Detection of the amount of phenolic compounds and mineral elements in the residues of date molasses (kernels and pith of Zuhdi dates)

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ABSTRACT

The study included the study of the chemical components of date molasses residues (the pith and seeds of Zuhdi dates) extracted by the cold method, and the preparation of aqueous and alcoholic extracts (ethanolic and methanolic) for each of Nawiya, Bethel and Al-Zuhdi, and studied the chemical content of Bethel powder and date seeds. The total content of phenols and flavonoids was estimated for the aqueous and alcoholic extracts. And then the minerals were measured in both The two powders and the active compounds in these aqueous and alcoholic extracts were identified by an apparatus HPLC measurementThe results of the mineral elements for each of the powder of the kernels and the pits of Al-Zuhdi dates showed the mineral elements, as the kernel powder contained (209.60, 51.63, 29.02, 1.36, 0.74, 61.68, 0.94 (0.21 mg/100gm) of potassium, magnesium, calcium, iron, manganese, selenium, zinc, phosphorus and copper, respectively, while the content of the Zuhdi dates was of mineral elements. In the same order, respectively (145.01, 1.0968, 15.02, 0.6089, 0.1618, 54.843, 0.22, 234.6, 0.4609)of nuclei and pith of ascetic dates, as the results showed that there were significant differences between the aqueous and alcoholic extracts at the level of potential($P<0.05$) The ethanolic extract was superior in its content of flavonoids, as the content of the ethanolic extract of the nuclei and pith of ascetic dates was the highest concentration of flavonoids, as it reached It reached (50.11, 52.16) .mg rutin/ml, followed by the methanolic extract (35.01, 46.16) mg rutin/ml. Respectively, followed by the aqueous extract with a content of (33.32, 27.00) mg rutin/ml for each of three Al-Zahdi date pits, respectively.The aqueous extract of date seeds contains the following phenols (Pyrogallol, Gallic acid, Rytin, Kaempferol, Cinnamic, Catechol, 4-hydroxy benzoic, Cinnamaldehyde, Eugenol, Lignan, Chlorogenic, Qurctin) At concentrations of (0.05, 0.01, 0.09, 0.26, 9.920, 20.55, 34.08, 21.29, 1.94, 1.823, 10.87, 5.359) ppm, respectively, the highest concentration of phenols identified in the aqueous extract of nuclei Dates are (4-hydroxybenzoic, as its concentration was 34.08 ppm). As for the alcoholic extract of date pits, it contains the same compounds with concentrations (0.124, 0.04, 0.787, 0.304, 21.69, 22.97, 94.44, 26.435, 4.412, 0.966, 10.97, 6.655) ppm. Respectively, the highest concentration of phenols identified in the alcoholic extract of date seeds was (4-hydroxy benzoic, with a concentration of 94.44 ppm). (0.16, 0.019, 0.45, 0.58, 34.83, 157.27, 83.18, 4.467, 0.225, 4.37, 2.219, 122.87) ppm respectively The highest concentration of phenols identified in the aqueous extract of ascetic date pits was for the phenolic compound (Catechol), which was (157.27) ppm. While the concentrations of phenolic compounds diagnosed by HPLC technology in the alcoholic extract of ascetic date bark were(0.14, 0.05, 0.28, 0.60, 50.60, 27.88, 150.99, 3.49, 0.33, 6.88, 1.99, 24.17) ppm Respectively, the highest concentration of phenols identified in the alcoholic extract of ascetic date pith was for the phenolic compound (l4-hydroxybenzoic), whose concentration was (150.99) ppm.

Key words: Date molasses waste- zuhdi - Like dates
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INTRODUCTION

Date palm cultivation has been historically associated with the Arab world, and its original home is considered, especially the Shatt al-Arab, the head of the Arabian Gulf, and from there it moved to all areas with a suitable climate for its cultivation (1). The date palm is of great economic importance because the fruits give it great nutritional importance. And it contributes to the national income(46). The Zuhdi variety is one of the semi-dry dates, and it is a well-known commercial variety that ranks first in terms of number and quantity of production, due to its tolerance to various types of soils. It is often exported in khasaf or bags, and part of it is in wooden boxes (9). The Iraqi Zuhdi dates are among the largest ethnic varieties in production and are consumed in two parts. Direct consumption, while the second goes to industries based on various dates, including liquid sugar, molasses, alcohol, vinegar, and yeast (18). Iraq enjoys an important position in the production of dates among the producing countries of the world in terms of the number of palm trees, and the Zuhdi variety is at the forefront, which constitutes 70% of the total production of dates, in addition to the economic importance of palm trees and their products (dates). Dates are used as fodder, food and fuel (29). The growth of the fruit and the stages of its ripening go through five different stages: Al-Hababuk, Al-Jamri, Al-Khalal, Al-Rutab, and Al-Tamr, depending on the color, softness, moisture, and sugar content. The fruit consists of two main parts, the fleshy part It is the part that is eaten and represents 85-87%, and the seeds, which represent 13-15 of the weight of the date. The diet part contains high sugars 44-88%, fat 0.2-0.5%, protein 2.3-2.19% and 15 types of mineral elements, vitamins and Fiber 6.4-11.5% (39). The kernels represent the solid part that is not optimally exploited, and it is a waste from the production of many industries based on the technical conversion of dates. Large quantities of kernels can be collected from date factories or from production waste. Several researches have been conducted on date kernels and their chemical composition, and they were as follows (moisture 5 -10%, protein 5-7%, oils 7-10%, , ash %65-55crude fiber 10-20%, carbohydrates 1-2%) and its proteins contain many amino acids such as (glutamic, aspartic acid, arginine), which represent the basic amino acids and also contain acids (isoleucine, lysine, leucine) and good proportions of the minerals P, Ca, Mg, K and a few from , Zn, Mn, Fe, Na Due to the nutritional components possessed by date stones, they can be included in the nutrition of both animals and humans. Date seeds oils can be used in many food industries such as mayonnaise and cream, as well as in the pharmaceutical industries such as cosmetics (Cosmetics) and medical preparations (2). As pointed out (36) to the use of date stones in several fields, as it was used as fuel in traditional copper bleaching furnaces. The seeds are also used as animal fodder after crushing them or soaking them with water, as they are a rich source of carbohydrates, fat and protein. Some poor peoples use them as food, as they cook them as they cook legumes to take advantage of their high content of basic food ingredients, in addition to their fiber content. Which protects the body from the symptoms of indigestion, constipation and colon, and the oil extracted from it can be used as an ointment to treat rheumatism, gout and joint pain, and the crushed seeds mixed with rose water were used to treat the eyes and used after softening it as a substitute for kohl (source). The oil extracted from the kernels can be used directly in the manufacture of soap, after roasting and then

grinding them Boil it with water (source). Recent studies have found that date pits have high levels of phenolic compounds, including flavonoids, which act as antioxidants, and dietary fiber, higher than those found in the fleshy part. There are high levels of α-Tocopherol, Ascorbic acid, and Glutathione. Among the multiple phenolic acids is Sinapic. acid, Caffeic acid with amounts of Protocatechenic acid (44). Many aromatic compounds were also identified in date seeds, including alcohols, esters, Aldehydes, terpenes, ketones, saturated hydrocarbons and unsaturated hydrocarbons (9) There is a study on the phenolic content, flavonoids content and antioxidant activity of date pits extracts. As for the waste of date molasses, it is a third of dates rich in fiber, if the ratio reaches between (8.1 to 15.7), and the most important of these fibers are pectin, cellulose, semi-cellulose and lignin (10), and the percentage of fiber also depends on the date variety, so that Fully ripe soft dates contain less than 2% of fiber, and one of its benefits is that it resists constipation and fats that may block the coronary artery (25).

The date palm occupies the main rank in the diets of ruminant animals because it contains a high percentage of fiber, protein, carbohydrates, fats and a large part of the minerals. It also contains effective compounds and substances of nutritional value and can be used as medical and nutritional preparations. The chemical composition of the date palm contains moisture 19.99-17.8%. Protein 11.7-13.33%, carbohydrates 10.92-14.5%, ash 12.24%, fat 2%. It was also noted that the dietary fiber extracted from date pits is rich in phenols (21), and (39) stated that the content of date pits of phenolic compounds ranges from (21.0-62.0) mg gallic acid/100g(33).

MATERIALS AND METHODS

Determination of the mineral elements of the kernels, pith and oil of Al-Zuhdi dates by means of anatomic absorption spectrometer and an optical flame

Estimate the content of mineral elements in a sample of date seed oil, pith powder and pits of Zuhdi dates By atomic absorption spectrometer and optical flame at Ibn Sina State Company / Ministry of Industry And minerals / Republic of Iraq Samples of date nuts, date palm extract and extracted oil were digested And by using the method of Pearson, 1976 (42) to estimate the elements, if 1 gm of The oil sample was placed in a digestion flask and 10 ml of concentrated nitric acid was added to it and heated At a temperature ranging between (250_300)ºC for 30 minutes, after that it is left to cool, then 5 ml is added to it. of concentrated sulfuric acid and complete the digestion at the same temperature until it becomes a clear color with Taking into account that the sample did not dry out, and when the color of the sample changed to black, a few drops of liquid were added Concentrated nitric acid, then complete the digestion until the foam ended and the color became clear, then the volume was completed To 25 ml using deionized water, then the sample is ready for the determination of mineral elements.

2. Preparation of Date Seeds Extracts Water Extracts

I followed the method(**10**) with a weight of 100 gm of kernels powder and phthalates Dates and 500 ml of distilled water were added to it, leaving the beaker in the shaking incubator Shaking incubator for 24 hours at a temperature of 25°C, then centrifuged Centrifuge Xg for 10 minutes, then filter the clear with Whatman No.1 filter paper. The filtrate was concentrated by an evaporator Rotary Vacuum Evaporator until a thick liquid is obtained, then placed in the incubator at 37 °C for 48 hours to obtain

The dry powder of the extract, then the percentage of the yield was calculated through the following equation:

Dry extract = quotient %
100model weight× /weight

Then the extract was placed in tightly closed and opaque containers and kept in the refrigerator at a temperature of 4 °C until use.

3. Alcoholic Extracts

The ethanolic alcoholic extracts were prepared by weighing 100 gm of kernel powder and date parsley. It was dissolved in 500 ml of 70% ethyl alcohol, mixed well, and left for 24 hours at laboratory temperature. (25-30) Cº, filter the extract using Whatman No.1 filter paper, then concentrate the filtrate Rotary vacuum evaporator at a temperature of 40 ° C and leave the filtrate at the laboratory temperature until obtaining On a dry substance, it was kept in dark, airtight containers and kept in the refrigerator until use(19,53).

5 Detection of active substances in bitter gourd and dates

The active compounds in the plant extracts used in the study were identified using a device HPLC high performance liquid chromatography. Samples are injected into the apparatus and defined Paeks curves based on the database. Estimation of the total content of flavonoidsThe method of (51,52) was followed, as 1 g of date seed extract was dissolved in 1.5 ml ethanol and an equal volume of aluminum chloride (AlCl3.6H2O) was added (2%) It was prepared in 100 ml of methanol with a concentration of 70%, the mixture was shaken well, and after 10 minutes the absorbance was measured at a wavelength of 415 nm.

Standard curve

Concentrations of the flavonoid compound Rutin (10-150 mg / ml) were prepared, then the amount of flavonoids in the extract was estimated based on the graphical relationship between the concentration of the standard solution of Rutin and the absorbance at 415 nm wavelength, Figure (2-3).

The concentration of the rutin compound is mg/MlThe standard curve of the rutin complex

RESULTS AND DISCUSSION

Mineral elements of seed and pith of Zuhdi dates

The results shown in the table (3-1) the mineral elements of each of the powdered kernels and the third of Al-Zuhdi dates, as the kernels powder contained (209.60, 51.63, 29.02, 1.36, 0.74 (0.21, 0.94, 61.68 mg/100gm) of potassium, magnesium, calcium, iron, manganese, selenium, zinc, phosphorus and copper respectively, while the content of zahdi dates was of Metallic elements in the same order, respectively (145.01, 1.0968, 15.02, 0.6089, 0.1618, 54.843, 0.22, 234.6, 0.4609) The content of the mineral elements of the date seeds powder was close to what was mentioned by **(23)**, where the researcher indicated that the content of the date seeds of sodium ranged between 7-15 mg/100g and potassium between 175-240 mg/100g. Calcium between 13-34 mg/100g, Iron between 1.3-5 mg/100g, Copper between 0.1-0.6 mg/100g, Magnesium between 58-89 mg/100g, Zinc between 1-1.6 mg/100g and Phosphorus. Between 110-146 mg/100g and manganese between 0.6-1.3 mg/100g. The results were similar to what was found by **(35)** when studying the content of the kernels of the salvation variety. Of the mineral elements, it contained (16, 18, 248, 1.9, 0.5, 125, 81, 0.1, 0.7) mg/100g of the mineral elements sodium, calcium, potassium, iron, copper, phosphorus, magnesium, zinc and manganese, respectively. The results were similar to what was found by **(22)** when studying the content of mineral

elements of the date seeds of the Deglet Nour and Allig cultivars, as the seeds of the Deglet Noor variety contained (229, 51, 38, 68, 10, 2) mg/100 gm of potassium, magnesium, calcium, phosphorus and sodium. and iron, respectively, while the content of the nuclei of the Allig variety of metallic elements was in the same order (293, 58, 28, 83, 10, 2) mg/100g. While the results were different from what was found by **(15)** when studying the content of the nuclei of the two cultivars of Dukla-Nuru Allig of mineral elements, as it was stated that The content of the two varieties of potassium was 0.238 and 0.289 mg/100g, respectively, of magnesium 0.048 and 0.048 mg/100g in the same order, of calcium 0.034 and 0.026 mg/100g, and of phosphorus 0.058 and 0.07 mg/100g. The results indicate a clear decrease of the mineral elements in the peat of Zuhdi dates, and this is due to the fact that most of the mineral elements are soluble in water, which is excluded during the process of preparing molasses by the pressing process. The reason for the high concentration of phosphorus in the protein center is due to its association with protein and its high concentration, i.e. the date seed proteins may be of the type of phosphoproteins.

Table (3-4) Mineral elements content of Al-Zuhdi date kernels and pittal powder extracted from it mg/100g

potassium	magnesium	Calcium	iron	manganese	selenium	zinc	phosphorous	copper	Contents
249.60	63.51	39.02	2.36	0.92	61.68	1.04	252.3	0.43	Date seed powder
145.01	1.0968	15.02	0.6089	0.1618	54.843	0.22	234.6	0.4609	Dates powder

Total Flavonoids Content

Table (3-2) shows the content of flavonoid compounds in the extracts of the seeds and pith of ascetic dates, as the results showed that there were significant differences between the aqueous and alcoholic extracts at the level of potential (P<0.05)The ethanolic extract was superior in its content of flavonoid compounds, as the content of the ethanolic extract of the kernels and piths of ascetic dates was the highest concentration of flavonoids, reaching (50.11, 52.16) mg rutin / ml. Then, it was followed by the methanolic extract of the pits and pits of Zuhdi dates, with a content of (35.01, 46.16) mg rutin/ml, respectively, and then followed by the aqueous extract with a content of (33.32, (27.00 mg rutin/ml) for each of the pits of Zuhdi dates, respectively, and the result was less than what was found To him **(9)** when studying three varieties of Moroccan dates, it was found that the content of flavonoid compounds increased to 1844 mg rutin equivalent / 100 gm in the seeds of Al Boustahami dates. Bousthammi, while the lowest amount of flavonoids in Boufgous date kernels was 1224 mg rutin equivalent / 100 g, and the results were among the findings of (29,56). It was found that the amount of

flavonoids in the ethanolic extract of date pits was 45.28 mg/100g, and that the main reason for the large diversity in the amount of flavonoids in date pits was due to To many reasons, including growth conditions, stage of maturity, geographical location, fertilization method, types of soil diseases, storage conditions, as well as the system used in extraction (47).

Active compounds in the extracts of the pith and pits of ascetic dates The results showed in Table No. (4-6) that the aqueous extract of date seeds contained phenols (Pyrogallol, Gallic acid, Rytin, Kaempferol, Cinnamic, Catechol, 4-hydroxy benzoic, Cinnamaldehyde, Eugenol, Lignan, Chlorogenic, Qurctin) at concentrations of (0.05, 0.01, 0.09, 0.26, 9.920). , 20.55,34.08, 21.29, 1.94, 1.823, 10.87, 5.359) ppm respectively and a holding time per minute (2.872, 3.688, 4.280, 5.364, 7.704, 8.348, 9.892, 10.544, 13.856, 16.792, 17.048, 10.1) Respectively, the highest concentration of the identified phenols in the aqueous extract of date seeds was (4-hydroxybenzoic), with a concentration of 34.08 ppm. The results in Table (4-6) also showed the alcoholic extract of dates nuclei containing the following phenolic compounds (Pyrogallol, Gallic Acid, Rytin, Kaempfeol, Cinnamic, Catechol, 4-Heydroxy Beenzoic, Cinnamaldehyde, Eugenol, Lignan, Chlorogenic, Q. URCTIN) and Brakses (0.124, 0.04, 0.787, 0.304, 21.69،6.655, 94.44, 22.97, 26.435, 4.412, 0.966, 10.97, 6.655) ppm Sequence and detention time per minute (2.90, 3.82, 4.25, 5.24, 8.09, 8.48, 9.98, 10.47, 13.91, 16.85, 17.1, 10.1), respectively, as the highest concentration of phenols identified in the alcoholic extract of date seeds was (4-hydroxybenzoic, with a concentration of 9 4.44)) ppm Whereas the results showed in Table (4-7) the concentrations of phenolic compounds diagnosed above by HPLC technology and their retention time in the aqueous extract of ascetic dates (0.16, 0.019, 0.45, 0.58, 34.83, 157.27, 83.18, 4.467, 0.225, 4.37, 2.219, 122. 87) ppm on In succession, the retention time for these phenolic compounds per minute was as follows (2.35, 3.88, 4.01, 5.23, 8.25, 8.35, 9.82, 10.38, 13.72, 16.97, 17.16, 10.02), respectively. (Catechol), which was (157.27) ppm, while the concentrations of phenolic compounds diagnosed by HPLC technology and their retention time in the alcoholic extract of ascetic date palm were (0.14, 0.05, 0.28, 0.60, 50.60, 27.88, 150.99, 3.49, 0.33, 6.88, 1.99,24.17) ppm, respectively, and the retention time for these phenolic compounds per minute was as follows (2.80, 3.57, 4.24, 5.32, 8.08, 8.43, 9.98, 10.40, 13.82, 16.84, 17.14, 10.09). Respectively, the highest concentration of the phenols identified in the alcoholic extract of the ascetic date pith was for the phenolic compound (l4-hydroxy benzoic), whose concentration was (150.99) ppm, and these results came Consistent with what was obtained by the researcher (38,54), who found the highest concentration of phenolic compounds when studying the powder of ascetic date kernels, it is for the phenolic compound (4-hydroxy) benzoic at a concentration of 213.871 ppm and the phenolic compound (chlorogenic) at a concentration of 273.208 ppm, while the results were inconsistent with what was obtained by (11,55) who found that Rutin represents the most abundant flavonoid in date seeds, with a concentration ranging from71.74 to 86.32 mg/100 gm, followed by quercetin with a concentration ranging from (23.71-34.06 mg/100 gm) and luteolin with a concentration ranging from (9.17-13.24) mg/100 gm. Results can be explained by various factors such as variety, growth condition, maturity, season, geographic origin, Soil type, storage conditions,

amount of sunlight received, culture methods, stability conditions, the use of different analytical methods, the use of different methods. technical standards.

Table (4-4) shows the total content of flavonoid compounds for extracts of drupes and pits of date

methanolic extract	ethanolic extract	aqueous extract	Subject
mg/mL 46.16	mg/mL 52.16	mg/mL33.32	Zuhdi date seeds
mg/mL 35.01	mg/mL 50.11	mg/mL 27.00	Like the ascetic dates

Table (4-5) shows the retention time (RT) of standard phenolic compounds in the HPLC apparatus

liquid concentration	time of detention	compound name
20 PPM	2.782	Pyrogallol
-	3.607	Gallic acid
-	4.032	Rutin
-	5.448	Kaempferol
-	8.00	Cinnamic
-	8.300	Catechol
-	9.953	4-hydroxy benzoic
-	10.403	Cinnamaldehyde
-	13.890	Eugenol

-	16.740	Lignan
-	17.083	Chlorogenic
-	10.092	Qurctin

Table (4-6) shows the retention time (RT) and concentration of phenolic compounds in date seeds of the aqueous and ethanolic extracts

Zuhdi date seeds				compound name
aqueous extract (ppm)	holding time (in minutes)	alcoholicextract (ppm)	holding time (in minutes)	
0.057979	2.872	0.1248912	2.904	Pyrogallol
0.019961	3.688	0.0423450	3.820	Gallic acid
0.095722	4.280	0.787944	4.252	Rytin
0.267710	5.364	0.304440	5.244	Kaempferol
9.920450	7.704	21.69101	8.096	Cinnamic
20.55077	8.348	22.97880	8.484	Catechol
34.08260	9.892	94.44707	9.980	4-hydroxy benzoic
21.29020	10.544	26.43577	10.472	Cinnamaldehyde
1.946334	13.856	4.412055	13.912	Eugenol
1.823129	16.792	0.966957	16.852	Lignan
10.8778	17.048	10.97044	17.104	Chlorogenic

5.359750	10.100	6.655230	10.144	Qurctin

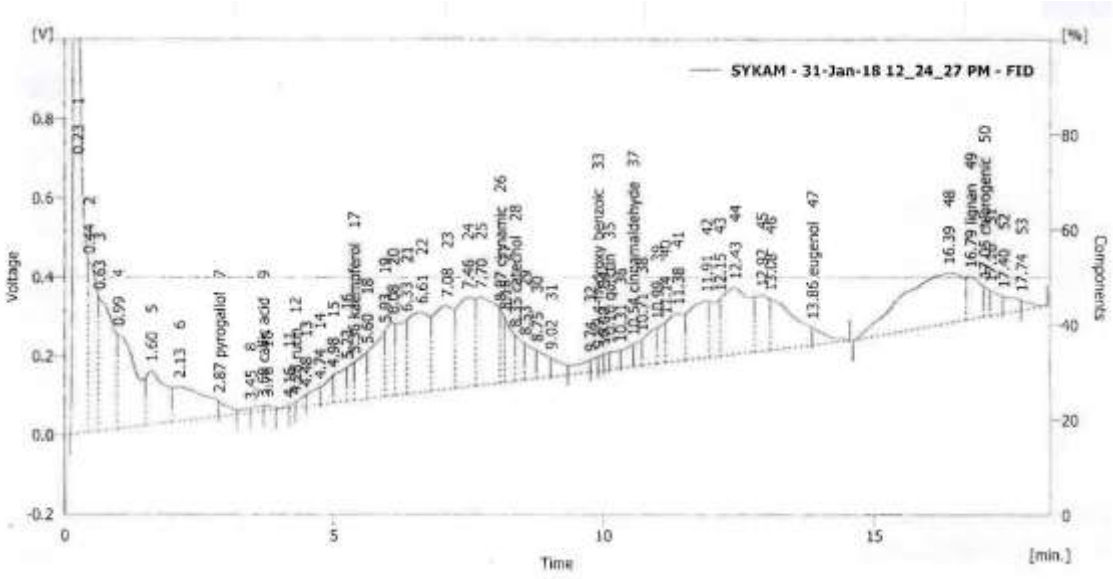

Figure (1-4) shows the active compounds in the aqueous extract of ascetic date seeds, which were measured by HPLC

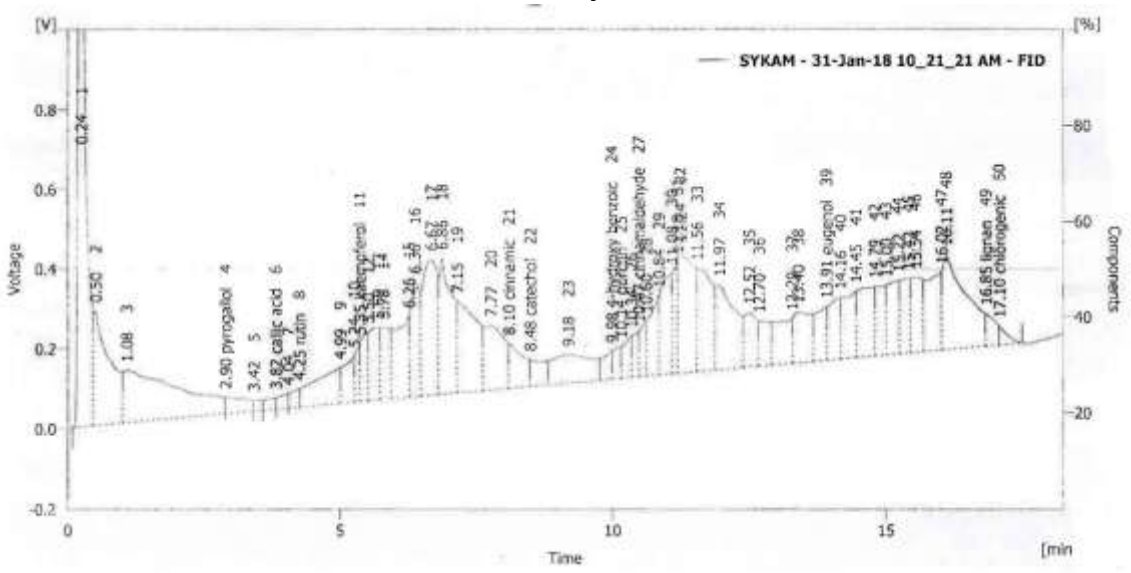

Figure (2-4) shows the active compounds in the alcoholic extract of ascetic date pits, which were measured by HPLC

Table (4-7) shows the retention time (RT) and concentration of phenolic compounds in the date bar of the aqueous and ethanolic extracts

Like the ascetic dates				compound name
aqueous extract (ppm)	holding time (in minutes)	alcoholic extract (ppm) (ppm)	holding time (in minutes)	
0.16050	2.356	0.146514	2.808	Pyrogallol
0.019092	3.888	0.05460	3.572	Gallic acid
0.457610	4.016	0.28223	4.240	Rytin
0.583000	5.236	0.60518	5.328	Kaempferol
34.83502	8.256	50.6011	8.080	Cinnamic
157.2722	8.352	27.8833	8.436	Catechol
83.18477	9.824	150.994	9.984	4-hydroxy benzoic
4.467101	10.388	3.491970	10.404	Cinnamaldehyde
0.225880	13.720	0.332422	13.824	Eugenol
4.374488	16.976	6.882500	16.848	Lignan
2.219636	17.164	1.992057	17.140	Chlorogenic
122.8750	10.072	24.17661	10.096	Qurctin

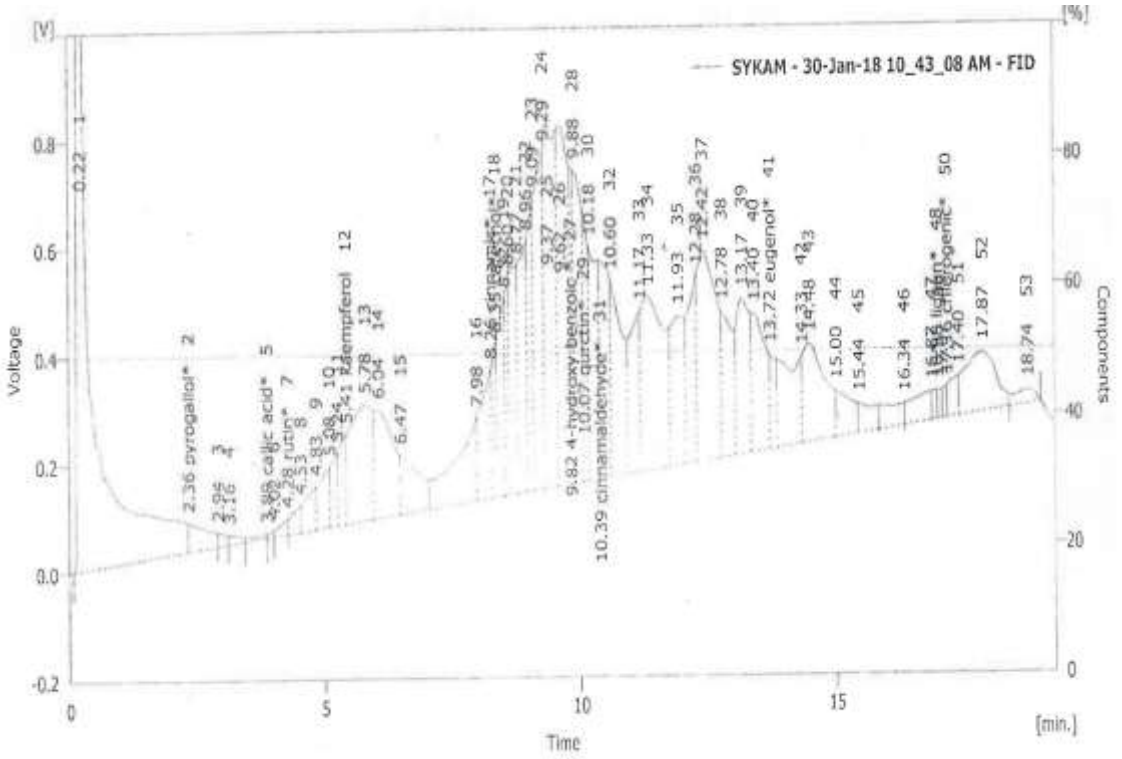

Figure (3-4) shows the active compounds in the aqueous extract of Zuhdi dates, which were measured by the HPLC device

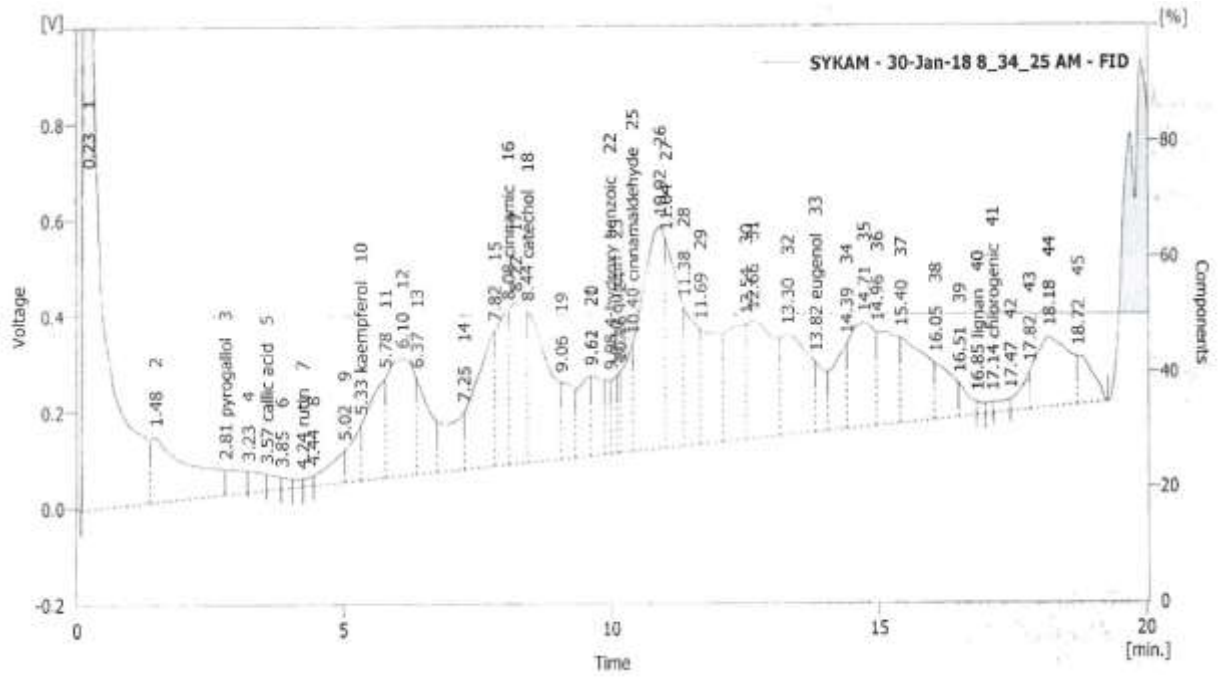

Figure (4-4) shows the active compounds in the alcoholic extract of Al-Zahdi Dates, which were measured by the HPLC device.

Moisture content

Figure (17) represent the effect of treatment with ethyl formate on the varieties of dates (Zuhdi, Al-Sayer, and Barben) during the storage periods on the percentage of moisture, where we find a high percentage of moisture in general for all varieties during the storage periods. The Barben variety sample, which is considered one of the soft dates, had the highest value of moisture percentage in the pre-storage period for the control sample and the

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