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SILPHIUM PERFOLIATUM A PROMISING ENERGY CROP FOR PHYTOREMEDIATION OF HEAVY METAL CONTAMINATED SOILS

Violina ANGELOVA, Vera KOLEVA

Agricultural University - Plovdiv, 12 Mendeleev Blvd, Plovdiv, Bulgaria

Corresponding author email: vileriz@abv.bg

Abstract

Comparative studies have been carried out to determine the quantities and depositions of heavy metals, macro, and microelements in the vegetative organs of Silphium perfoliatum, the efficiency for phytoremediation and the quality of the biomass as a renewable energy source for the combustion process. The field experiment was conducted on an agricultural field contaminated by the Non-Ferrous Metals Plant near Plovdiv, Bulgaria. Silphium perfoliatum is tolerant to heavy metals and can be grown on highly contaminated soils (1671.6 mg/kg Zn, 1694.8 mg/kg Pb, and 54.8 mg/kg Cd). Pb, Zn, Hg, Cu, Fe, Mn, Ca and Mg accumulated in leaves, Cd and P - in roots, and K in stems. Cup plant can be classified as a exluder plant with bioconcentration factor < 1 and can also be used in phytoremediation of contaminated soils. Silphium perfoliatum can be a significant source of good-quality raw material in producing solid biofuels. The biomass studied is of high quality (high in C and H and low in ash, N, Cl, and S) and has high energy potential. Biomass of Silphium perfoliatum from highly contaminated soils could be used as a source of energy.

***Key words**: cup plant, polluted soils, proximate and ultimate analysis, combustion.*

INTRODUCTION

The cup plant (*Silphium perfoliatum* L.) is a honey-bearing herbaceous perennial plant of the Asteraceae family. The plant was brought to Europe from North America in the 18^{th} century because of the plant's ornamental value. *Silphium perfoliatum* L. is cold hardy (withstanding winter temperatures down to minus 30-35°C), disease and pest-resistant, undemanding to soils, tolerant to droughts, and can be grown effectively throughout Europe (Gansberger et al., 2014). Due to its phenotypic plasticity, it can be cultivated on degraded soils (Zhang et al., 2010; Gansberger et al., 2014). In the first year, it forms a 60-70 cm diameter rosette of leaves, and in the 2^{nd} year, the plant forms numerous flowering stems up to 1.5 m tall, which secrete a resinous sap with a strong odor similar to that of turpentine. It has many leaves (8-14 pairs of opposite leaves with an area of 85-120 cm^2) arranged alternately at 10-18 cm (Peni et al., 2020).

The biomass can be harvested twice during the growing season (Cumplido-Marin et al., 2020; Stolarski et al., 2020). The plant is characterized by a high regrowth ability maintained for up to 15-20 years. Sunflower-like yellow flower heads form in branched corymbs.

Phytoremediation is a process by which plants effectively remove heavy metals by absorbing them from the contaminated substrate (Chaney et al., 1997). It is desirable for plants to have a rapid growth rate with high biomass production potential, a deep and branching root system, and to be tolerant to biotic and abiotic stress. Recommended plant species for heavy metal extraction should have translocation factor and bioaccumulation factor values above 1. *S. perfoliatum* L. is one of the proposed species for remediation of heavy metal contaminated soils as it has an intensive growth rate, is tolerant to biotic and abiotic stress, produces a significant amount of plant biomass that can be harvested and used in biogas production, and direct combustion (Sumala et al., 2020; Nescu et al., 2022; Sumalan et al., 2023).

Figas et al. (2015) indicate that the plant can potentially rehabilitate degraded land through phytoremediation. Preliminary studies suggest cup plant is a good candidate for phytoremediation of Cd-impacted soils (Zhang et al., 2010). However, according to Sumalan et al. (2023), *Silphium perfoliatum* is a potential hyperaccumulator.

The purpose of this study is to conduct systematic research that will allow us to determine the quantities and the deposits for accumulation of heavy metals, macro- and microelements in the vegetative organs of cup plants, the quality of biomass as a biofuel, as well as the possibilities to use the plant for phytoremediation of heavy metal contaminated soils.

MATERIALS AND METHODS

The experiment was carried out on an agricultural field contaminated with Zn, Pb, and Cd, located 0.5 km from the source of contamination - a non-ferrous metals plant (NMP) near Plovdiv, Bulgaria. The soil used in this experiment was slightly alkaline (pH 7.6) with moderate in organic matter (2.5%). The total Zn, Pb, and Cd contents were high (1671.6 mg/kg Zn, 1694.8 mg/kg Pb, and 54.8 mg/kg Cd, respectively) and exceeded the maximum allowable concentrations (MAC) (400 mg/kg Zn, 100 mg/kg Pb, 3.0 mg/kg Cd) (Table 1). The Hg content in soils is lower than the MAC.

Table 1. The total content of Pb, Zn, Cd (mg/kg), and Hg (ng/g) in soil sampled from NFMW-Plovdiv

Element	Pb	Zn	Cd	`Hg
x±sd	1671.6±3.1	1694.8±3.5	54.8±0.9	574.8±10

x - average value (mg/kg) from 5 repetitions; sd - mean standard deviation
MAC (pH >7.4) - Pb - 100 mg/kg, Cd - 3.0 mg/kg, Zn - 400 mg/kg, Hg - 1.5 mg/kg

The field tests with the cup plant were set after the block method in four replications. The size of the test plot was 100 m^2. Plants were planted in May by planting the seedlings at 0.75 m intra-row and inter-row spacing. Whole plants (3 plants from each replicate) were taken for analysis in mid-November before frost fall.
The plants were collected, and the content of heavy metals and macro and trace elements in their different parts - roots, stems, and leaves, was analyzed separately. The samples were dried at room temperature to obtain an air-dry mass and then dried at 105°C.
The total metal composition of the soils was determined according to ISO 11466.
Plant samples were processed by the microwave mineralization method. An inductively coupled emission spectrometer (Jobin Yvon Horiba "ULTIMA 2", France) was used to determine the content of heavy metals, micro- and macroelements in plant and soil samples.
Heating value, ultimate and proximate analysis, are among the main parameters in the evaluation of biomass in the direct combustion process.
Proximate analysis. The samples were characterized according to standard methods: moisture content (EN 14774-2:2009), ash (EN 14775:2009), fixed carbon (by difference), and volatile matter (EN 15148:2009).
Ultimate analysis. Total C, H, N, and S were determined by dry combustion in a Vario Macro CHNS analyzer (Elementar Analysen systeme GmbH, Germany), according to the protocols (EN 15104:2011) and (EN 15289:2011). The O content was calculated by difference.
Heating value. The heating value was determined by the ISO method (EN 14918:2010) using an IKA C200 oxygen bomb calorimeter (IKA Analysentechnik GmbH, Heitersheim, Germany).

RESULTS AND DISCUSSIONS

Accumulation of heavy metals in vegetative organs of cup plant

Table 2 presents the results obtained for the content of heavy metals in the vegetative organs of the study energy crop.
Significant differences were found in the content of the elements in the different parts of the cup plant. Pb, Zn, Hg, Cu, Fe, Mn, Ca and Mg accumulated in leaves, Cd and P - in roots, and K in stems.
Sumalan et al. (2023) found good absorption and selective accumulation capacity of cup plant. Cu and Zn mainly accumulate in the stems, Cd in the roots and stems, while Pb primarily accumulates in the roots.
The heavy metal contents of Pb, Zn, and Hg were lower compared to the leaves, while the opposite trend was found for Cd. The Pb content of the roots reached 42.1 mg/kg, Zn - 172.1 mg/kg, Cd - 20.3 mg/kg, and Hg- 41.0 µg/kg. The contents of micro- and macro elements (except P) were also lower in the root system.

Table 2. Content of heavy metals, macro- and microelements in vegetative organs of cup plant

	Roots	Stems	Leaves
Pb, mg/kg	42.1	40.3	268.2
Cd, mg/kg	20.3	0.66	3.3
Zn, mg/kg	172.0	73.7	291.0
Cu, mg/kg	12.0	13.5	51.4
Fe, mg/kg	261.6	77.9	681.7
Mn, mg/kg	27.8	20.2	120.2
P, mg/kg	1438.3	383.2	847.2
Ca, mg/kg	2487.1	4758.2	28052.6
Mg, mg/kg	1064.0	726.2	2710.7
K, mg/kg	5760.7	8589.4	6016.5
Hg, µg/kg	41.0	23.0	215.9

A probable reason for this is that the root system of the cup plant is fibrous, consisting of a central main root that reaches a penetration depth of 80 cm and shallow rhizomes that help the vegetative spread of the plant.

However, studies conducted by Fitzgerald et al. (2003) have shown that the level of heavy metal accumulation in plants is directly related to their concentration in the soil and that the root system of plants is their main storage organ. Similar results were also obtained by Sumalan et al. (2020), who found that heavy metal accumulation in *S. perfoliatum* was higher in the roots compared to the leaves in the early stages of vegetative growth. The amount of metals absorbed depends on the metal content of the soil as well as the developmental stage of the root system. The content of heavy metals and micro and macro elements in the stems was lower compared to the root system (except K), which showed that their movement through the conductive system was strongly restricted. The content of Pb in the stems of cup plants grown at 0.5 km from NFMW reached 40.3 mg/kg, Zn - 73.7 mg/kg, Cd - 0.66 mg/kg, and Hg - 23.0 µg/kg.

The movement of Pb from the roots to the aboveground parts of plants is typically limited. Once Pb enters the plant's roots, it promptly interacts with phosphates, carbonates, and bicarbonates in high concentrations within the intercellular spaces. This interaction causes Pb to precipitate as phosphates or carbonates, preventing its transportation through the xylem (Kabata Pendias, 2001).

Cd is a very mobile element that moves from the roots to the aboveground mass, but this was not observed in our studies. Probably, Cd is mainly bound to the cell walls and stored in vacuoles of the root cortex and not transported to the shoots (Zhang et al., 2010).

The content of Pb in the leaves of cup plants grown at 0.5 km from NFMW reached 268.2 mg/kg, Zn - 291.3 mg/kg, Cd - 3.25 mg/kg, and Hg -.215.9 µg/kg. A probable reason is that the leaves of the cup plant are rough, hairy, large, and deeply feathery with a coarse texture, which is a prerequisite for their aerosol contamination. The accumulation values and heavy metal concentrations in *S. perfoliatum* plants during rosette leaf formation are significantly lower than those for hyperaccumulators (Sumalan et al., 2020), which is confirmed by obtained results. Studies by Sumalan et al. (2023) showed that Zn and Pb mainly accumulate in leaves. The ability of the aboveground mass to accumulate greater amounts of heavy metals compared to the root system has been confirmed by Nouri et al. (2009) on various species of the Asteraceae family grown on contaminated soils.

Translocation factor and bioconcentration factors were calculated to determine the phytoremediation potential of the plant.

The translocation factor (TF) provides information on the ability of plants to absorb heavy metals through the roots and translocate them to the aboveground mass (stems and leaves). For Pb and Hg, the TF values are more significant than 1 (6.37 and 5.27, respectively). for Zn about 1 (1.69) and for Cd less than 1 (0.16). TF>1 were also found by Sumalan et al. (2020), which follow the order Pb>Zn>Cr. TF>1 for plants growing on soils contaminated with heavy metals were also found by Zhang et al. (2010) and Nescu et al. (2022).

The bioconcentration factor (BCF) determines the effectiveness of phytoremediation. BCF root is a ratio of the content of heavy metals in roots to soil content (BCF roots = [Metal] roots/[Metal] soils). The results show that BCFroots reaches up to 0.025 for Pb, 0.37 for Cd, 0.10 for Zn, and 0.07 for Hg.

BCFshoot is defined as the ratio of the metal concentration in the aboveground mass of the plant (stems and leaves) and in the soil (BCF shoot=[Metal] shoots/[Metal] soils) and is a measure of the plant's ability to absorb and move the metals to the aboveground mass, which can be easily harvested.

The results show that the BCFroot for Pb reaches 0.16, Cd from 0.09 to 0.26, Zn 0.17, and Hg 0.38. A plant is an excluder if BCF < 1, an accumulator if 1 < BCF < 10, and if BCF > 10, the plant is a hyperaccumulator.
The study results show that the cup plant can be classified as an excluder since BCF is less than 1. This confirms the results obtained by Zhang et al. (2010) which found that *S. perfoliatum* is a Cd excluder.

Table 3. Translocation factor (TF) and bioconcentration coefficients (BCF roots, BCF shoots) of cup plant

Coefficient	Pb	Cd	Zn	Hg
TF	6.37	0.16	1.69	5.27
BCF root	0.025	0.37	0,10	0.07
BCF shoot	0.16	0.069	0.17	0.38

BCFroot=[Metal]shoots/[Metal]soil, TF=[Metal]shoots/[Metal]roots, BCFshoot=[Metal]shoot/[Metal]soil

The cup plant accumulates a small amount of heavy metals in the leaves and has no potential for phytoextraction. This is in contradiction to the results of Sumalan et al. (2020), according to which *S. perfoliatum* has the potential to bioaccumulate heavy metals in soils contaminated with Cu, Zn, Cr and Pb Mockeviciene et al. (2023) found an accumulative capacity of the cup plant concerning Cu and Zn, while the BCF (for Cr, Ni and Pb) was relatively low, indicating that the plant could only absorb but not accumulate these heavy metals.

Heating value, ultimate and proximate analysis

Moisture content (MC), ash content (AC), volatile matter (VM), nitrogen (N), sulphur (S) and oxygen (O) are undesirable components in biomass, in contrast to fixed carbon (FC), carbon (C), hydrogen (H) and lower heating value (LHV), whose higher levels improve biomass quality when it comes to direct combustion. The lower heating value (LHV), moisture and ash contents depend on the harvesting time of *Silphium perfoliatum*, while the higher heating value (HHV), C, H and S contents are less affected by these factors (Stolarski et al., 2018).
The results obtained for the plant biomass of cups grown on heavy Metal-contaminated soils are presented in Tables 4 and 5. The tables also present the values of the solid fuels standard EN ISO 17225-6 for non-wood pellets and data from the analysis of cup plant biomass grown in different countries.

Table 4. Proximate analyses and lower heating value of cup plant biomass

Parameter	Moisture %	Ash, %	FC, %	VM, %	LHV, MJ/kg
Cup plant	9.1	6.1	16.56	77.34	16.49
Reference	3.84-13.0	2.04-9.22	9.28	77.45	15.7-16.61
Standard	≤12	≤6-10	-	-	≥14.5

Table 5. Ultimate analysis of Miscanthus x giganteus biomass

	N, %	C, %	S, %	H, %	O, %	Cl, %
Cup plant	0.846	40.5	0,052	5,7	46.03	0.074
Reference	0.37-0.68	42.94-45.44	0.02-0.07	5.28-5.30	38.57-50.92	0.026
Standard	≤1.5	-	≤0.2	-	-	≤0.1

The moisture content is influenced by the environmental conditions, in particular by, temperature, and humidity, but also by soil properties (soil type and altitude), genetic characteristics, the applied agrotechnique (mainly fertilization), as well as the development and phenophase of the plant (Kowalska et al., 2020). The results show that the moisture content reaches 9.1%, which are in accordance with the standard EN ISO 17225-6 (≤12). The moisture content of the biomass varies from 3.84% to 13.0% (Fraczek et al., 2011; Bury et al., 2020; Stolarski et al., 2020).
The ash content is one of the main factors determining biomass quality. In direct combustion, it is desirable to have as low ash content as possible since ash has no calorific value and thus reduces the efficiency of the combustion system. The results show that the ash content reaches up to 6.1%, which is in accordance with the standard for solid fuels EN ISO 17225-6 (6 to 10%). The ash content in cup plant biomass varies from 2.84% to 14.54% (Stolarski et al., 2004; Fraczek et al., 2011; Wever et al., 2019; Bury et al., 2020; Suric et al., 2022).
Higher values of fixed carbon affect biomass quality due to the higher heating values. The fixed carbon content is calculated by the formula Fixed Carbon (%) = 100 - Ash (% Dry Basis) - Volatile Matter (% Dry Basis).

The results show that the fixed carbon varies reaching 16.56%. Significantly lower results were obtained by Suric et al. (2022) (9.28%).

Volatile matters are the components released at high temperatures when the fuel is heated, without considering the moisture that is part of the combustible gases (CxHy gases, CO or H_2) and non-combustible gases (CO_2, SO_2 or NO_x) (Garcia et al., 2012). The results obtained show that volatile matter reaches up to 77.34%.

Heating values decrease with higher moisture content, with higher levels leading to lower combustion temperatures and affecting quality (Garcia et al., 2012). The net calorific value of biomass depends on the chemical composition, moisture content, and carbon (C), hydrogen (H) and ash contents, with moisture content being the main cause of variation in results.

The results show that the net heat of combustion on a dry basis reaches up to 16.49 MJ/kg. According to this parameter in the EN ISO 17225-6 standard, the biomass from cup plant is characterised as a valuable energy raw material suitable for use in the combustion process. Similar results for LHV for cup plant biomass were found by Stolarski et al. (2020) (15.7 MJ/kg) and Bury et al. (2020) (16.61 MJ/kg). The calorific value of cup plant biomass is in the same range as the energy crop *M. x giganteus* (Stolarski et al., 2020). Carbon is the main and most important element in all types of fuels, and its content determines their quality, i.e. higher carbon levels increase fuel quality. Cup plant biomass contains carbon in the range of 42.0-51.9%, indicating a high potential for thermal energy. 51.9% (Peni et al., 2020). Similar results were obtained in this research (40.5% carbon).

Hydrogen, together with nitrogen, forms the basic fuel composition of any fuel, and increased hydrogen content improves fuel quality by positively affecting oxygen levels. Peni et al. (2020) found H content of 5.75%, which agrees with the obtained results (5.7%). The oxygen content is calculated by the formula Oxygen (%) = 100 - Carbon (% Dry Basis) - Hydrogen (% Dry Basis) - Nitrogen (% Dry Basis) - Sulphur (% Dry Basis) - Ash (% Dry Basis). The oxygen content reaches up to 46.03%. According to the Siaudinis et al. (2015) and Suric et al. (2022) the content of O varies from 38.57% to 50.92%.

From an environmental point of view, N and S contribute to the increase of greenhouse gases and are considered unfavourable elements in biomass. The content of N reaches up to 0.846% and of S up to 0.0524%. The values obtained are below the permissible values in the standard for solid fuels EN ISO 17225-6 (N is less than 1.5%, and for S is 0.2%). The low levels of S in the cup plant, is an indicator of the potential of the plant for direct combustion.

Chlorine content reaches 0.074%, values below the permissible values in the standard for solid fuels EN ISO 17225-6 (<0.1%). Lower values were also found by Peni et al. (2022) (0.026%).

CONCLUSIONS

The cup plant is tolerant to heavy metals and can be grown in heavy metal polluted soils (1671.6 mg/kg Zn, 1694.8 mg/kg Pb, and 54.8 mg/kg Cd), and can be successfully used in the phytoremediation of heavy metal polluted soils. Significant differences were found in the content of the elements in the different parts of the cup plant. Pb, Zn, Hg, Cu, Fe, Mn, Ca and Mg accumulated in leaves, Cd and P - in roots, and K in stems.

Cup plant can be classified as an excluder plant with BCF < 1. The moisture content of biomass in cup plant is low and within the standard.

Ash content as an indicator of fuel quality is also low, highlighting the good quality of the biomass studied.

The cup plant biomass studied is of high quality (high in C and H and low in ash, N, Cl, and S) and has high energy potential.

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