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Comparison of Solvent Mixing in the Extraction Process of Active Compounds in Dates using GCMS

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Abstract:- In a previous study we used three solvents, each one separately: methanol, chloroform and hexane. The results showed that each solvent showed different results, whether in the compounds or in the ratio of the compounds if they were similar. Therefore, in this study we mixed the three solvents in a ratio of mixture of three solvents (33.3 %: 33.3 %: 33.3 %) for each one. The results showed different products that included all the compounds that were extracted previously when we used each compound separately.

Keywords*: Dates, Mixture design, BSTFA, TMCS, GC-MS, LC-TOF-MS, Biochemical Compounds.*

1. Introduction

Dates are a rich source of at least six vitamins, including vitamin C, vitamin B1 (thiamine), B2 (riboflavin), niacin (nicotinic acid), and vitamin A, as noted by Ahmed et al. (1995). Regular consumption of fruits and vegetables has been linked to a reduced risk of various chronic diseases, as demonstrated by Nicoli et al. (1999). This is largely due to the presence of phytochemicals such as dietary fiber, phenolics, natural antioxidants, and other bioactive compounds. Antioxidants, which can delay or inhibit the oxidation of lipids or other molecules by preventing the initiation or propagation of oxidative reactions, play a key role in this protective effect (Velioglu et al., 1998). Phenolic compounds, including flavonoids (Pietta, 1998), phenolic acids, and phenolic diterpenes (Shahidi et al., 1992), are largely responsible for these properties. Their antioxidant activity arises from their redox behavior, enabling them to neutralize free radicals, quench reactive oxygen species, or decompose peroxides (Osawa et al., 2003). The antioxidant properties of these phytochemicals are associated with a lower incidence and mortality rate of cancer. Date fruits specifically contain phenolic compounds (mainly cinnamic acids) and flavonoids (such as flavones, flavonols, and flavanones) that contribute to their antioxidant activity, as reported by Velioglu et al. (1998), Vayalil (2002), and Mansouri et al. (2005). Al-Farsi et al. (2005) investigated the compositional and sensory characteristics of three sun-dried date varieties native to Oman, alongside their antioxidant activity, anthocyanins, carotenoids, and phenolics. They emphasized that increased research into the health-promoting components of dates would boost the recognition of their role in functional foods and nutraceuticals. Furthermore, Iranian dates have undergone analysis to determine their antioxidant capacity using assays such as ABTS (Miller et al., 1993). **Chemical Materials Methanol,** chloroform, and hexane—purchased from Merck KgaA, Germany—were utilized as GC-grade solvents. The reagents BSTFA (N,O-bis(trimethylsilyl)trifluoroacetamide) and 1% TMCS (trimethylchlorosilane) were obtained from Sigma-Aldrich.

Instruments

The gas chromatography-mass spectrometry (GC-MS) analysis was conducted using an Agilent 7890A GC system connected to an Agilent 5975C inert MSD with a triple-axis detector. The BP20 (WAX) column used measured 30 m × 0.25 mm with a polyethylene glycol stationary phase and a film thickness of 0.25 µm (SGE).

Sampling

Rutab dates were purchased from local supermarkets and stored at 0 °C in chilled conditions. ### Sample Preparation Deseeded Rutab dates were blended into a homogeneous paste for analysis.

Mixture Extraction

Methanol, hexane, and chloroform were each employed as solvents to extract biochemical compounds from the blended date mixture. The process involved placing 2 g of prepared sample into a beaker and submerging it in 20 mL of each solvent for 2 hours at room temperature. This allowed for comparison to determine the most effective solvent for extraction. Following extraction, the solutions were filtered through Whatman No. 1 filter paper and concentrated using a rotary evaporator at 40 °C. The residue was derivatized with BSTFA prior to GC-MS analysis. ### Derivatization of the Extract Five hundred microliters of the filtered extract were transferred into a 2 mL vial and mixed with 80 µL of BSTFA and 20 µL of TMCS.

The vial was sealed and heated in an oven at 65 °C for one hour.

GC-MS Analysis

The GC-MS protocol followed the method established by Hema et al. (2010). For analysis, 20 mg of the crude extract was dissolved in 2 mL of hexane alongside 20 µL of 2 N potassium hydroxide in methanol. The mixture was vortexed for one minute, centrifuged at 10,000 rpm for ten minutes, and 1 mL of the clear supernatant was transferred into an amber vial for injection into the GC-MS system. Detection was performed using electron ionization at 70 eV. Helium served as the carrier gas at a constant flow rate of 1 mL/min. The injection volume was set at 0.5 µL with a split ratio of 10:1; injector temperature was maintained at 250 °.

2. Result and Discussion

Methanol – Chloroform - Hexane Extract (33.33 % - 33.33 % - 33.33%)

(mixed dates)

The extract of M 33.33 % + Ch 33.33 % + H 33.33 % as shown in Table 4.5 consist of peaks that have area between 0.11 - 17.37 %. Six of the peaks have similarity higher than 90 %. Compounds detected consist of; Furfural (C5H4O2) with retention time of 13.69 min and peak area of 1.46 %; 2-furanmethanol (C5H6O2) with retention time of 18.40 min and peak area of 0.31 %; Hexadecanoic acid methyl ester (C17H34O2) with retention time of 29.48 min and peak area of 0.17, n-Hexadecanoic acid (C16H32O2) with retention time 40.39 min and peak area 1.07 %; Octadecanoic acid (C18H36O2) with retention time of 43.27 min and peak area of 0.21 %; 9,12-Octadecadienoic acid (Z,Z) (C18H32O2) with retention time 44.34 min of and peak area of 1.88 %.

Tebla 1: Compounds detected in dates from Methanol – Chloroform - Hexane (33.33% - 33.33% - 33.33%) extract

peak number	Retention time (min)	Peak's area %	Compound	CAS	Molecular formula	Molecular weight (MW) g/mol	Similarity index (%)
4	13.69	1.46	Furfural	000098-01-1	$C_5H_4O_2$	96	91
6	18.40	0.31	2-Furanmethanol	000098-00-0	$C_5H_6O_2$	98	94
9	24.92	0.23	2,5-Furandicarboxaldehyde	000823-82-5	$C_6H_4O_3$	124	87
14	29.48	0.17	Hexadecanoic acid, methyl ester	000112-39-0	$C_{17}H_{34}O_2$	270	97
15	30.145	1.61	4H-Pyran-4-one, 2,3-dihydro-3,5-dihydroxy-6-methyl-	028564-83-2	$C_6H_8O_4$	144	87
16	31.34	0.11	2-Propanamine, N-methyl-N-nitroso	030533-08-5	$C_4H_{10}N_2O$	102	68
17	32.851	0.21	2-Furancarboxylic acid	000088-14-2	$C_5H_4O_3$	112	72
19	34.214	17.3	5-Hydroxymethylfurfural	000067-47-0	$C_6H_6O_3$	126	62
23	40.396	1.07	n-Hexadecanoic acid	000057-10-3	$C_{16}H_{32}O_2$	256	99
26	43.27	0.21	Octadecanoic acid	000057-11-4	$C_{18}H_{36}O_2$	284	96
27	43.65	0.92	9-Octadecenoic acid,	000112-79-8	$C_{19}H_{36}O_2$	196	99
27	43.27	o.92	cis-Vaccenic acid	000506-17-2	$C_{18}H_{34}O_2$	282	99
28	44.34	1.88	9,12-Octadecadienoic acid (Z,Z)	000060-33-3	$C_{18}H_{32}O_2$	280	99
30	45.91	2.02	D-Allose	002595-97-3	$C_6H_{12}O_6$	180	86

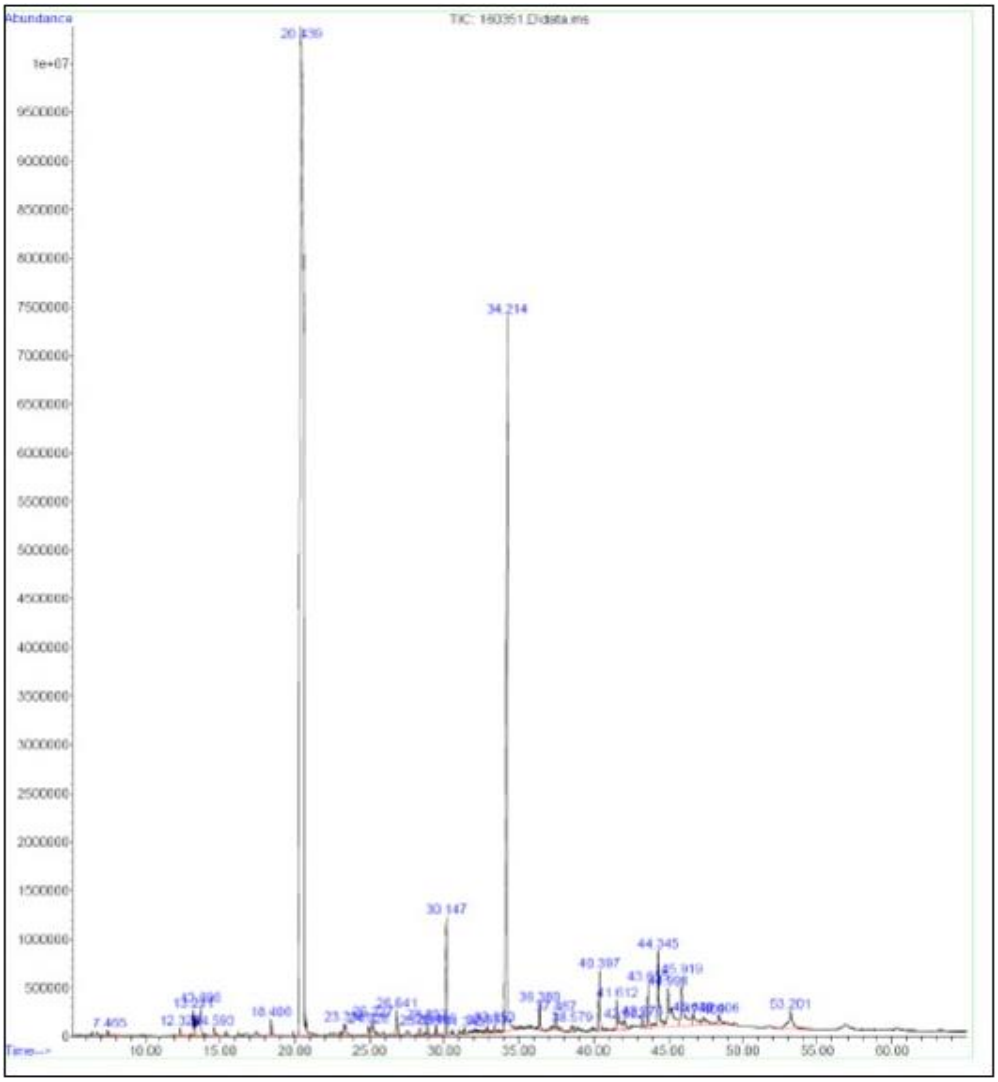

Figure 1: GC-MS of total compounds in dates sample extracted with combination of three solvents together

3. Conclusion and Recommendation

In this study, we found that the compounds extracted from mixing the three solvents were higher than extracting each solvent separately, while polar compounds, medium-polar compounds, and other non-polar compounds appeared. We found that the a-Hexalecanoic acid compound was not found when we extracted with methanol or chloroform. Therefore, we recommend mixing the solvents together to obtain better results, improve the extraction process, and ensure excellent results.

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