Cd(II) and Cu(II) Profile of Commercially Available Essential Oils : A Solid Phase Extraction Preconcentration prior to Determination by FAAS

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INTRODUCTION

Industrialization and environmental contamination have significantly increased human exposure to heavy metals, which are among the most important environmental risk factors affecting health. Among them, cadmium (Cd) and copper (Cu) are of particular concern due to their documented roles in reproductive toxicity and infertility (1,2).

Cadmium is a highly toxic, non-essential metal widely distributed in the environment and enters the body primarily through food, water, and inhalation. It accumulates in tissues because of its long half-life and exerts toxic effects on multiple organs, including the reproductive system (3). It also induces oxidative stress, increases reactive oxygen species (ROS) production, and decreases the antioxidant defense capacity in the testes (4). Cigarette smoking is considered one of the major sources of non-occupational cadmium exposure, with each cigarette containing approximately 1.5 µg cadmium, contributing to infertility and asthenozoospermia in smokers (5,6).

Copper, unlike cadmium, is an essential trace element that plays a vital role in hemoglobin synthesis, enzymatic reactions, and oxygen transport (7,8). Excess copper promotes oxidative stress, leading to lipid peroxidation, sperm membrane damage, and reduced acrosome integrity, which compromises fertilization potential. Experimental studies in animals have also demonstrated that copper concentrations above physiological levels lead to testicular tissue degeneration, reduced spermatogenesis, and impaired fertility outcomes (9).

Cadmium and copper act through multiple mechanisms, including direct cytotoxicity, oxidative damage, and endocrine disruption, making them critical factors in infertility (10). Considering the global rise in infertility rates, further understanding of the reproductive toxicity of cadmium and copper is essential for developing preventive measures and reducing their burden on human fertility (4,10).

Essential oils (EOs) are complex and unstable compounds processed from aromatic plants including terpenes, alcohols, esters, aldehydes, etc. These oils are commonly used in food, pharmaceutical, and cosmetic industries regarding their natural antioxidant and antimicrobial features. Therefore, the identification and accurate determination of both organic and inorganic contaminants in EOs are becoming increasingly important (11). In this study, commercially available rose, apricot, jasmine, black cumin, pine turpentine, orange flower, lavender, mint, garlic, white rose, jasmine, eucalyptus and castor oils were analyzed for their Cd(II) and Cu(II) contents. The solid phase extraction method was used as a preconcentration procedure . Previously synthesized and characterized magnetite was used as solid sorbent in this method. The preconcentrated sample solutions easily analyzed by relatively less sensitive, cheap and accessible instrument such as flame atomic absorption spectrometry (FAAS) (12).

MATERIALS AND METHOD

The samples were collected from local supermarkets in Balıkesir, Türkiye. Previously synthesized and characterized magnetite was used as solid sorbent in this method. All chemicals were analytical grade and used without any purification step. A Perkin Elmer Aanalyst 200 FAAS (Waltham, MA, USA) was utilized to perform the determination Cu(II) and Cd(II) equipped with D2 background determination.

Certain amount of essential oil sample was added on 0.26 g magnetite and vortexed for 41 s for sorption of the analytes. Then, oil sample was decanted and excess of the oily matrix on solid phase was removed by washing with n-hexane. Followingly, 6.2 mL of 0.61 mol L^{-1} HNO_3 was added on solid phase and vortexed for 66 s for elution. The solid phase extraction procedure was summarized below. Then, the analyte concentrations in eluate were determined by flame atomic absorption spectrometer (FAAS).

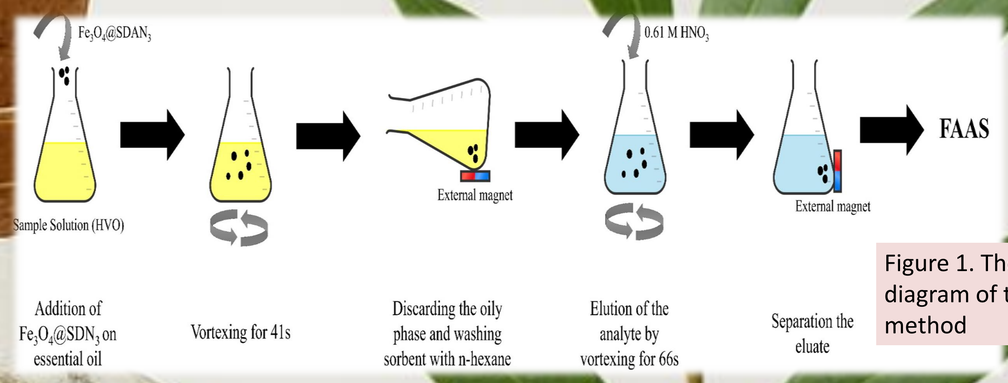

Figure 1. The schematic diagram of the improved method

RESULTS

The eluates were analyzed by FAAS, the calibration graphs and obtained results were given in Table 1. The measured experimental data were also evaluated according to the dermatological health risk assesment. The calculated data and the risk parameters are also given in Table 2.

Table 2. Dermatological health risk assessment for Cu(II) and Cd(II)

Essential Oil	Margin of Exposure (MoS)	Hazard Quotient (HQ)	Hazard Index (HI)	Cancer Risk (CR)
Black seed oil	3.7×10^7	0.44	0.44	1.7×10^{-7}
Pine turpentine	3.1×10^7	0.52	0.52	2.1×10^{-7}
Apricot oil	4.1×10^7	0.40	0.40	1.6×10^{-7}
Peppermint oil	3.2×10^7	0.50	0.50	2.0×10^{-7}
Garlic Oil	5.4×10^7	0.30	0.30	1.2 xx 10^{-7}
Rosa alba (white rose) oil	-	-	-	-
Argan oil	-	-	-	-
Coconut oil	4.4×10^7	0.37	0.37	1.5×10^{-7}
Lavender oil	-	-	-	-
Eucalyptus oil	-	-	-	-
Castor oil	4.2×10^7	0.39	0.39	1.6×10^{-7}
Jasmine oil	4.6×10^7	0.36	0.36	1.4×10^{-7}
Orange blossom oil	3.6×10^7	0.45	0.45	1.8×10^{-7}

$$\mathbf{MoS} = \frac{\mathbf{NOAEL}}{\mathbf{SED}} \mathbf{\ x\ 100} \qquad \mathbf{HQ} = \frac{\mathbf{ADI}}{\mathbf{RfD}} \qquad \mathbf{HI} = \frac{\mathbf{ADI}}{\mathbf{RfD}} \qquad \mathbf{CR = SED\ x\ CSF}$$

Symbol	Explanation
MoS	> 100 there is no toxicity ; $> 10^5$ no apparent risk for human
NOAEL	No of observed adverse effect level (mg/kg /day)
SED	Systemic exposure dosage (mg/kg /day)
ADI	Average daily intake (kg/day)
RfD	Reference dose (0.04 and 0.001 mg/kg/day for Cu and Cd respectively)
HQ	To assesment of non-carcinogenic risk
HI	Cumulative risk of different metals (≤ 1 no significant cumulative risk)
CDI	Chronic daily intake (mg/kg/day)
CSF	Carcinogenic slope factor (6.3 and 1.5 mg/kg/day for Cd and Cu resp.) (the potency of a substance in causing cancer)
CR	$<10^{-6}$ negligible; $> 10^{-4}$ unacceptable ; (10^{-6} - 10^{-4}) acceptable limit

Table 1. The Cu(II) and Cd(II) levels of essential oils

Essential Oil	Cu (mg/kg)	Cd (mg/kg)
Black seed oil	2.68 ± 0.19	< LOD
Pine turpentine	3.21 ± 0.19	< LOD
Apricot oil	2.46 ± 0.10	< LOD
Peppermint oil	3.15 ± 0.29	< LOD
Garlic Oil	1.85 ± 0.19	< LOD
Rosa alba (white rose) oil	< LOD	< LOD
Argan oil	< LOD	< LOD
Coconut oil	2.30 ± 0.05	< LOD
Lavender oil	< LOD	< LOD
Eucalyptus oil	< LOD	< LOD
Castor oil	2.41 ± 0.14	< LOD
Jasmine oil	2.19 ± 0.20	< LOD
Orange blossom oil	2.78 ± 0.13	< LOD

LOD Values : **Cu 0.41 mg/kg ; Cd 0.28 mg/kg**

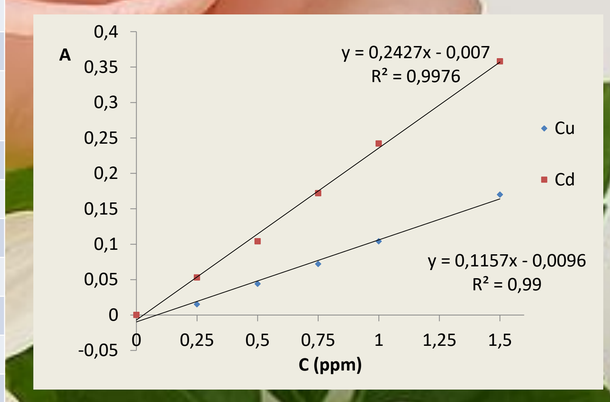

Figure 2. The analytical calibration graph for Cu(II) and Cd(II)

CONCLUSION

The conventional methods for trace element determinations in oil generally include a sample decomposition and determination by sensitive instrument. This method use solid phase extraction method as a preconcentration procedure. The preconcentrated sample solutions easily analyzed by relatively less sensitive, cheap and accessible instrument such as flame atomic absorption spectrometry (FAAS). The preconcentration procedure that was based on solid phase extraction was utilized for separation of analytes . In this way, a difficult matrix such as oil was eliminated. This simple method has facilitated the determination of Cu(II) and Cd(II) in essential oils. Thus, the monitoring of risk factors has been facilitated.

The tolerable upper intake levels established by NIH for adults are 10 mg/day and 62 µg/day for Cu and Cd, respectively. Measured values for whole essential oils are lower than these limits. Additionally, evaluating the Cu(II) and Cd(II) concentrations in selected oils allowed for the assesment of certain risks. In terms of MoS, HQ and CR, none of the samples exhibited and toxicity and cancer risk.

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