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Assessment of major and trace element bioavailability in vineyard soil applying different single extraction procedures and pseudo-total digestion

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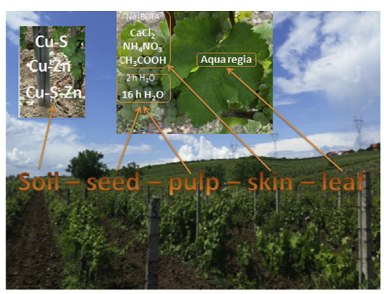
HIGHLIGHTS

- NH_4NO_3, $CaCl_2$, and 16 h water extracted significantly correlated content of Cu and S.
- The content of bioavailable elements in soil correlated with those in grapevine parts.
- Parts of Prokupac species show no similarities between element concentrations.
- The grapevine leaves indicate Zn and Cu content in the vineyard soil.

GRAPHICAL ABSTRACT



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ABSTRACT

A different single extraction procedures (CH_3COOH, Na_2EDTA, $CaCl_2$, NH_4NO_3, deionized water), and pseudo-total digestion (aqua regia) were applied to determine major (Al, Fe, K, Mn, Na, P, S, and Si) and trace (Cd, Co, Cr, Cu, Mo, Ni, Pb, V, and Zn) element bioavailability in a topsoil from the experimental vineyard ("Radmilovac", Belgrade, Serbia). For the first time, the extraction with deionized water during 16 h was tested as an alternative method for isolating bioavailable major and trace elements from the soil. Concentrations of the elements were determined by inductively coupled plasma – optical emission spectroscopy (ICP-OES). The extraction of Cu and S from the soil by deionized water during 16 h extracting, NH_4NO_3, and $CaCl_2$ indicated that these elements could originate from the anthropogenic sources, such as fungicide. In addition according to the soil – plant experiment, performed as a preliminary experiment for future studies in vineyards, deionized water was recommended for isolation of bioavailable elements from grape seed and grape pulp; CH_3COOH, Na_2EDTA, $CaCl_2$ and NH_4NO_3 for grape skin, while for assessment of leaf bioavailable elements from soil fraction, aqua regia was recommended. In addition, identification of similarities between the plant parts and the plant species were performed. Applying environmental risk assessment formulas, the most polluted vineyard parcel in the vineyard region "Radmilovac" was determined. The leaves of some grapevine species showed the high ability for accumulation some of the potentially toxic trace elements from the soil.

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1. Introduction

Soil could be contaminated by pollutants originating from different natural or anthropogenic sources, such as toxic elements released from wastewater, sewage sludge, chemical fertilizers, pesticides, solid waste disposal, vehicular exhaust, etc. (Khan et al., 2008; Islam et al., 2015). In the vineyard soils, the major impact of the soil pollution could be caused by the potentially toxic elements (especially Cu and Zn) from the chemical fertilizers and pesticides (Kabata – Pendias and Mukherjee, 2007; Tiecher et al., 2016). The presence in increase concentration of trace elements in soils can cause a potential risk to human health because of their subsequent involvement into the food chain by plant uptake (Radha et al., 1997; Alonso Castillo et al., 2011; Sakan et al., 2011; Islam et al., 2015). Translocation of major and trace elements from soil to plant has a major impact on the plant growing and quality of its products. Human activities can increase the content of pollutants, up to phytotoxic. Trace and other elements in soils may affect human health through the inhalation of dust, ingestion of soil, or by dermal contact (Morel, 1997; Kabata - Pendias and Kabata, 2001; Hazrat et al., 2013; Sylvain et al., 2016).

Research on element bioavailability of contaminated agricultural soil, monitoring and management attracted attention at international level and it has been ongoing for more than 20 years (Ruby et al., 1996; Pelfrêne et al., 2012). With an aim of determination of element bioavailability to plant and metal availability in the environment from the 1980s to now, there are many publications based on developing and applying procedures of single and sequential extraction procedures (Quevauviller, 2002). Nowadays many procedures are included in national and international regulations or they have been considered in the framework of normalization bodies as CEN or ISO (Quevauviller et al., 1996). In German protocol, 0.1 mol L^{-1} NH_4NO_3 is the standard used to estimate mobile and potentially hazardous forms of trace elements (DIN 19730) (Hall et al., 1998). In Dutch legislation, 0.01 mol L^{-1} $CaCl_2$ is the standard used for the assessment of nutrients and heavy metals in soils (Pueyo et al., 2004).

Many single extraction procedures are recommended for isolation of major and trace elements from agricultural soils. For example, aqua regia and Na_2EDTA procedures were useful for determination environmental risk assessment while 0.05 mol L^{-1} Na_2EDTA procedure was usually useful for agricultural and environmental studies. The weak extractants such as 0.01 mol L^{-1} $CaCl_2$ and 0.1 mol L^{-1} NH_4NO_3 or deionized H_2O were usually applied for soil – plant uptake studies. In addition, aqua regia and 0.01 mol L^{-1} $CaCl_2$ were used to determine soil fertility (McGrath, 1996; Quevauviller et al., 1996; Pueyo et al., 2004). These single extractions are a simple and cost – effective way to extract elements, and they give detailed information on the assessment of the labile elements in soils (Meers et al., 2007; Rao et al., 2008; Santos et al., 2010).

Because of its stringent safety precautions, usually used HF procedure is not recommended for routine analyses (Sastre et al., 2002; Sandroni et al., 2003). In addition, the hardly available soil fraction of the elements is unavailable to plants. Moreover, HF could react with Ca, and it could form Ca–F complexes which could trap trace elements (Marina et al., 2008). An alternative to this procedure is pseudo-total digestion by aqua regia (US EPA, 1996). The European Community Bureau of Reference has certified several soil and sediment samples based on it (Quevauviller et al., 1993; Chen and Ma, 2001; Relić et al., 2011).

According to the available literature, there is no a comprehensive study comparing performances of these five above – mentioned extraction procedures. For the first time, the extraction using deionized water during 16 h was performing as an alternative single extraction procedure. In this study, five single extractions (CH_3COOH, Na_2EDTA, $CaCl_2$, NH_4NO_3, deionized water) and pseudo-total digestion procedures were used for extraction of major and trace elements from the vineyard soil. In addition, for the first time, it was tested which extraction procedure should be the best option for determining bioavailability elements soil fractions which have an influence on different grapevine parts. The aims of the study were to assess:

1) a suitability of using deionized water for 16 h as an alternative procedure to the other single extraction procedures;
2) which single extraction procedure is the best for extraction of major and trace elements from the vineyard soil;
3) whether concentrations of major and trace elements in the soil are in relationship with their content in the grapevine (leaf, seed, pulp, and skin); and
4) environmental risk assessment of major and trace elements in the agricultural area such as a vineyard.

2. Materials and methods

2.1. Study area

In this study, soil samples were collected from the vineyard "Radmilovac", Serbia during the grapevine season of 2013. "Radmilovac" is a suburban settlement of Belgrade, (44° 45′ 24″ N; 20° 34′ 54″ E), the capital of Serbia. The experimental agricultural area of "Radmilovac" is the original core of the neighborhood and it covers an area of 88 ha. The vineyard parcels are located between the Institute of nuclear research "Vinča", the hazardous waste landfills and the main road. The institute landfill is oriented towards the vineyard parcels of agricultural area "Radmilovac" (Fig. 1).

2.2. Sampling

Surface soil layer (0–30 cm), was collected from nine different vineyard parcels, marked as tables T1, T2, T3, T4, T5, T6, T10, P and K. In each parcel, the soil samples were taken as the composite samples of 10 subsamples sampled along the diagonal. The samples T2 and T4 were collected from the central part of vineyard area. The P table is situated close to the local stream. Tables T1, T5, and K are without grapevine plants. Table K was used as a control sample and to determination of the initial values of the measured elements (Fig. 1).

In the vineyard, seven different grapevines (*Vitis vinifera* L.) species are planted. They were located by the order: T2 – *Riesling Rain* and *Burgundac*, T3 – *Cabernet Sauvignon* and *Riesling Italian*, T4 – *Prokupac* and *Cabernet Sauvignon*, T6 – *Cabernet Franc*, T10 – *Cabernet Franc* and *Merlot*, P – *Cabernet Franc*. For need of the pilot study, the grape leaf and grape were sampled from each table (parcel).

2.3. Chemical analyses

The experiment was designed to determine the behavior of the content of 17 major and trace elements in the vineyard soil, grapevine (seed, pulp, and skin) and grape leaves samples. All chemicals used in the experiment were of analytical grade and produced by Sigma – Aldrich.

2.3.1. Soil sample preparation

Soil samples were stored at the room temperature in the laboratory. Each soil sample was air-dried, sieved through 2 mm