

Studies on Libyan Date-Syrup (Rub Al-Tamr)

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ABSTRACT

Representative samples of date-syrup were obtained from the local market and analysed. The chemical composition and some characteristics of these samples were investigated. The data showed that Libyan date-syrup was characterised by its valuable nutrients when being considered for direct edible purposes. The reducing sugars of the syrup comprised about 95% of its total-sugar content. The major sugars present were glucose—48.70%, fructose—45.21% and sucrose—6.09%. Furthermore, the colouring matters of date-syrup were isolated, separated and further investigated using sophisticated techniques. It was concluded that each of the colour groups, degradation products of reducing-sugars, melanoidines and iron-polyphenolic complexes contributed to the colour of date-syrup. The melanoidine-type compounds, which comprised the major part of syrup colourants showed a low selective adsorption tendency on both charcoal and anion resins. An efficient clarification of date-syrup has been achieved by the use of calcium phosphate precipitation. The results obtained may be of help in devising industrial processes for the utilization of dates, abundantly grown in Libya, in the production of 'total invert liquid sugar' with multiple commercial uses of its own.

INTRODUCTION

Since ancient times, dates have been considered one of the most important fruits of desert regions. There are about 90 million palm trees throughout the world, which yield about 1.25 million tons of dates annually (9,25). They are distributed in 22 countries, mostly in the desert regions of North Africa and south-western Asia.

According to the Ministry of Agriculture in Libyan Jamahiriya (2), there are more than 4 million palm trees in Libya which yield around 87,000 tons of dates. The present Libyan production represents about 5% of the total world production (2). There are many varieties of dates grown locally, but Bokrari, Khadhrari and Taasfiri constitute the main varieties that are grown in Libya (9). No statistical data is available concerning the individual production of each variety.

Date-syrup, locally known as Rub Al-Tamr, is a popular food commodity produced by a small plant located in Tripoli (capacity of 550 kg syrup/day) from Bokrari dates. In 1977 a modern date-syrup producing plant was established in El-Khoms, with an annual capacity of 8,000 tons of syrup. Considerable amounts of date-syrup are also produced domestically from some local date varieties.

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Owing to the absence of any sugar industry in Libya, large quantities of sugar are imported annually for domestic consumption. Simultaneously, there are some food industries in Libya that consume considerable quantities of sugar which is considered a subsidised commodity. Consequently, the abundance of dates grown in Libya and their high sugar-content should justify their use as a source of liquid sugar suited to many food industries. However, the large amount of colouring matter found in date-syrup may present a handicap to its direct use for this purpose.

The production of liquid sugar from dates has been investigated by many workers (4,10,19,20,21,26). Ehrenberg (10) described that treating date extract with lime, followed by purification with cation and anion exchangers, yielded a syrup with a purity of 99.26%. Nakahara *et al.* (19) reported on the use of ultra filtration membrane with >20,000 molecular weight to obtain a clear date syrup which can be used as a flavouring agent for processed foods. According to Nowatzyk (20) an excellent liquid sugar could be obtained from date extract by clarification with lime at pH 10.6 followed by desalting by ion exchangers and concentrating in vacuum. Wolf *et al.* (26) studied the effect of different types of ion-exchange resins on the decolorization of date juices. They concluded that the use of sulfonated divinylbenzenestyrene copolymer considerably improved the decolorization of date juice.

The aims of the experiments run in the present investigation are:

1. To evaluate the gross composition and study the principal physico-chemical properties of date-syrup produced in Libya. Such analyses would be of help in establishing standards and general specifications of the Libyan date-syrup known as 'Rub Al-Tamr'.
2. To obtain relevant information of properties and structure of the colour components of date-syrup.
3. To study the possibility of decolorizing date syrup to produce a liquid sugar from it.

MATERIALS AND METHODS

The samples of date-syrup used in this study were obtained at random from the local market. These samples were thoroughly mixed together and a representative portion taken and analyzed.

Methods of analysis

The following determinations were run in triplicate. The results were reported as a mean of the three runs. The physico-chemical properties measured for the date-syrup were: viscosity, refractive index, polarization, colour, pH and the indicator property. The UV-absorption spectra were obtained from aqueous solutions of the original date-syrup and its colorants. Infra-red analysis was carried out for the colouring matters isolated from date-syrup. Total soluble solids, acidity, total ash, potassium, sodium, calcium, and iron content were also determined. Amino acids of the date-syrup proteins were qualitatively determined by paper chromatography using Whatman paper No. 1 in descending system. The preparation of the hydrolysate, the solvent system employed, and the development of the chromatograms were done as described by Pescok *et al.* (24).

Date-syrup samples were analysed for total sugars and reducing sugars using the 'Lane-Eynon' volumetric method. The sucrose content was determined using three different techniques, namely, polarimetric, 'Lane-Eynon' volumetric method, before and after inversion, and thin layer chromatography (TLC). All the above mentioned determinations were carried out according to the methods described in (6,7,8,22,23,24)

employing: 'Abbe-refractometer; pH-meter' 'Bekman, Chem-Mat'; viscosimeter 'Rheomat 15 T'; polarimeter 'Karl Kolb, model 14.220'; flamephotometer 'Carl Zeiss-pf 5'; spectrophotometer 'Double beam, Coleman-124; and infra-red spectrophotometer 'Perkin-Elmer-567'.

The individual sugars, glucoses, fructose and sucrose were qualitatively and quantitatively determined using a combination of chromatographic and micro-colorimetric procedures (6,15), with some modifications to suit the material analysed. The technique employed herein was selected after carrying out a series of initial experiments. It is summarized as follows: thin layer chromatography (TLC) on plates containing 'Cellulose DF' was applied to separate the sugars. Samples consisting of 800 micrograms in solutions were spotted. The plates were then multiple developed according to the method described by Berger *et al.* (6). Pure sugars were chromatographed along with the date-syrup samples in order to identify the sugars present. The quantitative determination of the separated individual sugars was carried out as described by Hodge *et al.* (15).

Isolation of the colouring matter from date syrup

The date syrup, after dilution, was centrifuged at 5000 rpm for 15 minutes. The colouring matter was then adsorbed on a mixture of equal parts of animal charcoal and activated vegetable carbon. After filtration, the charcoal bed was washed with hot distilled water followed by another wash with ethyl alcohol. Then the adsorbed colourants were extracted from charcoal using 70% pyridine. The pyridine was evaporated under vacuum at 55°C. The moistened colouring matter remaining after evaporating was further dried in desiccator over H₂SO₄. After recording the weight of dried colouring matter, it was ground to powder and stored for the following analysis.

Ion exchange chromatography

The colouring matter was adsorbed from date-syrup on an anion exchange resin (Amberlite IRA-401-Cl⁻ form). For this purpose a column (4 × 45 cm) was prepared by the usual procedure (24). A diluted solution of date-syrup containing 15% refractometer solids was first centrifuged at 5,000 rpm for 15 minutes, then 20 ml of the supernatant was applied into the column. Chromatography was carried out with stepwise elution using H₂O, 0.3 M-NaCl, 0.5 M-HCl, 0.5 M-NaOH, and 2 M-NaCl. The flow rate was about 1 ml/min. Column effluents were collected in 10 ml portions. The optical density was measured for each portion at 560 nm. UV-absorption spectra were obtained for the fractions separated by column chromatography.

Decolorization of date syrup

A diluted solution of date syrup (20% T.S.) was heated to about 70°C, then treated with milk of lime (1%) and H₃PO₄ to a pH value of 7.5. The defecated solution was left for three hours to settle. The heavy precipitate yielded by this treatment was separated by decantation and filtration. The clarified date-extract was then boiled for 10 minutes with a mixture of vegetable and animal charcoal (0.3%) to remove the remaining colouring matter. After filtration, the purified date-extract was used for further analyses.

RESULTS AND DISCUSSION

1. Date-syrup characteristics and composition

Dates are generally rich in sugars and minerals. They are considered a good source for calories, mineral and amino acids (5,9,11,17,21,25). Data concerning the average

Table 1 Physico-chemical properties and average composition of the date-syrup.

Characteristic measured		Libyan Standard
Constant direct polarization at 20°C (angular degree)	-5.92°	
Invert polarization at 20°C (angular degree)	-6.75°	
Colour ^a	10.00	
Viscosity at 20°C (P)	17.17	
pH	3.80	
Refractive index at 20°C	1.4788	
Degree of acidity ^b	8.90	
Total soluble solids % (by refractometer)	75.80	> 70
Total sugars % ^c	70.81	> 68
Purity % ^d	93.42	
Protein % (N × 6.25)	1.02	
Total ash %	2.03	< 1.5
Na (mg/100 g)	70.40	
Ca (mg/100 g)	37.70	
K (mg/100 g)	217.00	
Fe (mg/100 g)	9.30	
Total colouring matters %	4.08	
Colouring matters adsorbed on charcoal %	1.86	
Colouring matters not adsorbed on charcoal %	2.22	

^aDetermined as attenuation index (8)

^bCalculated as milliliters of 0.1 N NaOH required to neutralize 10 g. of the date syrup.

^cCalculated as invert sugar.

^dCalculated as (total sugar/total soluble solids) × 100.

composition and principal characteristics of Libyan date-syrup are presented in Table 1. It is evident that optical activity of a syrup largely depends on the types and relative proportions of the sugars present. The date-syrup studied was levorotatory and it exhibited a slight difference in the polarization value after and before inversion. This may indicate a low sucrose content. The relatively high viscosity of the syrup; 17P, was undoubtedly affected by the presence of non-sugar materials present, such as proteins, pectins and dextrans. The acids present in the syrup contributed to its flavour complex. The high level of acidity pH 3.8, contributed to its stability against micro-organisms. More than twelve amino acids have been identified in dates (5,17). Dates appear to be the richest natural source of amino acids among fruits (17,25). Figure 1 illustrates the amino acids chromatogram of the Libyan date-syrup. The following amino acids could be identified: Argenin, glutamic acid, methionine, histidine, alanine, lysine and glycine. However, the presence of other amino acids was not examined. Data reported in Table 1 indicated that the date-syrup ranked high in its nutritive value. Beside sugars, proteins and minerals, especially calcium and iron were other constituents of significance when considering the date-syrup for direct consumption. It is worthy of notice that the analytical results obtained agreed with those recorded for date-syrup produced in other countries (4,10,19,20). Data also conformed with that mentioned in the Libyan standard specifications for local date-syrup (1). The only exception was that the date-syrup samples had a higher total ash content than that reported in the Libyan standard specifications.

The sugar analysis of date-syrup was accomplished employing the above mentioned three different techniques. The data obtained were reported in Tables 1 and 2. Figure 2 illustrated the sugar chromatogram of the Libyan date-syrup. It could be noticed that date-syrup sugars developed on the cellulose plate were resolved into discrete spots. Glucose, fructose and sucrose were identified. These findings agreed with the results reported by many investigators (3,11,17,20). The data of Tables 1 and 2 indicated that reducing-sugars comprised about 95% of the total sugars. The glucose

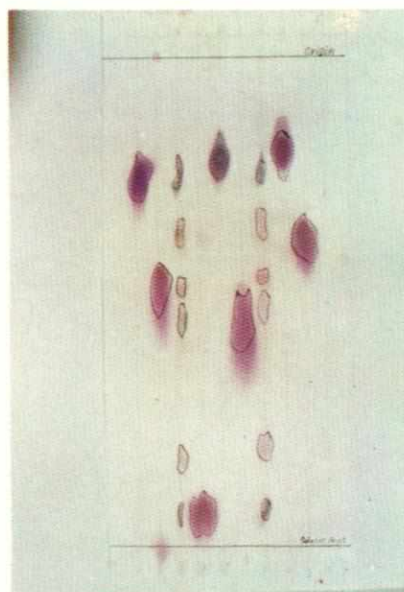


Fig. 1. Amino-acids content of 'Rub Al-Tamr'.

concentration slightly exceeded that of fructose. The purity of the date-syrup produced in Libya reached 93.42%. Such characteristics enable date-syrup to be used as less expensive substitute for glucose-syrup in those countries having a ready supply of dates. However, the high colour, reported in Table 1, necessitates of clarification of date-syrup before using as a liquid invert sugar.

2. Isolation, separation, and characterization of the colouring matter of date syrup

The method used for the isolation of the coloured components from date-syrup did not affect their chemical structure. It has to be mentioned that charcoal mixture adsorbed only a part of the date-syrup colourants—45.6%, while the remaining part was precipitated after the treatment of the charcoal-bed filtrate with ethyl alcohol. The alcohol precipitated colourants comprised about 54.4% of the total colouring substances of date-syrup. The powder of the colouring matter adsorbed on charcoal had a brownish-grey colour and an odour characteristic of dates. It was easily soluble in water at room temperature. The alcohol precipitated colourants were darker in

Table 2 Sugar analysis of the date-syrup.

Method of analysis	Sucrose %	Glucose %	Fructose %	Reducing sugars %
TLC	3.97	33.32	30.93	68.42*
Lane-Eynone				
Volumetric method	3.62	—	—	67.01
Polarimetric method	2.98	—	—	—

*Calculated as equivalent total invert sugar.

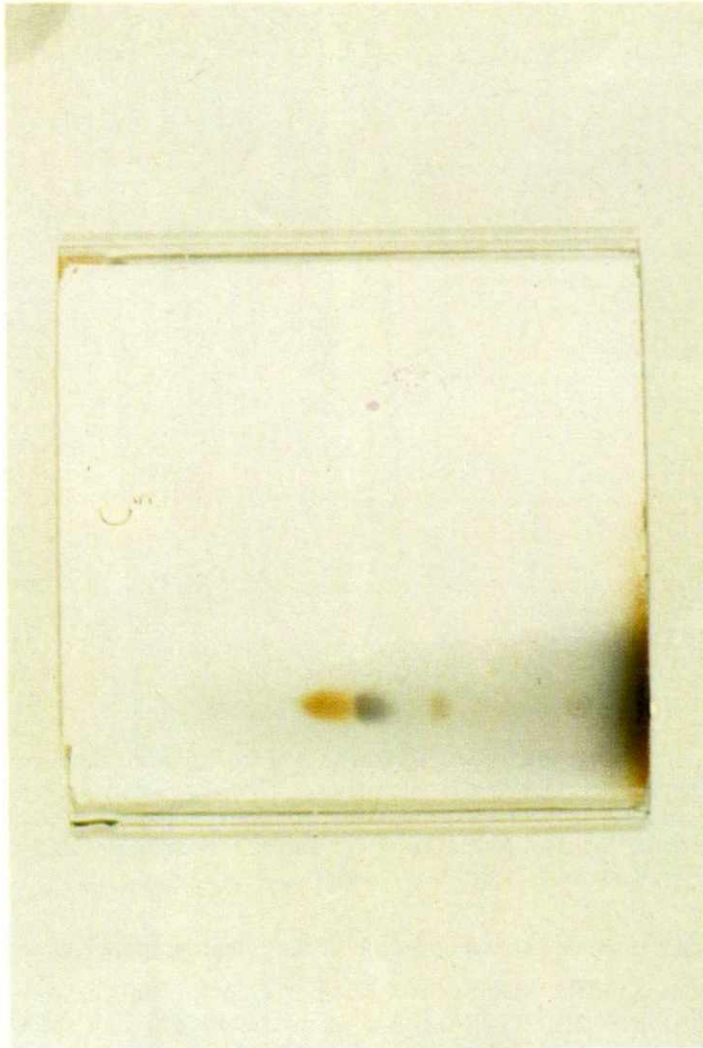


Fig. 2. Sugar's chromatogram of 'Rub Al-Tamr'.

colour, odourless, insoluble in water. Generally, date-syrup contained total colouring-matter as high as 4.08% (see Table 1).

It has long been recognized that UV-spectroscopic examination provided some valuable information on the principal functional groups and on classification of compounds (7,14,22,24). Figure 3 illustrates UV-absorption spectra for an original sample of date-syrup and for the filtrates after the treatment with charcoal and alcohol respectively. The extinction curve of the original syrup, curve A, exhibited a minimum absorption at 265 nm and a maximum absorption at 275 nm. The extinction curve of the adsorbed-on-charcoal colourants—Curve C—showed a sharp absorption peak at the wavelength of 295 nm, while the filtrates after treatment with charcoal and alcohol, curves B and D respectively, exhibited an intensive absorption at the range of

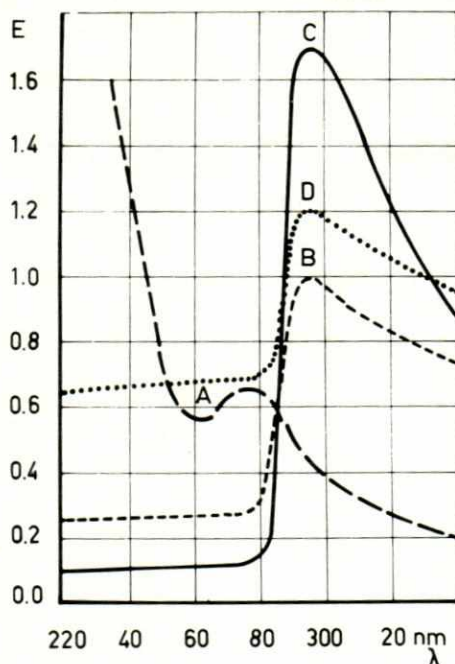


Fig. 3. UV-absorption spectra of: (A) The original samples of 'Rub Al-Tamr', (B) The filtrate after treatment with charcoal, (C) The colouring matters adsorbed on charcoal, (D) The filtrate after treatment with alcohol.

290–295 nm. This could be attributed to the existence of different groups of chromophores and auxochromes, which have intensive absorption in the preceding wavelength ranges of spectra (7,24). The wavelength range 270–275 nm, for example, may be associated with compounds of the type:

$R-CH = C(OH)-CHO$, which could be detected among the degradation products of reducing sugars. The UV-absorption band in the regions 285–295 nm has been recognized as indication of amino-carbonyl products known as melanoidines (7,12,13). Moreover, phenolic compounds present in ripe dates—(about 0.5%)— may form iron-polyphenolic coloured complexes not adsorbed on charcoal with characteristic absorbance at 272–273 nm (12,13,17,18). This may be an indication that each of the groups of degradation products of reducing sugars, melanoidines and iron-polyphenolic complexes contributed to the colour of date-syrup. However, the conditions prevailing during the processing of date-syrup, i.e. prolonged heating for a long time of solutions of reducing sugars in the presence of amino acids, phenolic compounds and metals, are considered favourable to the formation and accumulation of such type of compounds. It should be pointed out that a minor part of the charcoal-nonadsorbed colourants, probably of melanoidines type, was not precipitated with alcohol and appeared in the final filtrate giving it a very weak, grey hue (curve D in Fig. 3).

It is now well known that the concentration of hydrogen ions is considered to be one of the most important factors, which define the equilibrium of the coloured complexes (18,22,24). Figure 4 illustrates changes in the optical density depending on the pH values for the colouring matters under study. Noticeable differences could be seen in configuration and extreme points between curves A and B. This could be

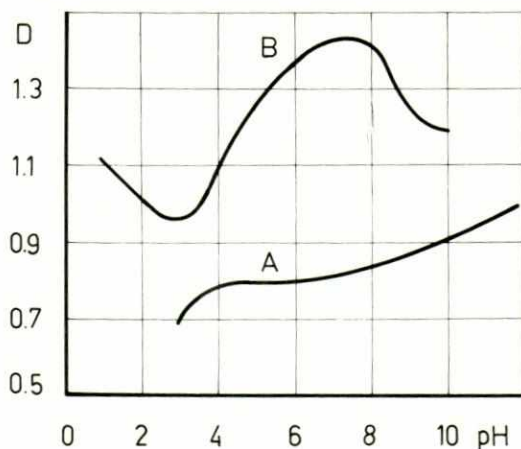


Fig. 4. The indicator property of the coloring matters of "Rub Al-Tamr". A—The coloring matters adsorbed on charcoal. B—The filtrate containing the coloring matters not adsorbed on charcoal.

regarded as an indication of the difference in the nature of the two types of colourants shown in this figure.

The infrared spectra of the colouring matters isolated from date-syrup were illustrated in Figure 5. The patterns of these colourants displayed at least sixteen significant bands for both of the two groups of colourants. The identification of the functional groups assigned to these bands was shown in Table 3.

Figure 6 illustrates UV-absorption spectra of the colour fractions of date-syrup separated by column chromatography. The date-syrup colourants were separated into two fractions: water fraction of brownish-red hue, and 2M-NaCl fraction of light yellowish-brown hue. The water fraction had an absorption peak at 295 nm, while NaCl fraction exhibited an intensive absorbance at the wavelength range of 305–315 nm. The latter band has usually been assigned to the absorption of melanoidine-type compounds resulting from amino-carbonyl reaction with the contribution of various amino acids (12,13,14,18,24). Regarding configuration and extreme points, a kind of similarity can be noticed between curve E (Fig. 6) and the curves B, C and D (Fig. 5). This may be due to the fact that the melanoidine-type compounds which comprise the major part of date-syrup colourants have quite low selective adsorption tendency on both charcoal and anion resins. Only melanoidines of highly complex structure, NaCl fraction, exhibited noticeable selective adsorption property on anion resins. The data obtained could confirm the complexity of the chemical nature of date-syrup colourants. Such data could be of help in devising processes for their reduction or elimination on industrial scale.

3. Decolorization of date-syrup

Besides sugars, date-extract contains different amounts of pectins, proteins, minerals and hemicellulose substances (4,5,21,25). Pectins, which are considered hydrophilic

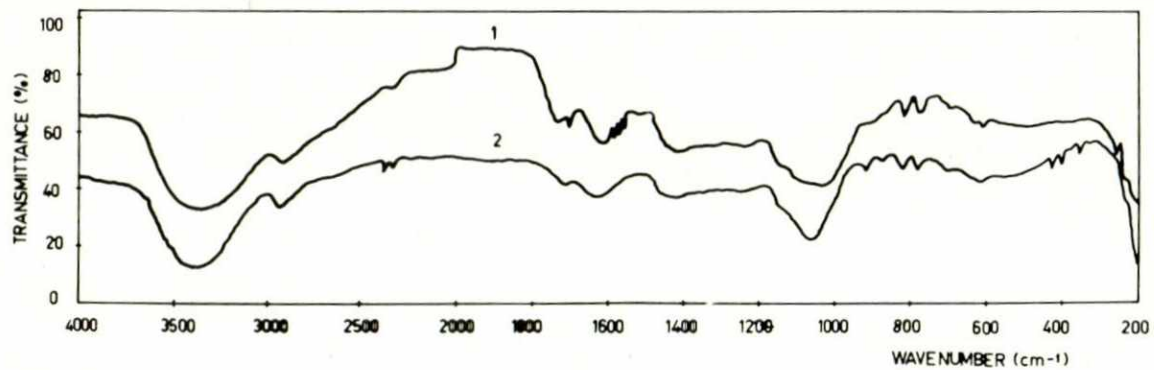
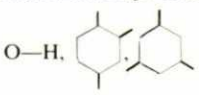
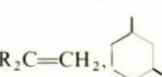
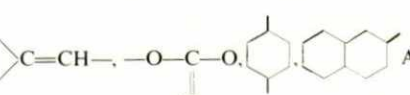
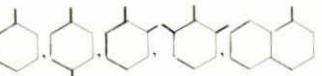
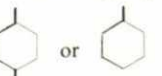




Fig. 5. Infrared spectra of: (1) Colouring matters of 'Rub Al-Tamr' not adsorbed on charcoal. (2) Colouring matters of 'Rub Al-Tamr' adsorbed on charcoal.

Table 3 Identification of the functional groups of the date-syrup colourants*

No.	Absorption band CM^{-1}	Characterization of band	Corresponding functional group	Colourant group**
1	3400	Very strong, very broad	$=\text{N}-\text{H}$, $> \text{C}-\text{H}$, $\text{HN}=\text{C}(\text{O}-\text{R})_2$	A and B
2	2950	Medium, sharp	$-\text{CH}_2-(\text{C}=\text{O})-\text{CH}_2-(\text{C}\equiv\text{N})$	A and B
3	2380	Weak, sharp	$-\text{C}\equiv\text{N}$	B
4	2340	Weak, sharp		B
5	2320	Weak, sharp		A
6	2020	Weak, broad	$=\text{C}=\text{C}$	A
7	1730	Weak, broad	$-\text{CH}_2-\text{CO}-\text{O}-\text{R}$, $\text{O}=\text{C}(\text{O}-\text{R})_2$	B
8	1725	Weak, sharp		
9	1710	Weak, sharp	$\text{CH}_2-\text{CO}-\text{O}-\text{R}$	A
10	1625	Medium, broad	$-\text{NH}_2$, phenyl ring	B
11	1610	Medium, broad	Phenyl ring	A
12	1580	Weak, very sharp	$\text{O}=\text{N}=\text{O}(\text{Nitro})$	A
13	1570	Weak, very sharp		A
14	1560	Weak, very sharp		A
15	1430	Weak, broad	Ionized carboxyl, alkene group	A
16	1425	Weak, broad		B
17	1060	Very strong, sharp	O-H, 	B
18	1050	Very strong, Very broad		A
19	925	Weak, sharp	} $\text{RCH}=\text{CH}_2$	B
20	910	Very weak, sharp		B
21	870	Weak, sharp	$\text{R}_2\text{C}=\text{CH}_2$, 	B
22	820	Medium, sharp	$\text{C}=\text{CH}-$, $-\text{O}-\text{C}(=\text{N})-\text{O}-$, 	A and D
23	780	Very weak, sharp	} 	A
24	775	Medium, sharp		Alkane group A
25	700	Very weak, sharp	 or 	A
26	630	Very weak, sharp	} Acetate or Acetyl	A
27	620	Very weak, sharp		B
28	610	Very weak, sharp		B
29	430	Medium, sharp	 , Ionic sulfate	B
30	400	Medium, sharp	Ionic sulfate	B

*The identification has been performed according to Creswell *et al.* (7) and Pecsok *et al.* (24).

**A—Colorants not adsorbed on charcoal, B—Colorants adsorbed on charcoal.

colloidal substances, cause remarkable increases in date-syrup viscosity. Their presence retards the rate of filtration and renders clarification of the date extract quite difficult. Proteins have also the same effect. It has been generally agreed by most of the investigators that the elimination of proteins and pectins needed specific materials

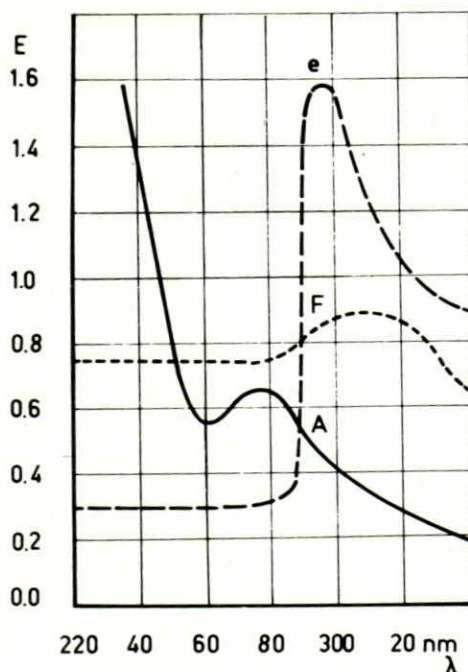


Fig. 6. UV-absorption spectra of: (a) The original sample of Rub Al-Tamr. (e) The H₂O-fraction from ion-exchange column. (f) NaCl-fraction from ion-exchange column.

having the ability to adsorb such colloidal matters (16,19). An efficient clarification of date-syrup could be achieved by employing calcium phosphate precipitation (10,20). In the present study an attempt was made to clarify date-syrup using this chemical treatment. The decolorizing efficiency was about 98%, the loss in soluble solids amounted to 2.7% and a decrease of 1.9% in ash content had been achieved along with decolorization. After decolorization, the purity of date extract increased to 99.3%. The data obtained may furnish verified basis for the use of dates grown in Libya in the production of a type of liquid sugar known as 'total invert liquid sugar', having important uses in food industries.

The results obtained in the present work lead to the following conclusions:

1. The date-syrup produced in Libya is characterized by its high nutritive value when considering for direct edible purposes. Its analytical data conformed with the Libyan standard specifications with the exception of the high total ash content.
2. The reducing-sugars of Libyan date-syrup comprised about 95% of its total sugars content. The major sugars present were glucose 48.70%, fructose 45.21% and sucrose 6.09% of the equivalent total invert sugars.
3. The Libyan syrup contained total colouring matter as high as 4.08%, only 45.6% of which could be adsorbed directly on charcoal. The remaining part has to be eliminated by an appropriate chemical treatment.
4. The ultraviolet spectroscopic examinations indicated that each of the following groups, degradation products of reducing-sugars, melanoidins and iron-polyphenolic complexes contributed to the colour complex of date-syrup.

5. The melanoidine-type compounds, which comprise the major part of date-syrup colourants showed a low selective adsorption tendency on both, charcoal and anion resins. Only melanoidines of highly complex structure exhibited noticeable selective absorption property on anion resins.
6. An efficient clarification of date-syrup has been achieved by the use of calcium phosphate precipitation. The results obtained may be of help in setting some practical basis for utilizing the dates grown in Libya in the production of 'total invert liquid sugar', which has numerous uses in food industry.

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دراسات على شراب التمر المنتج في ليبيا " رب التمر "

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المستخلص

تم دراسة الصفات الفيزيوكيماوية وتركيب شراب التمر المنتج في ليبيا والمعروف باسم " رب التمر " . ولقد أوضحت نتائج الدراسة احتواء هذا المنتج الغذائي على عناصر ذات قيمة غذائية عالية للمستهلك . الى جانب السكريات والبروتينات يحتوي " رب التمر " على أملاح هامة كالحديد والكالسيوم والصوديوم والپوتاسيوم . أثبتت التجارب أن السكريات الرئيسية الموجودة هي الجلوكوز 70.48% ، الفركتوز 21.45% ، السكروز 9.67% .

ولقد أمكن فصل المواد الملونة لرب التمر وتجزئتها والتعرف على خواصها وذلك باستخدام طرق التحليل الحديثة . ولقد دلت النتائج على أن اللون الداكن " لرب التمر " ينتج من تواجد مركبات من المجاميع التالية : نواتج تحليل السكريات المختزله - الميلائويدين - المركبات المعقدة من المواد الفينولية مع الحديد . أثبتت الابحاث أن مركبات الميلائويدين التي تكون الجزء الاعظم من ملونات رب التمر لها خاصية ادمصاصيه ضعيفة على الفحم المنشط وعلى الراتنجات السالبة .

تم في هذه الدراسة التوصل الى طريقة فعالة في ازالة ألوان رب التمر وذلك باستخدام راسب فوسفات الكالسيوم وتعديل الاس ايدرجيني للشراب . وتعتبر النتائج المتحصل عليها ذات أهمية تطبيقية خاصة عند وضع الاسس العملية لاستخدام التمور المتوفرة محليا في صناعة نوع من الشراب السكرى هو " الشراب السكرى المحول كليا " ذات الاهمية الصناعية في انتاج بعض الأغذية المصنعة مثل الحلوى والمربيات والفواكه المعلبة والمشروبات الغازية ومنتجات المثلوجات وغيرها .