

PLUM SPIRIT AS A SOURCE OF LEAD EXPOSURE – RESULTS OF CONCENTRATION ANALYSIS
SLIVKOVÝ DESTILÁT AKO ZDROJ EXPOZÍCIE OLOVOM – VÝSLEDKY ANALÝZY KONCENTRÁCIE

TATARKOVÁ Mária, SOVIČOVÁ Miroslava*, ULBRICHTOVÁ Romana, KUKA Stanislav,
DUTKOVÁ Michaela, NOVÁK Martin, ŠTEFANOVÁ Eliška, MALOBICKÁ Eva,
HUDEČKOVÁ Henrieta, BAŠKA Tibor

Department of Public Health, Comenius University in Bratislava, Jessenius Faculty of Medicine in Martin
* *Corresponding author*

ABSTRAKT

Úvod: Niekoľko štúdií naznačilo, že kontaminácia domácich destilátov olovom môže predstavovať relevantný verejnozdravotný problém.

Cieľ: Cieľom štúdie bolo stanoviť koncentrácie olova v tradičných slivkových destilátoch určených na vlastnú spotrebu vo vzťahu k vybraným faktorom, ktoré ju môžu ovplyvňovať.

Metódy: Analyzovali sme 22 vzoriek tradičných slovenských slivkových destilátov určených na vlastnú spotrebu. Vzorky boli destilované v legálnych pestovateľských páleniciach v rôznych častiach Slovenska. Príprava vzoriek pozostávala z predchádzajúcej mineralizácie pomocou mikrovlnného rozkladného systému MULTIWAVE 60 50 Hz. Vzorky boli analyzované atómovou absorpčnou spektrometriou s grafickou pieckou (AAS GBC XPLORAA 5000 s GF 5000).

Výsledky: Olovo bolo zaznamenané v 19 vzorkách slivkových destilátov. Najvyššia koncentrácia olova bola vo vzorke z okresu Banská Bystrica ($328,63 \pm 57,12$) a Martin ($105,26 \pm 2,46$). Väčšina vzoriek ($N = 13$) mala hladiny nižšie ako je limit kvantifikácie (LOQ). Priemerná koncentrácia olova bola $49,53 \mu\text{g/l}$. Priemerná koncentrácia etanolu bola $51,32 \%$.

Záver: Zistenia naznačujú, že olovo je rozšíreným kontaminantom slivkových destilátov na Slovensku, keďže bolo zistené vo väčšine vzoriek bez ohľadu na iné faktory. Vzhľadom na vysokú popularitu slivkových destilátov na Slovensku, má tento problém značný verejnozdravotný význam a zaslúži si pozornosť.

Kľúčové slová: Olovo. Domáci alkohol. Tradičný slivkový destilát. Analýza. MOE.

ABSTRACT

Introduction: Several studies have suggested that lead contamination of home-made spirits could represent a relevant public health problem.

Objective: The objective of the study was to determine lead concentration in traditional plum spirits for own consumption in relation of selected factors that may influence it.

Methods: We analysed 22 samples of traditional Slovak plum spirit intended for own consumption. Samples were distilled in legal craft fruit distillery in different parts of Slovakia. Sample preparation consisted of previous mineralization using microwave decomposition system MULTIWAVE 60 50 Hz. The samples were analyzed by atomic absorption spectrometry with graphic furnace (AAS GBC XPLORAA 5000 with GF 5000).

Results: Lead was detected in 19 samples of plum spirits. The highest concentration of lead was in the samples from Banská Bystrica (328.63 ± 57.12) and Martin (105.26 ± 2.46) districts.

Most samples ($N = 13$) had levels lower than the limit of quantification (LOQ). The average lead concentration was $49.53 \mu\text{g/l}$. The average concentration of ethanol was 51.32% .

Conclusions: The findings indicate lead as the widespread contaminant of plum spirits in Slovakia as it was detected in most of samples regardless of other factors. Considering a high popularity of plum spirit in Slovakia the issue has a notable public health significance and deserves attention.

Key words: Lead. Homemade alcohol. Traditional plum spirits. Analysis. MOE.

INTRODUCTION

According to data from the Financial Directorate of the Slovak Republic, approximately 10% of the total amount of alcohol that is subject to tax is produced in craft fruit distilleries across country. Among the traditionally produced distillates in Slovakia, plum spirit (“slivovica”) is well known, the production of which dates to the 17th century. This fruit spirit is characteristic not only for Slovakia, but also for Eastern, Central and Western Europe. In individual regions, it differs mainly in the variety of plums used, the method of preparing the fermented fruits suitable for distillation (destemming, pitting, length of fermentation), which may affect the presence of selected contaminants [1]. Traditional Slovak plum spirit most often contains 52 – 55 % alcohol by volume (ABV).

The International Agency for Research on Cancer (IARC) has classified alcoholic beverages and ethanol in alcoholic beverages as carcinogenic for humans (Group 1, IARC monograph 2012, volume 96, 100E). Besides ethanol, as the main carcinogen, these beverages may contain other proven or probable carcinogens [2]. Selected carcinogens can affect various target organs in human body through different toxic mechanisms. Currently, it is unclear whether these compounds, in combination with ethanol, can have additive or even synergistic effects in

the process of carcinogenesis. Lead is a common contaminant in these alcoholic beverages. Lead is classified by IARC as possibly carcinogenic to humans (Group 2B, IARC monograph 1987, volume 23, Sup 7). Lead can get into homemade spirits in several ways, directly from the raw materials used, through the production process, or through storage containers [1]. However, the most significant source of lead in legal fruit distillates intended for own consumption is the location of fermented fruit cultivation. Lead occurs naturally in an environment in low concentrations. However, the concentration has increased significantly through anthropogenic activity in certain localities. Some areas of Slovakia, such as Banská Štiavnica, Brezno and Banská Bystrica regions, have high concentrations of lead in agricultural soil [3].

The goal of the study was to analyse lead concentration in traditional plum spirits for own consumption distilled in Slovakia. To investigate the issue, beside chemical analyses of samples, it is essential to know all underlying factors possibly affecting lead levels in the analysed plum distillates. Such comprehensive approach can contribute to understanding the public health significance of lead contamination of fruit distillates intended for own consumption.

MATERIALS AND METHODS

Questionnaire

For a comprehensive assessment of contamination factors, we employed our questionnaire, which was used also in the previous studies. The questionnaire was distributed together with sample containers and included eight questions focused on the place of collection of fermented fruit (proximity to public roads, garden, orchard, etc.), location of fruit collection (specific village or city), fruit preparation (pitting and destemming), duration of fermentation, species of fermented fruit, year of distillation, and planned alcohol content in % ABV in the final product (after dilution). The questionnaire also included informed consent and the option to provide email or telephone contact for further contact in case of interest in the results [4, 5].

Samples

We analysed 22 samples of legal plum spirits intended for own consumption. Samples were dis-

tilled in official licensed craft fruit distilleries located across middle and eastern Slovakia (Table 3) and were obtained through prior cooperation with the owners of the fermented fruits. The oldest sample originated from 2012, and the newest one from 2025. Containers (bottles) used to collect samples were soaked in 10% nitric acid for 24 hours and afterwards washed twice with ultrapure water (Type 1 up H₂O) with a minimum resistivity of 18.2 MΩ cm. The ethanol content was determined by alcoholometric tables.

Instrumentation

Sample preparation commenced mineralization using a Multiwave 60 50 Hz. The plum spirit samples were diluted with up H₂O to a 10 % alcohol solution, which was then mineralized using the microwave decomposition system manufacturer (Multiwave 60 50 Hz) (Table 1). High-performance reaction vessels with pressure-activated-venting for routine and quality control applications made of PTFE-TFM with a volume of 40 ml were used for mineralization. Immediately prior to mineralization, 15 ml total volume samples were prepared, composed of 10 ml trace metal grade (TMG) HNO₃ and 5 ml of the 10 % plum distillate. As warned by the manufacturer, even at this 10 % ABV, alcohols can strongly react with HNO₃, even in the cold. Therefore, the mixture must pre-react under a fume hood before the vessel is closing and the microwave decomposition is performed, or a safer preparation technique should be employed.

Table 1 Digestion program of plum spirit for MULTI-WAVE 60 50 Hz

Step	Ramp time (mm:ss)	Temp. (°C)	Hold time (mm:ss)	Fan
1	20:00	130	0:01	1
2	05:00	180	5:00	1
3	-	70	-	3

The samples were analysed by Graphite Furnace Atomic Absorption Spectrometry GBC XplorAA 5000 instrument equipped with GF 5000 graphite furnace. We utilized pyrolytic-surface graphite cuvettes and employed the deuterium lamp background correction method. For the specific lead analysis, a hollow cathode lamp was used, set to a wavelength of 217.0 nm and a lamp current 5 mA. The temperature program used for the lead determi-

nation by GF AAS is presented in Table 2. This program, established by the instrument manufacturer, was modified to ensure measurement of lead in the presence of HNO₃. Argon was used as the inert gas at 300 ml/min (drying and ashing) except during the atomization step, the flow was stopped and 2.600 ml/min while cleaning condition.

Chemicals and reagents

In the analysis we used ultrapure water Type 1 (up H₂O) with minimum resistivity of 18.2 MΩ cm. Other used chemicals included nitric acid (trace metal grade – TMG HNO₃) and standard (Sigma-Aldrich: Pb) for AAS with concentration of lead 1000 ± 4 µg/l. The dosing volume of the sample was 20 µl. As the modifier, we used a mixture of palladium nitrate (0.05 %) and magnesium nitrate (0.03 %) in a volume of 5 µl according to the device manufacturer's recommendations. The standard for lead was diluted to 26 µg/l (max. recommended concentration by AAS manufacturers). From standard solutions, two additional calibration solutions with a quarter and a half concentration - three-point calibration - were programmed in the measuring device for calibration. Blank was prepared from ethanol for

Table 2 Graphite furnace temperature program for the study of lead in plum spirits

Nr.	Final Temp. (°C)	Ramp Time (s)	Hold Time (s)	Gas Type
1	-	-	-	-
2	40	2.0	1.0	Inert
3	80	5.0	5.0	Inert
4	110	10.0	5.0	Inert
5	400	10.0	5.0	Inert
6	400	1.0	1.0	None
7	2000	1.5	2.0	None
8	2200	1.0	1.0	Inert

analysis (same mineralization procedure as for samples). The concentration is expressed in µg/l. Limit of detection (LOD) and limit of quantification (LOQ) were different for each sample considering various dilution level (to achieve the same ethanol concentration before mineralisation).

RESULTS

Lead was detected in nineteen samples of plum spirits (Table 3). In three cases, the concentration was lower than the limit of detection (LOD).

Table 3 Results for Pb concentration in plum spirits

Sample No.	Year of distillation	Plums source (district)	Plum cultivation	Ethanol (% ABV)	Pb (µg/l)
1	2022	Martin	garden	52.3	<LOQ (LOQ = 45.35)*
2	2024	Trnava	orchard	52.1	57.35 ± 3.90
3	2023	Stropkov	orchard	57.6	49.96 ± 3.18
4	2014	Banská Bystrica	garden	51.3	<LOQ (LOQ = 44.43)*
5	2018	Banská Bystrica	garden	59.8	<LOQ (LOQ = 51.86)*
6	2025	Martin	garden	53.6	52.90 ± 4.95
7	2024	Snina	garden	55.4	<LOQ (LOQ = 48.06)*
8	2024	Ružomberok	orchard	50.7	<LOQ (LOQ = 43.97)*
9	2024	Martin	garden	49.1	<LOQ (LOQ = 42.58)*
10	2016	Trenčín	garden	51.7	<LOQ (LOQ = 44.80)*
11	2019	Prešov	garden	51.9	<LOQ (LOQ = 44.98)*
12	2023	Banská Bystrica	orchard	51.1	55.85 ± 1.65
13	2023	Ružomberok	garden	49.1	<LOQ (LOQ = 42.58)*
14	2023	Banská Bystrica	orchard	49.3	328.63 ± 57.12
15	2019	Púchov	garden	49.9	<LOQ (LOQ = 43.23)*
16	2022	Martin	garden	52.3	<LOQ (LOQ = 45.35)*
17	2024	Prešov	orchard	52.9	<LOD**
18	2017	Prešov	orchard	46.6	<LOD**
19	2022	Martin	garden	52.3	105.26 ± 2.46
20	2012	Prešov	garden	39.6	<LOQ (LOQ = 34.29)*
21	2025	Banská Bystrica	orchard	52.5	<LOQ (LOQ = 45.53)*
22	2024	Svidník	garden	50.7	<LOD**

Legend: * <LOQ – lower than the limit of quantification (LOQ value for sample), ** <LOD – lower than the limit of detection

Table 4 The margin of exposure (MOE) of lead in homemade plum spirits calculated for different contamination scenarios and drinking pattern

Scenario 1: one standard drink per day (low risk drinking guideline for females)		Scenario 2: heavy drinker (four standard drinks per day)	
MOE average contamination	MOE maximum contamination (worst case)	MOE average contamination	MOE maximum contamination (worst case)
41.2	6.4	10.3	1.6

The highest concentration of lead was found out in the sample number 14 from Banská Bystrica ($328.63 \pm 57.12 \mu\text{g/l}$) and plum spirit number 19 from Martin ($105.26 \pm 2.46 \mu\text{g/l}$) districts. The majority of fruit distillate samples ($N = 13$) had levels below the limit of LOQ. The average concentration of lead in our samples was $49.53 \mu\text{g/l}$. The average concentration of ethanol was 51.32 % ABV (Tab.3).

The analysis did not show statistically significant associations between lead concentration and the fermented fruit treatment (stemming and pitting) or the alcohol content (% ABV). The highest lead concentration (328.63 ± 57.12) was found in a sample from Banská Bystrica, the area known for significant lead contamination in agricultural soil. However, to confirm this association, it is necessary to analyse an expanded number of samples from this specific location.

For the risk assessment of lead intake from homemade plum spirit, we used the Margin of Exposure method (MOE) according to Lachenmeier et al. [2, 6, 7].

Table 4 shows MOE for two different exposure scenarios (one and four standard drinks per day) in combination with the average and maximum lead concentration in our samples. If the MOE value exceeds 10,000, the health risk from the substance is low. Values below 10,000 indicate a possible concern.

DISCUSSION

Regarding comparative risk assessment using the margin of exposure (MOE) methodology for lead, our results are consistent with other studies [7] indicating that lead contamination of plum spirits intended for own consumption is a widespread problem. Currently, there is no legislative regulation in the Slovak Republic that determines a maximum permissible level of lead in fruit spirits intended for own consumption. Therefore, the starting point for evaluation of the level of lead in fruit spirits intended for own consumption is the limit recommended by the AMPHORA project (Alcohol

Measures for Public Health Research Alliance) i.e. 0.2 mg/l [8]. As demonstrated by several studies, lead is a relatively frequent contaminant in fruit spirits (recorded and unrecorded), with considerably varying concentration [7, 9, 10, 11]. Differences in lead concentration in fruit spirits may be caused by several factors, such as the environment in which the fermented fruit is grown (soil composition, use of agrochemicals and pollution of the growing location), the method of fermentation, distillation and storage of the final products [1,10]. In our study, we did not demonstrate statistically significant differences in lead content in relation to the above factors. The results of the questionnaire show that 73 % ($N = 16$) of the fruit was pitted, 86 % ($N = 19$) of the fruit was destemming. Regarding the duration of fermentation, the majority of respondents (64 %; $N = 14$) reported a duration of fermentation of 2 – 3 months. Regarding the locality of cultivation, the highest lead concentration (328.63 ± 57.12) was recorded in the sample originating from the Banská Bystrica District, which is characterized by higher lead contamination of agricultural soil [3]. However, in case of two other samples from this locality, higher lead concentrations in plum distillate were not recorded (55.85 ± 1.65 ; $<\text{LOQ}$). To demonstrate this relationship, it is necessary to perform an analysis with a larger number of samples from the given region. None of our samples were from areas near public roads, and growers reported growing in orchards ($N = 8$; 36 %) or in their own gardens ($N = 14$; 64 %). Similarly, no statistically significant correlation was found between lead concentration and ethanol level. This may be due to the relatively high similarity of ethanol concentration (most samples had concentrations in the range of 49 – 53 % ABV, with a mean concentration of 51 % ABV).

CONCLUSIONS

Our results indicate that the lead is a significant contaminant of plum spirit. As it occurred in most of samples, regardless of other underlying factors, we can assume its widespread occurrence, i.e. each

one plum spirit intended for own use should be considered as potentially contaminated and thus having health effects. Moreover, considering the high popularity of plum spirits, having a long tradition in Slovakia, the issue seems to be a significant public health problem. Taking into account insufficient epidemiological data on this issue, our findings represent a significant insight to it as well as important starting point for further systematic investigation.

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