysis by 5ml of 2% H_2SO_4 for 20 hr at rome temperature ***(Neeru Agrawal, 2015)*** ,then add in water path at 50 ^{0}c for 3hr.

- Mixture was filtrated and then supernatant was diluted to 100 ml by distilled water.

2- Calibration curve:

Standard solution: 0.1g of D-glucose dissolved in 100 ml distilled water (1mg/ml) and 10ml of this mixture diluted to 100ml by distilled water (0.1mg/ml). Take 0.2-0.4- 0.6-0.8and 1ml in test tube and complete to 1ml with distilled water .

- Add 1ml of phenol 5% (5g of phenol crystal dissolve in 100ml distilled water- immediately prepare), then rapidly add 5ml of 96% sulfuric acid to the tube.
- Leave test tubes 10 min at rome temperature, after that put the tubes in water bath at 30-35 ^{0}C for 20 min, then read absorbance at 490 nm.

3- Estimation of total sugars in dates sample:

0.1ml of sample (mixture in step 1) complete to 1ml, then add 1ml phenol 5% and 5 ml sulfuric acid 96% ,leave the mixture at Rome temperature for 10 min, after that put the tubes in water bath at 30-35 ^{0}C for 20 min, Then read absorbance at 490 nm, and from calibration curve using to get concentration of sample that corresponding to absorbance and the total sugar percent calculated from the following equation.

T.S%=concentration of sample ×diluted factor×100

Extraction and determination of reducing sugars:

Reducing sugars were extracted from macerated dates with water at 70 ^{o}C for 2hr ***(El-Sharnouby, Aleid, & Al-Otaibi, 2015)*** and quantified by the 3,5-dinitrosallicylic acid method **(Miller, 1959)**. The sensitivity of the method is from 100 to 500 μg.ml^{-1} of reducing sugar. A standard glucose or fructose solution is used to build the calibration curve and get the straight line equation to quantify samples ***(de Toledo et al., 2012).***

Procedures:

1-Sample extraction:0.1gm of sample with 15ml distilled water heated at 70^0C in water path for 3hr., then filtrated and completed to 100 ml (1mg/ml).

2- DNS reagent : dissolve 1gm of dinitrosalicylic acid, 0.2gm (200mg) of phenol's crystals and 0.05gm (50mg) of sodium bisulphate in 100 ml sodium hydroxide(1%) and 40% Rochelle salt solution (potassium sodium tartrate ***(Miller, 1959).***

3- Calibration curve :0.1gm (100mg) of glucose dissolved in 100ml of distilled water (1mg /ml), (table 1).

Table 1. Prepare different concentration of working standard of glucose solution for calibration curve of DNS methode.

	glucose mL	water mL	Concentrations mg(1000µg)/mL	ML of glu. Water mix	ML of DNS	Heated in boiling water path for 5min.	40% Rochelle salt solution Ml
1	0.5	4.5	0. 1	3	3		1
2	1	4	0.2	3	3		1
3	1.5	3.5	0.3	3	3		1
4	2	3	0.4	3	3		1
5	2.50	2.5	0.5	3	3		1
blank	-	3			3		1

4- Estimation of reducing sugars in dates sample by DNS method

	Extract. mL	Water mL	Ml of mix.	Ml of DNS	boiling for 5min.	40% Rochelle salt solution
Sample	1.5	1.5	3	3		1

Note:1 mL of 40% Rochelle salt solution added while the contents of the tubes are still warm.

Determination of mineral elements:

K, Fe, Mg and other minerals were measured by inductively copledplasms emission spectrometer (ICAP 6200),

Determination of nitrogen and protein:

The protein content of dates were been determined on the basis of total nitrogen content. the Kjeldahl method is almost universally applied to determine nitrogen content, total nitrogen is then multiplied by a factor to arrive at the protein content, The Kjeldahl method consists of three basic steps ***(AOAC, 1990)***:

1) **Digestion:**
   1 g of Powdered sample was digested in a 500ml volumetric flask by boiling with 20 ml of concentrated sulfuric acid and (16.7 g K_2SO_4, 0.01 g anhydrous copper sulfate, and 0.6 g TiO_2)as catalyst to increase the boiling point of the medium (from 337 °C to 373 °C) ,until the mixture was clear, cool to room temperature, cautiously add 250ml distilled water to the volumetric flask (in digestion step, ammonia not lost because will be in ions form).
2) **Distillation**:
   Connected digestion flask to distillation system and distill at about 7.5 boil rate (temperature set to bring 250 mL water at 25^{o}C to boil in 7.5 min), Ammonia was steam distilled from of the digest to which had been added 80 ml of 45% sodium hydroxide solution to make mixture strongly alkali (slowly down side of flask, do not mix until the flask is connected to distillation system or ammonia will be lost). Until at least 150 mL distillate is collected in a conical flask containing 100ml 0.1N HCl and 3-4 drops of methyl red indicator (condenser tip is immersed in conical flask that containing 100ml 0.1N HCl to react all ammonia with HCl acid).
3) **Titration:**
   The ammonia that distilled into the receiving conical flask reacted with the HCl acid and the excess acid in the flask was estimated by back titration against 0.1NaOH with color change from red to yellow (end point).

Calculate the percentage of nitrogen and protein:

(100ml HCl×0.1N HCl)-(V NaOH×0.1NNaOH)=V(N)HCl that react with NH_3=V(N)NH_3

,so the amount of nitrogen present in

$$V\ (N)\ NH_3=14/1000\times V\times N$$

where 1000 ml of ammonia contain 14g nitrogen, then the percentage of nitrogen(N%) in sample was calculate as follow:

the amount of nitrogen / the wight of sample×100

Total equation ,

$$N\%= \frac{(100ml\ HCl\times 0.1N\ HCl)-(V\ NaOH\times 0.1NNaOH)\times 14\times 100}{1000\times 1g\ \text{of sample}}$$

Crude protein =N% ×protein factor

Protein factor=6.25

Determination of crude fat:

Crude fat were extraction using the soxlet extraction ***(Nielsen, 2014)*** using petroleum ether (40-60^{0}C) as organic solvent to dissolve the fat only, Other organic solvent dissolve most of contents in fruits especially sugars, then crude fat can be calculate using the following formula (% Crude fat = (W2 – W1) x 100/S); where W1= weight of empty flask (g),W2= weight of flask and extracted fat (g), S= weight of *dried* sample. In case of wet sample[%(the crude fate + moisture)] can be calculate using the following formula [(initial weight of sample-final weight of sample) x100/weight of wet sample],% crude fat= %(crude fat + moisture) - %moisture ***(Nielsen, 2014)***.

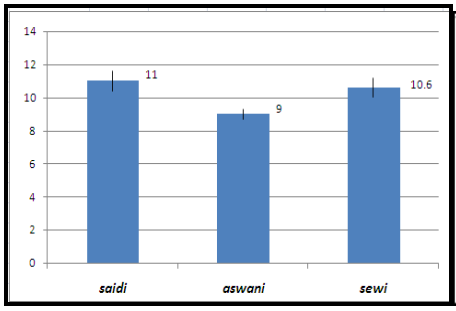
Results and Discussion

The moisture content ranged from 9 to 11%. Saidi type had the highest moisture content and Aswani the lowest. The results are near to those reported previously ***(Al-Shahib & Marshall, 2002; Assirey, 2015; Barreveld, 1993; Kacem-Chaouche et al., 2013)***, with some differences related to date variety , environmental conditions and agro-climatic ***(Assirey, 2015)***. The ash content ranged from 2 g/100 g dry matter in saidi to 2.31 g/100 g in aswani, comparable to those reported in ***(Assirey, 2015; Besbes, Blecker, Deroanne, Drira, & Attia, 2004; Hasnaoui et al., 2010)***. The results are indicated in Fig.1 and 2, and Table 2.

Table 2. Content of moisture and ashes (W%).

Date variety	Moisture	Ashes
Saidi	11±0.6	1.95±0.001
Aswani	9±0.3	2.31±0.013
Sewi	10.6±0.6	2.20 ±0.01

± the standard Error.



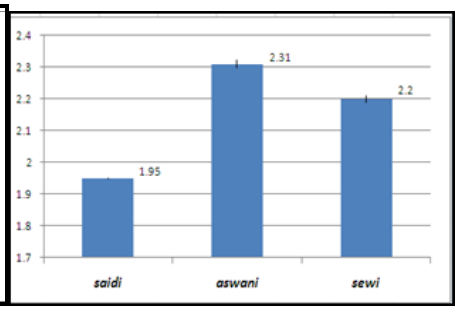


Fig1: **Moisture content in dates.** **Fig2**: **Ashes content in dates.**

Varieties of dates have been studied where they contain high value of the reducing sugars ranged from 65.6 % to 73.5% from dry matter, compared with non-reducing sugars that ranged from 6.32% to 8% from dry matter ,comparable to those reported in previous studies ***(Al-Orf et al., 2012; Assirey, 2015; Ragab, 2011)***. The highest value of sugars was founded in siwi cultivar (75.5 and 83.5) % for reducing and total sugar, respectively and the lowest value was founded in aswany cultivar (65.6 and 72.1)% for reducing and total sugar, respectively. The results are indicated in Fig. 3 and Table 3.

Table 3. Content of total ,reducing and non-reducing sugars.

Date variety	Total sugars%	Reducing sugars%	Non-reducing sugars%	Non-Reducing / reducing sugars ratio
Saidi	74	65.44±1.2	8.56	0.13
Aswani	71.9	65.6±0.5	6.32	0.1
Sewi	81.5	73.5±1	8	0.11

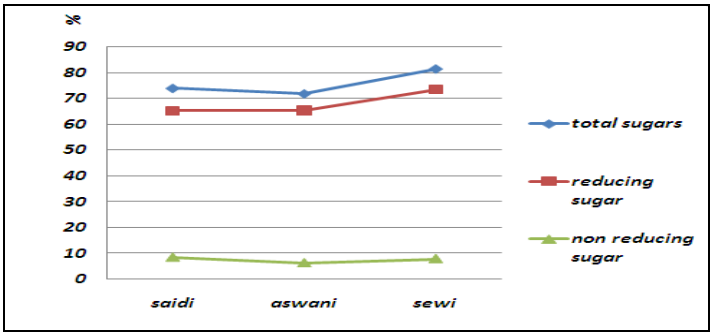


Fig 3: **Total ,reducing and non reducing sugars in dates.**

As indicated in Fig. 4 and Table 4, dates had low content of proteins ,with low difference of proteins content between three types of studied dates, with the highest value 3.86 g /100g was fund in Sewi, and the lowest value 3.37 g/100g in Aswani, so that dates are not a rich source of protein **(Al-Orf et al., 2012),** dates had very low content of fats ranged from (0.186-0.26) % in dry**.**

Table 4. Content of nitrogen and crude protein.

Date variety	Nitrogen /100g	Crude protein /100g	Fats /100g
Saidi	**0.595**	**3.72**	**0.186±0.006**
Aswani	**0.539**	**3.37**	**0.239±0.002**
Sewi	**0.617**	**3.86**	**0.263±0.007**

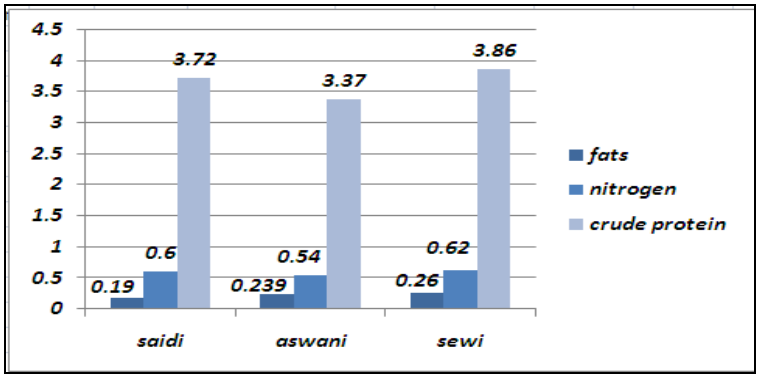


Fig 4:**Crude protein, nitrogen and fats content in dates.**

Table 5 shows The results of estimation of various minerals like sodium, potassium, calcium, copper, iron, magnesium, manganese and zinc indecated that the date cultivars were poor in micro-

elements (Fe, Zn, Cu, Mn), but they rich in most of macro-elements (Na, k, Mg, Ca), where The micro-elements content ranges for studied date types per 100g dry weight were 0.64-1.02 mg for zinc, 0.58-0.69 mg for manganese, 2.6-4.8 mg for iron and 0.58-0.81 mg for copper, For the macro-elements, their content per 100g dry weight were 528.1-766 mg for potassium, 75.3-119 mg for calcium, 57- 62 mg for magnesium and 74-77 mg for sodium, This results comparable to the reported result in ***(Assirey, 2015; S. AL-HOOTI, 1997)***.

Table 5. Content of minerals Elements.

Minerals \date varieties	Saidi	Aswani	Sewi
Micro-elements in mg/100g			
Zinc	**0.641**	**0.663**	**1.027**
Manganese	**0.589**	**0.676**	**0.676**
Iron	**4.09**	**4.79**	**2.59**
Cupper	**0.806**	**0.688**	**0.576**
Macro-elements in mg/100g			
Potassium	**528.1**	**627.8**	**765.7**
Calcium	**75.3**	**119**	**84**
Magnesium	**56.7**	**61.5**	**62.4**
Sodium	**7.7**	**7.4**	**7.7**

Conclusion

This study provides baseline information on three type of low grade date grown in upper Egypt. The results show that dates have a high content of sugar (71.9 –81.5%, dry weight), low concentrations of protein(3.37–3.86%) and very low content of lipids (0.19- 0.26%) ,ash content (2 – 2.31%), moisture content (9 -11 %) and The predominant mineral is potassium . These results suggested that dates are nutritious and can play a major role in human nutrition and health. In addition, low grade dates with high content of sugar is asuitable resource for bioethanol production.

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الملخص العربي

التركيب الكيميائي لثلاثة انواع من التمور قليلة الجودة في صعيد جمهورية مصر العربية

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يعتبر البلح من الفواكه المنتشرة بكثرة فى الشرق الاوسط والوطن العربي وفى بعض البيئات العربية والصحراوية يعتبر البلح جزءاً من ثقافتها ويتم تناوله بشكل يومي وفى العالم الإسلامي ويعتبر البلح من الفواكه التي ورد في فضلها وفى الاستشفاء بها احاديث كثيرة منها :

١ - عن عائشة – رضى الله عنها – أن النبي صلى الله عليه وسلم قال: " إن في تمر العالية شفاء– أوقال تريافاً " رواه مسلم وأحمد. وفي رواية لأحمد "في عجوة العالية شفاءأ وترياق أو البكرة على الريق ".

٢ -وعن سعد – رضي الله عنه – قال: قال رسول الله صلى الله عليه وسلم: " من تصبح كل يوم بسبع تمرات عجوة لم يضره في ذلك اليوم سم ولاسحر" (رواه البخاري ومسلم)

٣ - وعن أبي هريرة – رضي الله عنه –قال: قال رسول الله صلى الله عليه وسلم :" العجوة من الجنة وفيها شفاء من السم والكمأة من المن وماؤها شفاء للعين " رواه الترمذي وأحمد بإسناد صحيح.

وينتشر نخيل البلح في البيئات الصحراوية والمناطق قليلة المياه بسبب قدرتها على النمو فى ظروف لا تتحملها كثير من الاشجار الاخرى مثل الملوحة العالية للتربة والتي قد تصل 18جم/لتر ودرجات الحرارة شديدة الارتفاع وشديدة الانخفاض وتنمو ايضا فى الأراضي الفقيرة بالمواد العضوية ،ويوجد الكثر من انواح البلح تعد بالألاف والمشهور منها فى مصر (الامهات، بنت عيشا ،الحيانى، سمانى، الصعيدي، الزغلول، البريمى، العامري، السكوتى)

ويتركز زراعة نخيل البلح على ضفاف وادى النيل والصحارى المصرية ،وتعتبر مصر اكثر دول العالم فى انتاج البلح بانتاج ما يصل الى المليون ونصف المليون طن سنويا ويوجد فى مصر ما يقرب من 16 مليون شجرة نخيل منهم 12 مليون شجرة مثمرة ولكن معظم البلح المصرى من الانواع قليلة الجودة ،وفى هذا البحث تم دراسة وتقدير(الرطوبة ،الرماد، الدهون، النيتروجين والبروتين، السكريات الكلية والسكريات المختزلة ،العناصر الموجودة فى البلح) فى ثلاث انواع من البلح وتم التسمية كل نوع على اساس المناطق الجغرافية التى ينمو فيها (صعيدي وتم الحصول علية من محافظة سوهاج،ا لاسوانى، السيوى من الوادى الجديد) وتم تقديرهذه المكونات فى البلح فى مرحلة التمر بعد تجفيفه وطحنة، وكانت النتائج التى تم الحصول عليها كالتالي:

الجدول رقم 1. **تقدير نسبة الرماد والرطوبة**

الانواع	الرطوبة ٪	الرماد ٪
صعيدى	11±0.6	1.95±0.001
اسوانى	9 ±0.3	2.31±0.013
سيوى	10±0.6	2.20±0.01

الجدول رقم 2. **تقدير نسبة الدهون والبروتين**

انواع البلح	النيتروجين ٪	البروتين الكلى ٪	الدهون ٪
صعيدى	0.595	3.72	0.186±0.006
اسوانى	0.539	3.37	0.239±0.002
سيوى	0.617	3.86	0.263±0.007

الجدول رقم 3. **تقدير نسبة السكريات الكلية والسكريات المختزلة وغير المختزلة**

انواع البلح	السكريات الكلية %	السكريات المختزلة %	السكريات الغير مختزلة %	النسبة بين السكريات المختزلة والغير مختزلة
صعيدى	74	65.44±1.2	8.56	0.13
اسوانى	71.9	65.6±0.5	6.32	0.1
سيوى	81.5	73.5±1	8	0.11

الجدول رقم4. **تقدير نسبة المعادن الكبرى والصغرى**

العناصر	صعيدى	اسوانى	سيوى
العناصر الصغرى مليغرام/100غرام			
زنك	0.641	0.663	1.027
ماغنسيوم	0.589	0.676	0.676
الحديد	4.09	4.79	2.59
النحاس	0.806	0.688	0.576
العناصر الكبرى مليغرام/100غرام			
البوتاسيوم	528.1	627.8	765.7
الكالسيوم	75.3	119	84
منجنيز	56.7	61.5	62.4
الصوديوم	7.7	7.4	7.7