Fast, Robust and Reliable Method for the Identification and Quantitation of Sildenafil Residue in Honey using LC-MS/MS

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In recent years, natural products and herbal medicines are increasing in popularity all over the world. The adulteration of natural products with synthetic substances is a serious concern as it impairs therapeutic effects, causes unnecessary expenses and leads to patients suffering from adverse effects.

A sensitive and selective method for LC-MS/MS has been developed to identify, quantify and confirm residue of Sildenafil in honey based on the accurate determination of the effects of products.

**ABSTRACT**

In recent years, natural products and herbal medicines are increasing in popularity all over the world. The adulteration of natural products with synthetic substances is a serious concern as it impairs therapeutic effects, causes unnecessary expenses and leads to patients suffering from adverse effects. Adulterated honey is one of the serious problems in honey. Therefore, this work presents a fast, robust and reliable method for the identification and quantitation of Sildenafil residue in honey using LC-MS/MS. Adverse effects of Sildenafil especially cardiovascular risk are still under controversy. This highlights the need for a sensitive, specific and selective method for the detection of Sildenafil in food. Sildenafil is a phosphodiesterase type-5 inhibitor used for the treatment of erectile dysfunction and pulmonary hypertension.

**INTRODUCTION**

Honey is one of the precious food commodities from ancient times and there is high market demand for natural honey. However, honey adulteration is an increasing trend and some studies indicate that honey is one of the most frequently adulterated food products. Adulteration of honey may not only be due to the addition of foreign honey, but also to the addition of other products. To the best of our knowledge, sensitive and selective method to detect the levels of Sildenafil residue in honey samples is not yet available.

A LC-MS/MS method was developed using SCIEX Q3000 QTRAP system operated in Multiple Reaction Monitoring (MRM) mode to identify and quantify Sildenafil in honey with high volume and selectivity. This method was validated using six standards at four concentration levels. Comparison of the spectra showed that there is no interference from endogenous honey components and the ion transitions were specific to Sildenafil: multiply charged ion (m/z 544.4) was monitored in positive mode and product ions of (m/z 475.4, 85.5) were selected in negative mode. The average retention time of Sildenafil was 4.6 min. Linearity was demonstrated over a concentration range of 0.25 to 1000 ng/mL with the correlation coefficient 0.9995. Calibration curves were generated using matrix-matched standards. LOQ was established by injecting 6 replicates over period of 3 days and LOQ was 0.5 µg/kg.

**RESULTS**

A representative chromatogram of qualifier and quantitative MRM transitions is shown in Figure 2. Multiple MRM transitions were monitored as the rate of qualifier and qualifier for identification of Sildenafil in samples. In addition, the sensitivity of the MRM method was calculated using standard curve software version 3.0. The average MRM ratio of all standard injections with tolerance matching 0.0075% was displayed in the peak review window. The specificity, limit of detection and quantification (LOD and LOQ) were used to evaluate the linearity of calibration. LOD and LOQ were achieved to 0.5 and 0.5 µg/kg, respectively. The relative standard deviation (RSD) was 6.7% and 1.0% at 0.5 and 105.8 µg/kg, respectively. The recovery of Sildenafil at three concentration levels (LOQ, xLOQ, 0.5 LOQ) was measured at 98.5% (0.5 LOQ), 99.0% (LOQ) and 100% (xLOQ), respectively. The precision of the method was achieved at 100% in all concentrations. The data generated was satisfactory and within the respective limits of accuracy and precision. The data generated for the identification and quantitation of Sildenafil in honey samples are within the stipulated limits of accuracy and precision.

**REFERENCES**


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