DETERMINATION OF HEAVY METALS IN WOODEN TREES AND ASH RESIDUES IN KOSOVO

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Abstract. In this study, we examined the content of Fe, Mn, Cd, Pb, Cu, Zn and Ni in fresh wooden trees and ash wood residues. The material of analysis was sampled in four different zones in Kosovo. Initially, the samples of wood were grinded and dried in room temperature, and then they were treated with HNO_3 and H_2O_2. The content of heavy metals was determined with atomic absorption spectrophotometer technique. The following results show that we have higher concentrations of some elements such as Cu, Zn and Ni, in the ash residues more than the Environmental Protection Agency standard (EPA Ecological Soil Screening Level) predicts for these elements to be as wastes in the soil. In order to preserve our health and environment, we must be cautious towards the amount of waste we dispose in the ground, for the reason that they straightforwardly end up in our food chain.
Keywords: *environmental, pollution, atomic absorption spectroscopy, EPA*

Introduction

The environmental problem in Kosovo in recent years has been a big and very serious problem. According to various measurements and numerous reports, our country was ranked among the world's first countries in terms of pollution. The Hydrometeorological Institute dealing with environmental monitoring in Kosovo has cited that the main causes of pollution are industries, transport and fossil fuels. According to this Institute, the air pollution has been increasing so drastically in the five last years that in some cases the permissible values have been exceeded (Kosovo Environmental Protection Agency, 2017)

Today it is a known fact through different researches that accumulation of heavy metals is related to different industrial activities, such as mining, smelting, energy and fuel production, traffic activities, agriculture etc. (Balabanova et al., 2014; Mooreet al., 2013; Yan et al., 2012; Kouamé et al., 2013; Jusufi et al., 2017). In our day, what is also known is that plants uptake these metals from various reactions: redox and ionic exchange reactions, precipitation-dissolution etc. (Mirecki et al., 2015). It is impossible to imagine life without trees because of their role in economic, environmental and industrial aspects, as well as spiritual, historical and aesthetic (Seth, 2004). However, in terms of environmental pollution, trees can also serve as long term accumulators of heavy metals and act as bio indicators of contamination.

In this paper, we evaluate the chemical distribution of Cd, Mn, Pb, Cu, Ni, Zn and Fe in fresh wood and their ashes. The importance of determining the scale of contamination in ashes is enormous, since the possibility of wood ending up in environment as ash residue is huge. Also, knowing the forms of toxic metals present in wood ash could indicate how much the emitted particulates pose risks to the human health and the environment (Odlare et al., 2007; Pettersson, et al., 2008).

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Materials and methods

Area of study

Kosovo forests cover around 41 % (U.S. Department of State, 2008; Knaus and Warrander, 2010) of the entire territory of the country. Most of the forests are located in the southwest. There are various types of forestry in Kosovo, but for the most part are conifers. The regions with the highest diversity are the regions of the Sharr Mountains and the Albanian Alps. Kosovo's forests are threatened by fires and illegal logging. For this study, we chose four different regions in Kosovo where wood samples were collected.

Treatment procedure

In order to measure the heavy metal content, samples were inspected in fresh wood and calcinated form. The types of wood were beech and oak. The wood samples were mangled in fine parts before being grinded.

Initially, wood samples were left to dry at 60 °C for several hours before further study. For the analysis, 1 g of dried wood up to constant weight were taken and were placed in Teflon containers, where 12 ml of concentrated nitric acid (65%) was added. The mixture was heated at 105 °C and in small portions 4 ml of hydrogen peroxide (30%) were introduced to the mixture. The samples were left to evaporate until drying, then 1 ml of HCl were added. The mixture was filtered and leveled up to 50 ml with distilled water.

To treat the wood ash, fresh wood samples were initially calcinated at 400 °C for about two hours. 0.5 grams of calcinated sample were taken and placed in Teflon containers. 6 ml of HNO_3 were added and placed in heater at 105 ° C. Additionally, 2 ml of H_2O_2were added and after the sample was dried, it was removed and 0.5 ml HCl were added. The mixture was filtered and leveled up to 25 ml with distilled water.

Concentrations of heavy metals (Fe, Mn, Cd, Pb, Cu, Zn and Ni) were measured using the atomic absorption spectroscopy (AAS) technique.

Results and discussion

The purpose of this study was to determine the heavy metal content in wood which is burnt in conditions similar to domestic. A study of this form is very important since there are different studies of heavy metal uptake in vegetation (Peng et al., 2006; McLaughlin et al., 2011; Mingorance etal., 2007), but there are few data which examine the concentration of heavy metals in wood (Algreen et al., 2012; Świetlik et al., 2013).

The bioaccumulation of heavy metals in trees happens owing to climate, atmospheric depositions, the concentrations of heavy metals in soil, the nature of soil on which the trees are grown and the degree of maturity of the plants at time of harvest (Lake et al., 1984; Voutsa et al., 1996). Wood ash is one of the inorganic residues of wood burning, whereas CO_2 and water are the organic remains (Hannam et al., 2002; Reimann et al., 2008). The ash contents vary among types of trees, soil and climate. Campbell (1990) and Naik (2003) pointed out that it can also vary according to the method and manner of combustion, efficiency of the boiler, and other supplementary fuels used with wood.

Table 1 presents a summary of the wood samples: the ash content in percentage, moisture and the wood type.

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Table 1. *List of analyzed samples and their properties*

Sample	Regions	Ashes %	Moisture	Type
1	Drenica	<0.9	7.4	Beech/oak
2	Ferronickel	<0.7	8.58	Beech/oak
3	Sharrcem	<0.8	6.96	beech
4	Sharr	<0.6	11.88	beech

The moisture was determined by weight loss and was used to correlate the results with dry weight, while the ash content was calculated before and after the burn, and was left at 105 °C to constant weight. Studies show that the percentage of ash content in wood can vary from 0.1 to 1.4% (Fengel and Wegener, 1989; Prosiński, 1984; Rowellet al., 2005; Szczepkowski and Nicewicz, 2008).

Table 2 presents the following basic statistical data of dry wood samples in mg/kg: average (AVG), range (min-max), standard deviation (SD), variance, first and third quartiles (Q1 and Q3) and median.

Table 2. *List of analyzed elements in fresh wood*

Elements	AVG (mg/kg)	Min-max	SD	Variance	Q1	Q3	Median
Mn	400.475	(20.4-1440)	694	481034	29	1102	71
Fe	105.81	(0.149-172)	72.1	5191.2	40.6	171	81.1
Zn	19.6	(10.05-39)	11.52	132.67	11.4	27.8	16.6
Pb	5.26	(0.05-18.9)	9.98	99.54	0.25	18.9	1.65
Cu	4.96	(0.6-10.85)	4.96	24.58	1	10.85	7.02
Ni	0.812	(0.05-2.55)	1.284	1.649	0.074	2.55	0.55
Cd	0.748	(0.4-0.943)	0.246	0.061	0.488	0.932	0.825

The concentrations of heavy metals are ranked in descending order of average concentration as follows: Mn>Fe>Zn>Pb>Cu>Ni>Cd. The average concentration of Zn is 19.6 mg/kg and ranges from 10.05-39 mg/kg. The results for lead and copper in wood content were similar, ranging from 0.049 to 18.9 mg/kg for lead with the average of 5.26 mg/kg, and the average for copper was 4.96 mg/kg, whereas it ranged between 0.6 to 10.85 mg/kg. The lowest concentration of heavy metals was found for Ni and Cd, with the average of Ni being 0.812 mg/kg and the average of Cd 0.75 mg/kg.

Table 3 presents the following basic statistical data of ash residue samples in mg/kg: average (AVG), range (min-max), standard deviation (SD), variance, first and third quartiles (Q1 and Q3) and median.

The mobility of an element, the form it occurs in the environment and other conditions determine whether that element is likely to enter the food chain. Highly mobile elements such as Mn or Zn are mainly taken up by the root system and are then transported from there to the leaves. Concentration of manganese was shown to be very high, and it can be explained as a result of the high content of this easily assimilative element in soil (Szczepkowski and Nicewicz, 2008). In our study it ranges from 5.99 to 2596 mg/kg. Kuokkanen et al. (2009) also found higher concentrations of this element.

If we compare the average values of the concentrations of heavy metals, we find that their order in ash samples is Fe> Mn> Zn> Cu> Ni>Pb> Cd. The global average of

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cadmium content in soil is 0.41 mg/kg (Kabata-Pendias and Mukherjee, 2007), while in the surface layer of soil in Europe the average concentration of cadmium concentration is 0.15 mg/kg (Salminen et al., 2005). The standard of soils (Dutch list) defines the target concentration of Cd in soil to be 0.8 mg/kg. According to ecological soil screening levels established by USEPA (2003) (Eco-SSL) that serves for soil contamination, the maximum concentration of Cd is 32 mg/kg. In our samples, Cd ranges from 0.95 mg/kg to 7.85 mg/kg with an average of 4.51 mg/kg. By this definition of EPA, cadmium in our samples results to be in tolerated concentration.

Table 3. *List of analyzed elements in ashes residue*

Elements	AVG mg/kg	Min-max	SD	Variance	Q1	Q3	Median
Mn	978.25	(52.99-2596)	1192	1420019	67	2236	632
Cd	4.51	(0.95-7.85)	2.84	8.05	1.77	7.14	4.63
Pb	42.25	(32.35-48.05)	7.32	53.51	34.54	47.91	44.3
Cu	87.5	(38.25-195)	72.9	5308.6	40.5	163.6	58.4
Ni	58.8	(36.3-82.4)	20.2	407.4	39.6	78.6	58.3
Zn	229.39	(0.05-670)	300	89907	26	538	124
Fe	2095.013	(0.05-4705)	1956	3825547	393	4055	1838

The concentration of Cu in our samples ranges between 38.25 to 195 mg/kg, with an average of 87.5 mg/kg. The Dutch list defines the intervention concentration of Cu in soil to be 190 mg/kg. Eco-SSL classifies Cu as an element that should not exceed the concentration of 70 mg/kg when discharged on the soils. If we compare the average concentration of this element in ashes 87.5 mg/kg, we see that the maximum amount of copper is exceeded by almost 13%.

Zinc is an element with relatively low toxicity. In general, zinc toxicity is restricted to taking high doses. The global average of zinc content in soil is 90 mg/kg (Xie et al., 2000), while in the soils in Europe is around 48 mg/kg (Salminen et al., 2005). In our research for ash content of heavy metals, the average concentration of Zn is 229.39 mg/kg, ranging between 0.05 and 670 mg/kg. Eco-SSL maximum value for Zn is 160 mg/kg, thus it is over 40% higher than what EPA recommends.

The significance of nickel to the living organisms has been revealed in the last decades of the last century, bearing in mind by that time that nickel was considered to be toxic metal to the organism. Since then, this element is considered as “potential essential” for humans, although its role in the organisms is still relatively unknown. According to Kabata-Pendias and Mukherjee (2007), the global average content of Ni in soil is 29 mg/kg, while in the surface layer of soil in Europe is 18 mg/kg (Salminen et al., 2005). According to EPA’s program issued for *ecological* soil screening levels, nickel should not exceed the amount of 38 mg/kg. In our ash samples it ranges from 36.3 to 82.4 mg/kg with the average of this metal being 58.8 mg/kg. As a conclusion, we see that the concentration of nickel is higher by 20 mg/kg.

For the concentrations of Pb we can say that we have lower concentration than the standard Dutch list. This list sets the maximum allowed concentration of Pb to 530 mg/kg. However, the comparison of these elements is different, because Pb is recognized as a much more toxic and dangerous element even in lower quantities. According to Kabata-Pendias and Mukherjee (2007) the average concentration of Pb in

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soils of the world is 27 mg/kg, while in surface layer of soils in Europe is 23 mg/kg (Salminen et al., 2005). Eco-SSL defines the maximum concentration of lead as a discharged element to be 120 mg/kg. In our samples it varies from 32.35 mg/kg to 48.05 mg/kg.

It should be noted that these metals in the soil often come from various anthropogenic and natural pollutants, so their concentration is not dictated exclusively by ash residues. However, to avoid increasing the concentration of these metals, the most appropriate and ecofriendly methods should be identified.

To compare the results of heavy metals in fresh wood samples and their ash forms, a diagram is presented in *Figure 1*.

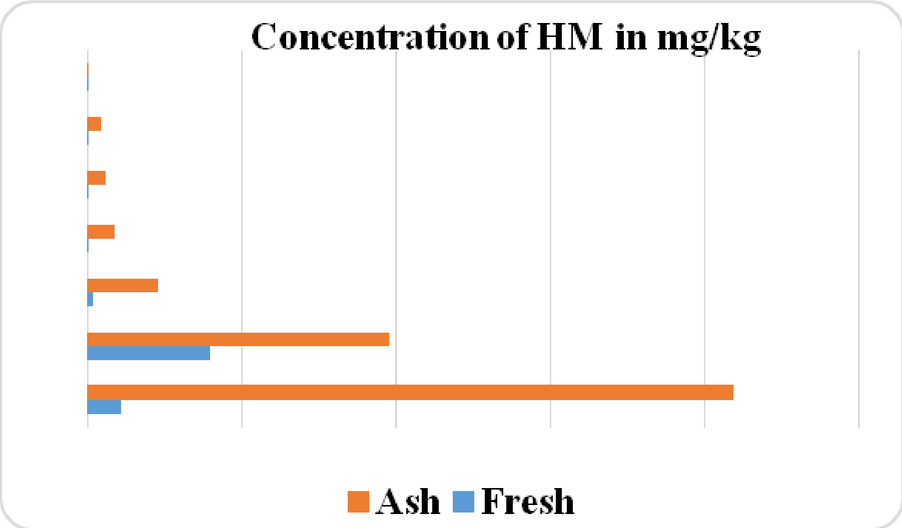

Figure 1. *Comparison of heavy metals in fresh and ash residues in woods*

In all cases we have a higher concentration for Zn, Fe, Mn, Ni, Cu, Pb and Cd. This is justified by the fact that ashes contain precisely these elements. Algreen et al. (2014) found similar results as our average results for Cd, Cu, Zn and Ni.

Correlation analysis

Table 4 shows the analysis of the similarity correlation of elements: Cd, Mn, Pb, Cu, Ni and Zn in measured areas. The statistical calculations were done by using the software Minitab 18.

Kosovo, though a rather small place in territory, is known as a rich country for many minerals. These elements in the environment can come from natural reserves, but anyway the anthropogenic influence has an impact on increasing their concentration.

From the correlation analysis it can be inferred that we have similarity sources of Mn and Pb in with more than 99% and Cd with Ni of about 99%. This shows that these elements occur as natural resources in Kosovo, but these elements can come also due to anthropogenic atmospheric pollution. On the other hand, we have a fairly large similarity of Fe with Cu, which stands at about 75 %.

Figures 2–7 show distribution of heavy metals in fresh wood and in their ash samples. The histograms show the frequency of different concentration brackets expressed in mg/kg.

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Table 4. *Correlation analysis elements*

	Cd	Mn	Pb	Cu	Ni	Zn
Mn	-0.944					
	0.056					
Pb	-0.928	0.994				
	0.072	0.006				
Cu	-0.675	0.822	0.774			
	0.325	0.178	0.226			
Ni	-0.988	0.981	0.974	0.73		
	0.012	0.019	0.026	0.27		
Zn	0.344	-0.187	-0.079	-0.401	-0.244	
	0.656	0.813	0.921	0.599	0.756	
Fe	0.085	-0.234	-0.148	-0.741	-0.117	0.576
	0.915	0.766	0.852	0.259	0.883	0.424

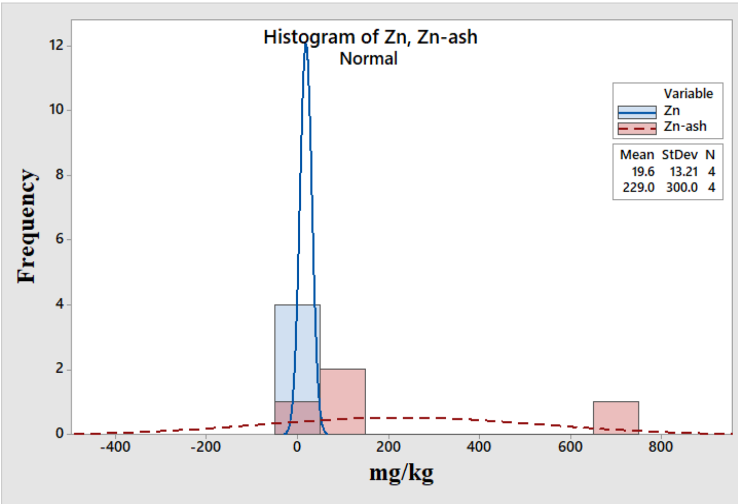

Figure 2. *Distribution of Zn in wood and ashes in mg/kg*

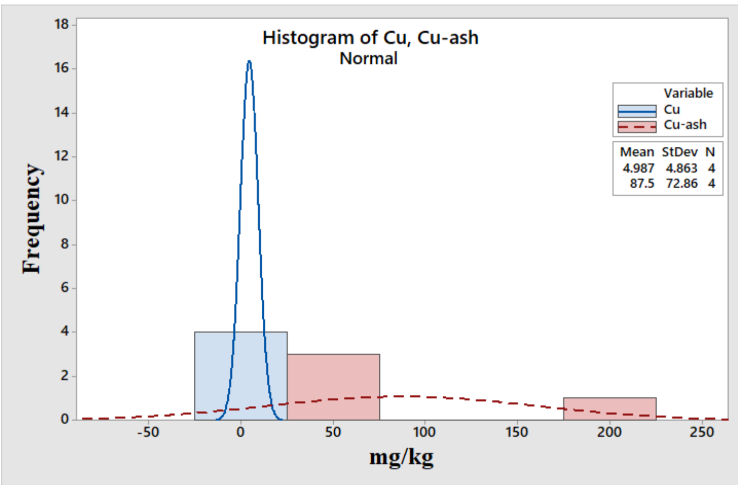

Figure 3. *Distribution of Cu in wood and ashes in mg/kg*

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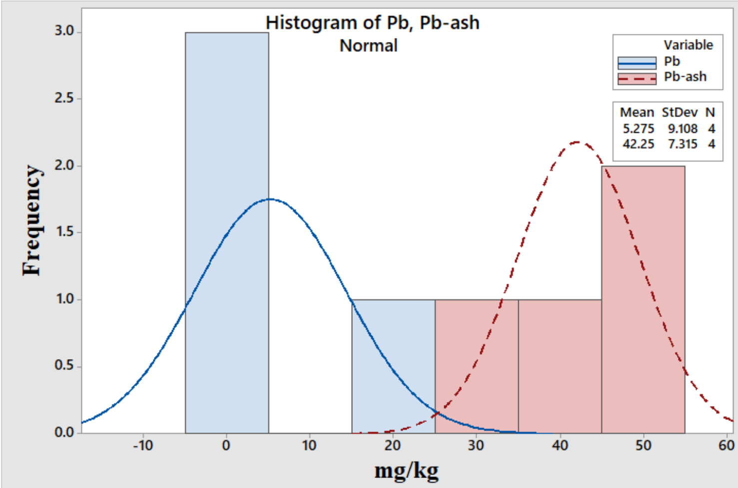

Figure 4. *Distribution of Pb in wood and ashes in mg/kg*

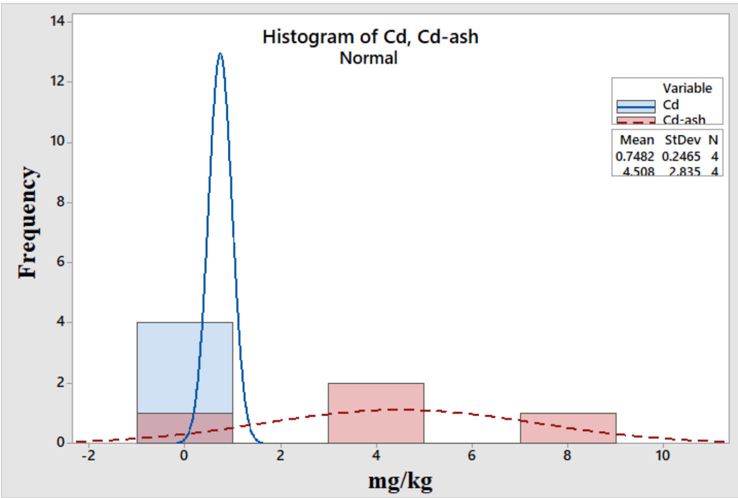

Figure 5. *Distribution of Cd in wood and ashes in mg/kg*

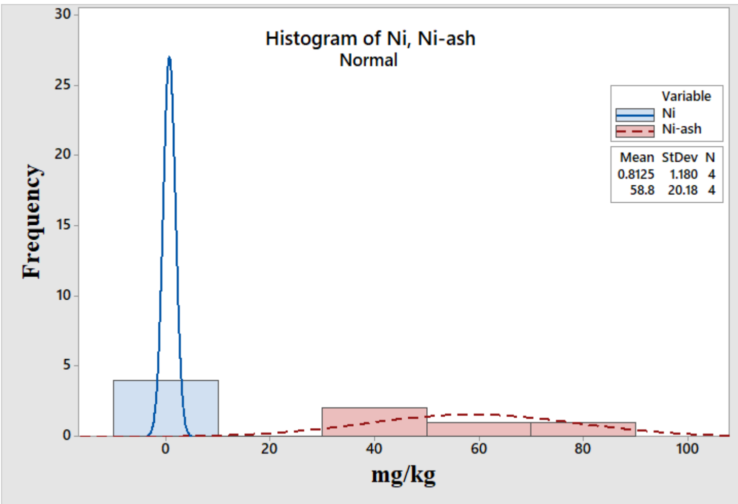

Figure 6. *Distribution of Ni in wood and ashes in mg/kg*

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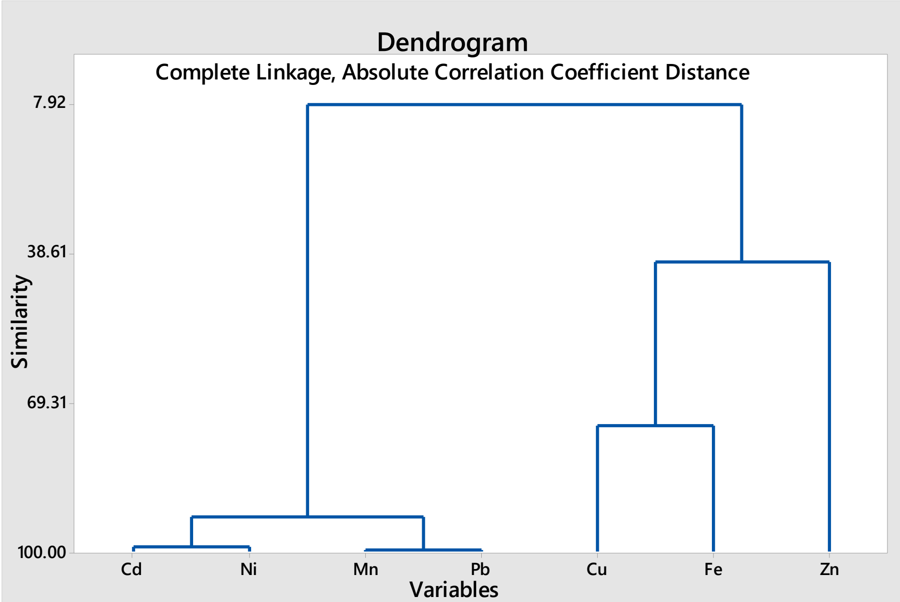

Figure 7. *Dendrogram showing the similarities of elements*

If we compare the results between fresh wood samples and ash residues, we see a significant difference in the concentration of heavy metals. In the case of manganese, its concentration is more than two times higher in ashes than in fresh wood; whereas in Fe, the concentration in ash residue is almost 20 times higher. In zinc, copper, lead, nickel and cadmium we also have significant increases in the concentration of heavy metals in ash samples. A part of these large increases in concentration can be explained by the fact that ashes themselves contain precisely these heavy metals.

Conclusion

The aim of this paper is the examination of the concentration of heavy metals in wood samples and in their ash form. In Kosovo, such a research is very important due to the many environmental problems and mismanagement of waste. In this country, the ash waste is typically disposed of in the ground, water or atmosphere without any of the necessary precautions. In this study, we have analyzed heavy metals and the consequences that can come from improper disposal of ash in the environment. The concentration of heavy metals is slightly higher than foreseen by the relevant standards due to pollution and lack of institutional control in these areas.

The presence of heavy metals in the ash samples from this study can lead to possible water contamination and environment in general.

In order to avoid environment pollution from ash, it is suggested to choose wood from unpolluted areas, free of contaminants, and the wood to undergo complete and careful burning.

If these conditions are met, then this ash can be used in various fields: regulation of pH levels in soil, heavy metal adsorption, remediation of pollutants in various forms, use in construction etc. But under the current conditions with the high concentrations of heavy metals, it is necessary to identify methods of prior treatment before considering the appropriate way of reusing the ash residue.

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