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Research Article

Application of HPLC for detection of sildenafil/tadalafil in marketed honey in Oman

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Abstract: Honey adulteration allegedly with phosphodiesterase-5 (PDE-5) inhibitors including sildenafil, and tadalafil is a common and dangerous practice. This study aimed to develop a procedure to detect the presence of common adulterants namely sildenafil and tadalafil using RP-HPLC. Seven commercial honey samples of local and international origin were collected from supermarkets and honey sellers. Both the adulterants in honey samples were identified and quantified with the help of an HPLC technique. Chromatographic separation was done in RP-HPLC mode using buffer: methanol: acetonitrile (5.8: 2.5: 1.7) mobile phase and diode array as a detector. The buffer used was 0.05 M Triethylamine orthophosphate pH (3.0). The results showed that four honey samples (HAD1, HAD5, HAD6, and HAD7) were adulterated with sildenafil, and among them, HAD5 contained the maximum amount of sildenafil as 22.65 mg/g of the honey sample. However, only 2 honey samples HAD4 and HAD6 were found to be adulterated with tadalafil (1.248 and 0.7 mg/g) of the tested honey sample. The result of this study warrants rigorous quality control of the commercially available honey products in Oman by the authorities. The consumption of adulterated honey samples may impact the health of consumers hence further detailed studies must be carried out to confirm the findings of the current study and novel analytical methods be developed to detect the level of other possible adulterants in this valuable product.

1. INTRODUCTION

Honey, a valuable foodstuff produced by bees contains polyphenols, organic acids, vitamins, amino acids, and enzymes. The unique flavor, aroma, and enzyme activity of honey is because of the compounds present in it which also determines its quality. Honey is a valuable traditional natural source of highly nutritive food besides possessing medicinal value. It has various pharmacological properties such as anti-inflammatory, anti-aging, antioxidant, and anticancer. European Union is the second largest producer of honey followed by China (González-Ceballos

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et al., 2023). Because honey is widely consumed owing to its nutritive and therapeutic benefits, hence it is more vulnerable to adulteration (Fakhlaei *et al*., 2020). This is achieved through two types of adulteration: direct (addition of adulterants) and indirect (mixing pure honey with low-quality, inexpensive honey or altering it through bee feeding)(Gong *et al*., 2017). Honey adulteration is effortless, economically motivated, and hard to detect. Most of the physical and chemical indices of adulterated honey are very similar to that of natural honey products (Xu *et al*., 2020; Dou *et al*., 2023). Adulterated honey is defined as any commercially marketed "pure" honey products that are found to contain more than a 5% mass ratio limit for sucrose or maltose (Ng & Reuter, 2015).

Numerous investigations showed that honey products were adulterated with natural drugs (ginseng), synthetic drugs like anti-obesity and sex stimulant agents such as sildenafil and tadalafil (Zakaria & Yacob, 2017). Tadalafil (sold under the brand name CIALIS®) and sildenafil (sold under the brand name VIAGRA®) are the two most popular phosphodiesterase 5 (PDE-5) inhibitors used to treat erectile dysfunction (ED) (FDA, 2022; Zakaria & Yacob, 2017). Erectile dysfunction (ED) a significant health issue is defined as the inability to obtain and sustain an erection strong enough for satisfying sexual performance, which has been linked to both organic and psychogenic reasons (Scaglione *et al*., 2017). Up to 60-70% of the patients suffering from ischemic heart disease (IHD) and hypertension suffer from erectile dysfunction. These PDE-5 inhibitors can interact badly with several medications used to treat patients suffering from cardiovascular diseases and thus can only be used in consultation with a licensed healthcare provider. Moreover, people with diabetes, hypertension, hypercholesterolemia, or heart disease frequently use nitrates (nitroglycerin), which may interact with these unreported substances that are used to adulterate honey and reduce blood pressure to dangerous levels (FDA, 2022). The FDA's laboratory issued a warning to four companies for selling honey-based products which upon testing were found to contain hidden active pharmaceutical ingredients including those present in the FDA-approved erectile dysfunction medications i.e., Cialis (tadalafil) and Viagra (sildenafil) (FDA, 2022). In Saudi Arabia, herbal medicines were found to contain various synthetic drugs such as sildenafil, tadalafil, and glibenclamide (Bogusz *et al*., 2006). A previous study conducted in the Sultanate of Oman showed that 7 of the 33 herbal medicines and food samples contain sildenafil, tadalafil, and vardenafil (Al Lawati *et al*., 2017).

The profit-oriented adulteration of honey is becoming a global problem. Besides being unethical practice, adulterated honey also has undeniable consequences on human health affecting kidneys, heart, brain, etc. Although the consumption of honey has increased in the last few years, the consumers' trust and interest in honey-based products due to adulteration is on the decline (Fakhlaei *et al*., 2020). Therefore, honey, a valuable food product, needs to be regularly monitored for its safety and quality. There are numerous methods available for honey adulteration detection including gas chromatography with flame ionization detector (GC-FID) or with mass spectrometry (GC-MS), high-performance liquid chromatography coupled with mass spectrometry (HPLC-MS) or tandem mass spectrometry (HPLC-MS-MS), refractive index detectors (RID), electrochemical detectors (ECD). near-infrared transflectance (NIRT) and Fourier transform infrared (FTIR) spectroscopy and many others (Cárdenas-Escudero *et al*., 2023; Mehryar & Esmaiili, 2011). These analytical techniques are modern and sophisticated and help in detecting honey adulteration but at the same time suffer from one or other limitations, i.e., cost of instrumentation, maintenance, the requirement of skilled personnel, and complicated and time-consuming. The limitation of the cost and skilled personnel was overcome with the use of high-performance liquid chromatography coupled with a diode array detector (HPLC-DAD). The main objective of the current study is to detect sildenafil and tadalafil as possible adulterants in commercially available honey products that are claimed to boost sexual performance.

2. MATERIAL and METHODS

A total of seven honey samples were collected from different grocery stores, and supermarkets in Oman. Collected honey samples were coded as HAD1 to HAD7 to hide the identity of the manufacturers and to avoid any conflict of interest. The collection location and coding of honey samples are presented in Table 1, which shows the location from where it was collected. The honey samples were tested for the presence of sildenafil and tadalafil contents using HPLC (Agilent, 1220 Infinity II). Each experiment was repeated twice. All the chemicals including the solvents methanol, acetonitrile, and the buffers triethylamine orthophosphate were used of HPLC grade and purchased by Merck AG, Darmstadt, Germany. The standard sildenafil and tadalafil were received as gift samples from the Ministry of Health, Sultanate of Oman. The standard stock solution of 200 µg/mL of sildenafil/tadalafil in methanol was prepared.

Table 1. Code, location, and the manufactured honey samples.

S. No	Code Assigned	Location
1	HAD1	Al Seeb
2	HAD2	Al Mawaleh South
3	HAD3	Al Khuwair South
4	HAD4	Al Ghubrah
5	HAD5	Al Seeb
6	HAD6	Al Mawaleh South
7	HAD7	Al Ghubrah
Total Samples		07

2.1. Apparatus

The HPLC separation was performed on a C18 Agilent HPLC column (250 × 4.6 mm), having a particle size of 5 microns using an Agilent 1220 LC system with a diode array detector (DAD).

2.2. Reference Standard Preparation

The pure drug sample of 10 mg of sildenafil (98.8% potency) or tadalafil (99.8% potency) was dissolved in 10 mL of methanol in a volumetric flask. The solution was sonicated for 10 min and then diluted up to 50 mL with methanol for HPLC analysis.

2.3. Sample Preparation

An accurately weighed honey sample (0.5 g) was dissolved in 50 mL of methanol. The extract was centrifuged for 5 min at 4000 rpm. The supernatant was collected and filtered through a 0.45 µm filter and used as such for the HPLC analysis.

2.4. Mobile Phase Preparation

The mobile phase consisted of buffer: methanol: acetonitrile in the ratio of (5.8:2.5:1.7) was prepared by adding 580 mL of 0.05 M triethylamine orthophosphate pH (3.0) solution, 250 mL of methanol and 170 mL of acetonitrile in 1000 mL of volumetric flask. The buffer used was 0.05 M Triethylamine orthophosphate pH (3.0) prepared by mixing 7 mL of triethylamine in 1 L purified water and the pH was adjusted to 3.0 ± 0.1 with concentrated orthophosphoric Acid. The solvent system was subjected to the process of sonication for 15 min to ensure proper mixing of the mobile phase. The mobile phase was filtered through a membrane filter with 0.45 µm pore size to remove any insoluble particulates.

2.5. HPLC Procedure

The RP-HPLC analysis was conducted using an Agilent 1220 LC system with a diode array detector (DAD). The injection volume was set to 20 µL. The elution run time of 15 min was employed in the isocratic mode of the mobile phase. The flow rate of 1 mL/min at 0-600 psi pressure was optimized with the column temperature maintained at 35 ^{o}C used for separation. The wavelength of the detection was set at 290 nm. A literature search was done and after the trials of various methods reported, the method from the Ministry of Health, Sultanate of Oman

was finally selected. In one of the studies retention times of the tadalafil was found to be 4.46 min when the solvent system selected was acetonitrile: acetate buffer pH 2.8 in the ratio of 55:45 v/v (Sutar *et al*., 2008).

2.6. Calculation

The following formula was used to find out the adulteration in the honey samples:

$$\left(\frac{mg}{g}\right) = PA * \frac{SC}{PA} * PS$$

PA: Peak area of sildenafil or tadalafil in sample
SC: Standard concentration
PS: Potency of standard

2.7. Validation

2.7.1. *Method development*

A single reversed-phase (RP)-HPLC method was used by applying in different compositions, ratios, and pH of mobile phases for the simultaneous estimation of sildenafil and tadalafil. This method shows a good linearity within the concentration range of 2-10 µg/mL for both analytes. The **s**ystem suitability tests were performed on freshly made 5 replicates from the standard stock solution of sildenafil and tadalafil. System suitability was evaluated using parameters such as retention time, % RSD of retention time, peak area, and theoretical plates.Validation of the developed RP-HPLC method was performed according to ICH guideline parameters and referencing some research papers (Agency, 1995; Pannu *et al*., 2022)**.**

3. FINDINGS

3.1. Validation

A rapid, sensitive, and robust RP-HPLC method was used for the simultaneous estimation of sildenafil and tadalafil in honey samples. This reported method is precise, accurate, linear, and specific to the parameters of validation. The linearity was found in the concentration range of 02-10 µg/mL for both analytes with a correlation coefficient of 0.9991 for sildenafil and 0.9995 for tadalafil.

This method was found to be sensitive to low LOD Values i.e., 0.42 µg/mL, and 0.30 µg/mL for sildenafil and tadalafil respectively. This method is validated at LOQ level i.e., 1.28 µg/mL and 0.90 µg/mL for sildenafil and tadalafil respectively. The % RSD for the method precision and robustness parameters were found within limits. The validation parameters are summarized in Table 2.

Table 2. Validation parameters.

Parameters	Analyte	
	Sildenafil	Tadalafil
Absorption maxima λ_{max} (nm)	290 nm	290 nm
Linearity (µg/mL)	2-10	2-10
Correlation coefficient(R^2)	0.9991	0.9995
Regression equation (y)	366.53x -30.143	788.53x + 60.19
Limit of detection (µg/mL)	0.42	0.30
Limit of quantification (µg/mL)	1.28	0.90
Intraday precision (n=6) (% RSD)	0.821	0.684
Interday precision (n=6) (% RSD)	0.877	0.432

3.2. HPLC Data

We validated a new, simple, highly sensitive RP-HPLC method for simultaneous estimation of sildenafil and tadalafil in marketed honey. The chromatographic separation was achieved on an ODS C18 Agilent (250 × 4.6 mm, 5µ) column with a mobile phase consisting of buffer: methanol: acetonitrile (5.8:2.5:1.7). Both peaks were resolved at a flow rate of 1.0 mL/min with 35 °C column temperature. The retention times of sildenafil and tadalafil were found to be 6 min and 11 min respectively. The method was found to be linear in the concentration range of 2-10 µg/mL for both analytes with correlation coefficients of 0.9991 and 0.9995 respectively. The method was successfully applied for detecting the sildenafil or tadalafil level in selected honey samples marketed in Oman.

3.2.1. *Assay of sildenafil and tadalafil in honey samples*

The results of the assay used for checking the content of sildenafil and tadalafil in marketed honey samples are shown in Table 3. The drug sildenafil or tadalafil were eluted at 6.244 and 10.898 minutes, respectively (C_{18} column Agilant coupled with DAD; mobile phase ratio buffer: methanol: acetonitrile in the ratio of (5.8:2.5:1.7) whereas in another study carried out by Abdelshakour *et al* obtained the retention time of sildenafil or tadalafil to be at 4.94 and 10.40 minutes (C_{18} column HPLC-UV; mobile phase with acetonitrile and 0.05% formic acid). The results showed that 4 honey samples were found to have sildenafil content. The contents of sildenafil were found to be higher in samples HAD5 and HAD1 and were 22.65 and 10.25 mg/g of the honey. The other two samples HAD6 and HAD7 showed much lower sildenafil contents i.e., 0.325 and 0.350 mg/g of honey tested. The study conducted in the Sultanate of Oman among 33 samples of herbal medicines and food supplements also showed that sildenafil was the most common adulterant with a percentage range from 0.7 to 12 wt % (Al Lawati *et al*., 2017). Similarly, sildenafil was considered as primary active compound detected in the range of 1.1 mg/sachet to 124 mg/sachet in a study carried out by Sirhan *et al*., in honey-mixed herbal sachets (Ala'Y *et al*., 2023). Out of 50 samples studied by Abdelshakour *et al*., almost all samples labeled to contain herbal or natural ingredients were found to be adulterated with PDE5 inhibitors such as sildenafil and tadalafil without being labeled in the package and sold as safe and natural products (Abdelshakour *et al*., 2021). Our study showed that 2 of the tested honey samples were adulterated with tadalafil. Among them, the sample HAD4 contains the maximum concentration of tadalafil i.e., 1.24 mg/g of tested honey. The HAD6 has a much lower concentration of tadalafil 0.7 mg/g of the honey sample tested, whereas Lawati *et al* reported tadalafil content in one of the food samples with 39 wt % (Al Lawati *et al*., 2017). In another study, Ala'Y *et al* detected tadalafil in few samples in the concentration range of 0.67 mg/sachet to 76.6 mg/sachet (Ala'Y *et al*., 2023). The HPLC chromatogram results are represented in the Figure 1.

Table 3. Assay determination of sildenafil and tadalafil in mg/g of honey sample by RP-HPLC.

Sample	Estimation (mg/g of sildenafil in honey samples)	Estimation (mg/g of tadalafil in honey samples)
HAD1	10.25	x
HAD2	x	x
HAD3	x	x
HAD4	x	1.248
HAD5	22.65	x
HAD6	0.325	0.7
HAD7	0.35	x

x: means no sildenafil/tadalafil present

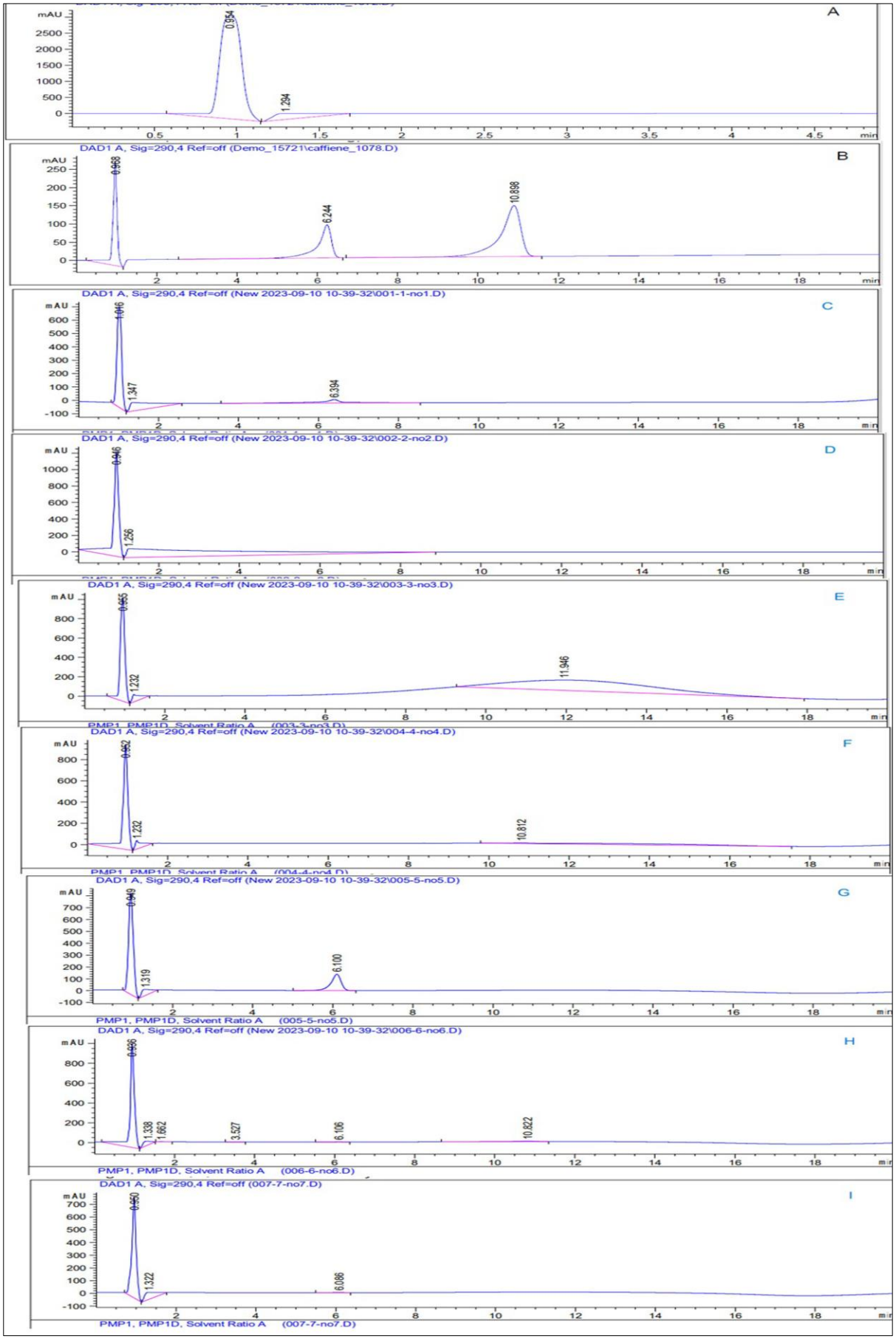

Figure 1. HPLC chromatogram of the solvent methanol (A), Combined sildenafil + tadalafil (B), HAD1 (C), HAD2 (D), HAD3 (E), HAD4 (F), HAD5 (G), HAD6 (H), HAD7(I) at a sample concentration of 10 mg/mL.

4. CONCLUSION

In this study, the HPLC procedure was applied for the determination of the sildenafil or tadalafil in the honey samples. HPLC is a valuable extension of the screening of the sildenafil or tadalafil not agreeable to UV-Vis as the drugs sildenafil and tadalafil have λ_{max} absorbance overlapping. A total of 7 honey samples were identified as the honey samples claimed to provide sexual enhancement and boost reproductive health. Among the tested samples 4 of the samples were found to be adulterated with sildenafil and 2 of the samples with tadalafil. The total sildenafil contents were found to be varied as samples HAD5 and HAD1 showed the maximum concentration whereas HAD6 and HAD7 had much lower concentrations of sildenafil. The tadalafil content was found to be maximum with sample HAD4 whereas HAD6 contains much lower contents of tadalafil. One of the samples HAD3 showed broad peaks at 11.946 which may be due to the presence of two or more poorly resolved compounds. These results provide the high quantitative capability of the HPLC method for determining honey adulteration with sildenafil or tadalafil. Furthermore, detailed studies must be carried out to confirm the findings of the current study, and novel analytical methods be developed to detect the level of other possible adulterants in this valuable product. Additionally, it would be interesting to perform other adulterant studies to determine the quality of honey samples in Oman and their impact on the health of consumers.

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Declaration of Conflicting Interests and Ethics

The authors declare no conflict of interest. This research study complies with research and publishing ethics. The scientific and legal responsibility for manuscripts published in IJSM belongs to the authors. **Ethics Committee Number**: EBS/PRES0401-COP/04/2022-23 College of Pharmacy, National University of Science and Technology, Azaiba, Muscat, Sultanate of Oman.

Authorship Contribution Statement

H.H. Aldhakhri: Investigation; Methodology. **F.H. Alqassabi**: Investigation; Methodology. **I.A. Alrasbi:** Investigation; Methodology. **D.A. Alhinai:** Investigation; Methodology. **SSR Al Salt:** Analyzed and Interpreted data **S. Pannu:** Formal analysis, supervision. **S. Al Balushi:** Formal analysis, supervision. **S.A. Khan:** Formal analysis, writing. **Md J. Akhtar:** Conceptualization, Data curation.

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