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Aqueous extraction of anions from coal and fly ash followed by ion-chromatographic determination

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Abstract: Three different techniques were applied for the aqueous extraction of anions from coal and fly ash: rotary mixer- and ultrasonic-assisted extraction with different duration time, and microwave-assisted extraction at different temperatures. Validation showed that the ion-chromatographic method was suitable for the analysis of anions in coal and fly ash extracts. The variations in the amounts of anions using different extraction times during rotary-assisted extraction were minimal for all investigated anions. The efficiency of ultrasound-assisted extraction of anions from coal depended on the sonication time and was highest at 30 min. The ultrasound-assisted extraction was less efficient for the extraction of anions from fly ash than rotary-assisted extraction. Increase of temperature in the microwave-assisted extraction had a positive effect on the amounts of all anions extracted from coal and sulphate from fly ash, while the amounts of fluoride and chloride in fly ash extracts decreased. The microwave-assisted extraction of coal at 150 °C was compared with standard ASTM methods, and results were in good agreement only for chloride. Changes in the pH value and conductivity during ultrasound-assisted extraction were measured in order to explain changes on the surface of coal particles in contact with water and different processes that occur under environmental conditions.

Keywords: coal; fly ash; anions; extraction; ion chromatography.

INTRODUCTION

Coal preparation, as well as disposal of coal fly ash, can lead to introduction of various materials into the environment through leaching process and cause water, air and soil pollution. Some novel research indicated that chlorine and fluorine in fly ash are enriched on the surface and that both halogens on the ash

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surface consist of organic and inorganic functionalities.^{1,2} The monitoring of sulphur, phosphorus and the halogens is of great importance for a number of reasons: with the exception of bromine, all of these elements are essential for the life of humans, animals and many plants; however, in elemental form and/or in certain compounds, they are highly toxic. All of the above-mentioned elements are vastly used in the chemical industry and, consequently, their effects on human health and the environment are important.³⁻⁵

The quantitative determination of halogens and sulphur in solid samples usually involves sample preparation (in order to bring the analytes into solution) followed by analytical determination. Sample decomposition is a critical step in halogen analysis. Halogens are volatile and readily lost when the sample is treated; furthermore, the reagents used for sample digestion may interfere with the determination process itself. The alternative to decreasing the time spent in performing analyses is the employment of ultrasonic baths or microwave ovens in the digestion or extraction process. The details related to coal, coal fly ash and extraction processes are given in Supplementary material to this paper.

The aim of this study was the extraction of fluoride, chloride and sulphate from coal and coal fly ash under aqueous conditions. Different extraction techniques were employed to extract anions from coal and the efficiencies of those techniques were investigated by ion chromatographic determination. In addition to the comparison of the efficiency of various types of extractions, the influence of extraction time was investigated with respect to the rotary and ultrasonic extraction, as well as, the influence of temperature during microwave-assisted extraction. Deionised water was used as a soft extraction solvent because of its hydrolytic and dissolving effect on some coal and fly ash constituents. Trace elements extracted by water are relatively labile and thus may be potentially bio-available. This phase contains water-soluble species consisting of free ions and ions complexed with soluble organic matter and other constituents. It constitutes the most mobile and potentially the most available metal, non-metal and metalloid species.⁶ Studies of drainage waters from coal mines and fly ash dumps showed that, depending on the pH value, they can contained Ca, Mg, Fe, Al, Si and trace elements, such as Mn, Zn, Ni and Co, and that the most important anions are sulphate, hydrogen carbonate and carbonate.⁷⁻⁹

EXPERIMENTAL

Materials and reagents

Composite samples (coal and fly ash) from Kostolac coal field, Serbia (see Supplementary material for details) and power plant were ground, homogenized and sieved through a sieve with 0.212 mm openings, corresponding to US standard mesh 70 and Tyler standard mesh 65. The samples were oven-dried at 105 °C overnight prior to use.

The working calibration solutions were prepared by serial dilution (10, 20, 50, 100 and 200 times) of the stock standard solution using deionised water (for details on reagents used, see Supplementary material).

Instrumentation

An Overhead Mixer Reax 20/8 (Carl Roth, Germany) rotary mixer and a Transsonic T 760 DH (Elma, Germany) ultrasonic bath with an ultrasonic frequency of 40 kHz and an effective ultrasound power of 170 W were used. The type of the microwave digestion system used for coal sample preparation was ETHOS 1, Advanced Microwave Digestion System (Milestone, Italy), equipped with 10 PTFE containers. The capacity of the containers was 100 cm³ each, with a maximum pressure and temperature of 10 MPa and 240 °C, respectively.

Ion chromatography (IC) measurements were realised using a Metrohm 761 compact IC system (Metrohm, Switzerland). The A Metrosep A supp 1 ion-exchange column, with polystyrene–divinylbenzene copolymer as the carrier material (particle size 7 µm, column dimensions 4.6 mm×250 mm) was used for the anion separation, followed by the Metrohm suppressor module and then a thermostated conductivity detector. As eluent 3.0 mmol dm⁻³ Na₂CO₃ was used at a flow rate of 1.0 cm³ min⁻¹. The full-scale range was 50 µS and the injected sample volume was 20 µL for each probe.

Extraction procedures

The extraction mixtures were prepared by mixing coal or fly ash sample with deionised water in the mass/volume ratio of 1 g:10 cm³. The extracted anions were identified and determined using ion chromatography.

Extractions were performed using a rotary mixer (RAE) in which the suspension was processed for the following extraction times: 30, 60, 90, 120 and 180 min by mixing at 10 rpm at room temperature (20 °C). The second technique involved the use of an ultrasonic bath (ultrasound-assisted extraction, UAE) with the mixtures being positioned at the same place in the bath and at the same initial water temperature of 17 °C. Extractions were made during extraction times of 10, 20, 30, 40 and 50 min. The third technique involved the use of a microwave system (microwave-assisted extraction, MAE) at the following temperatures: 50, 100 and 150 °C. The given temperature was achieved in 15 min for each extraction cycle, while the extraction itself lasted 15 min. Subsequently, the samples were cooled to room temperature.

Five replicate extractions of each sample were made and the results presented are the average value. A blank was prepared for each of the employed extraction procedure. When the extraction processes were finished, each of the extraction mixtures was first centrifuged and then filtered through a 0.22 µm pore size membrane filter. The extracts were preserved at 4 °C in a laboratory refrigerator for further analyses.

RESULTS AND DISCUSSION

Validation of IC measurements are given in Supplementary material to this paper.

Determination of anions in coal

The important inorganic anions in coal (fluoride, chloride and sulphate) were found as coal inorganic constituents and as pollutants. The chromatogram of a standard solution and a typical chromatogram for the coal extract are shown in Fig. 1.

The sulphate peak is well distinguished from the fluoride and chloride peaks at a considerably longer retention time of 13.24 min. All the analysed coal samples contained trace levels of fluoride, and therefore fluoride was detected in the aqueous extracts of the coals. Under the conditions used for the IC measurements (Fig. 1), fluoride is distinguished from acetate, which is also present in the extracts. The identification of acetate, to which the peak 2 on Fig. 1 (lower) is assigned, was made by spiking the measured extract with sodium acetate solution. The assignment of the fluoride peak is based on the confirmation of retention time by spiking the samples extract with a known quantity of fluoride, and by detection and quantification of fluoride using an ion selective electrode (ISE).¹⁰ The quantification of the fluoride content in the extracts using ISE was in good agreement with the results obtained by IC. The level of extracted fluoride appears to be greater for low-rank coals. Fluorine in coal is mostly associated with mineral matter, usually fluorapatite and clays, but fluorite and complex silicates, such as tourmaline, may also be present.¹¹ These minerals are finely dispersed throughout coal and it is feasible that the extraction process enables fluoride to be leached from these sparingly soluble materials. The results of the determinations of fluoride, chloride and sulphate in the coal extracts obtained using RAE and UAE are presented in Table I.

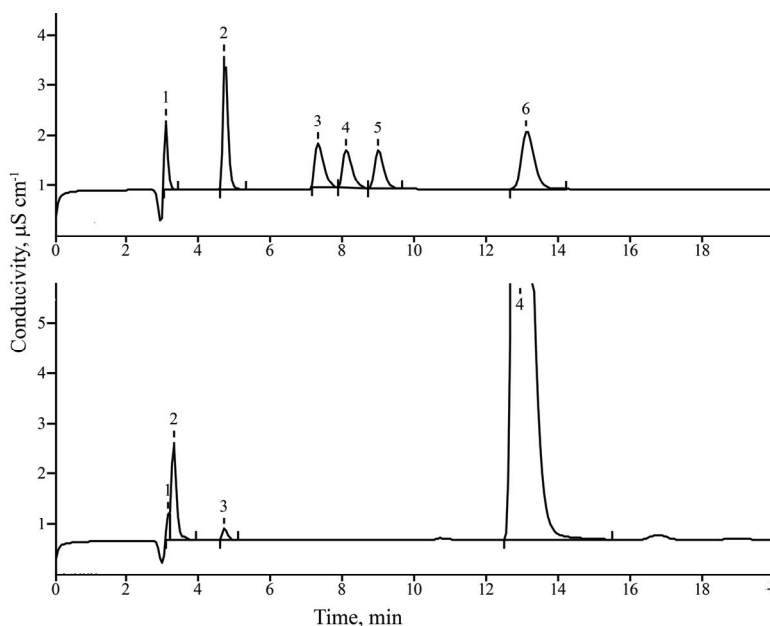


Fig. 1. Ion chromatograms of: (upper) standard solution (1 - F⁻, 2 - Cl⁻, 3 - NO₃⁻, 4 - Br⁻, 5 - PO₄³⁻, 6 - SO₄²⁻) and (lower) coal extract provided by rotary-mixing (1 - F⁻, 2 - CH₃COO⁻, 3 - Cl⁻, 4 - SO₄²⁻).

From the results listed in Table I, fluoride concentration was almost constant with time during rotary-assisted extraction. Considering ultrasound-assisted extraction of fluoride, the obtained results indicated that after initial increase, the concentration of fluoride in extracts did not change significantly with time. The extraction of fluoride by means of RAE yielded results that were, on average, comparable to the results realized using UAE. In the case of both extraction techniques, there was no positive correlation between the extraction time and the amount of extracted fluoride.

TABLE I. The results of the determination of anions ($\mu\text{g g}^{-1}$) after RAE/UAE ($n = 5$, uncertainty as standard deviation)

Extraction time, min	Fluoride	Chloride	Sulphate
RAE			
30	20.30±0.57	17.00±0.76	3014±89
60	19.50±0.13	19.08±0.12	2822±37
90	20.36±0.18	19.82±0.02	2976±26
120	20.20±0.01	18.60±0.42	3011±2
180	20.50±0.05	21.49±0.01	3016±2
UAE			
10	17.90±0.73	13.58±0.34	2409±89
20	20.42±0.30	19.55±0.21	3330±30
30	20.88±1.07	23.98±0.28	3622±16
40	21.75±0.07	20.44±0.15	3402±22
50	20.74±0.33	20.03±0.10	3080±76

The RAE of chloride (Table I) demonstrated that prolongation of the extraction time led initially to a slight increase in extracted amount between 30 and 180 min. The results presented in Table I indicated that prolongation of the UAE extraction time led initially to an increase in number of extracted ions, first by 43 % and finally by 22 % when the extraction was extended from 20 to 30 min. The highest chloride amount was extracted from coal after 30 min of ultrasound-assisted extraction. When compared to RAE, the UAE was more efficient for the extraction of chloride. The physical and chemical effects of ultrasound do not come from the direct interaction of sound with molecular species. When sound passes through a liquid, the formation, growth, and finally implosive collapse of bubbles occur. Very high effective temperatures (which increase solubility and diffusivity) and pressures (which favours penetration and transport) at the interface between an aqueous solution subjected to ultrasonic energy and a solid matrix, combined with the oxidative energy of radicals (hydroxyl and hydrogen peroxide) created during sonolysis, result in high extractive power.¹² The phenomenon of ultrasonic cavitation caused by implosion of cavitation bubbles and subsequent local pressure increases and elastic shock waves has been used for coal and fly ash extraction.^{13,14}

The results shown in Table I indicated that increasing the extraction time had no significant influence on the RAE of sulphate. The UAE of sulphate (Table I) demonstrated that maximum extracted concentration was obtained after 30 min, and with further increase in the extraction time, the concentration of sulphate decreased. The coal samples from Kostolac coalfield had up to 2 % of sulphur, and the extracted amount was small, meaning that most of the sulphur was insoluble in water.¹⁵ This is expected, since sulphur in coal exists as inorganic and organic sulphur components. The major inorganic sulphur compound in coal is pyrite; organic sulphur compounds are diverse and contain mainly thiol, sulphide, disulfide and thiophene groups.^{15,16}

Changes in pH value and conductivity of the extraction suspension as a function of sonication time are shown in Fig. 2. The results showed that the conductivity of the suspension increased during sonication until 30 min, partly due to an increase in the number of total charged particles in the suspension.

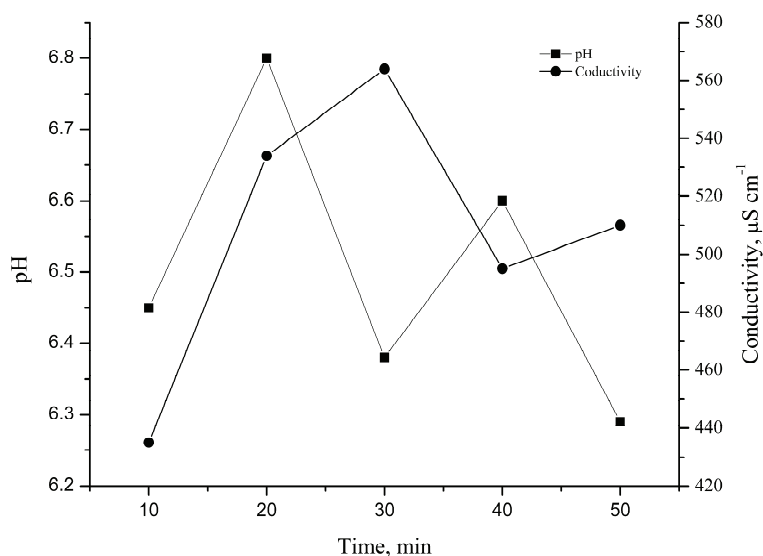


Fig. 2. Changes in the pH value and conductivity of the extraction suspension as a function of sonication time (at 23.6 °C).

Contrarily, a sharp decrease of pH value in the extract was observed for the same sonication time, which implies that hydrogen ions are released to the solution. With prolongation of the sonication time, the conductivity and pH value in the extract changed in the opposite manner. Along with anions, various cations released into the solution also caused the changes in the pH value and conductivity, which could further influence the adsorption and desorption processes to some extent.¹⁷ These processes are mainly related to competitive cation interact-

ions for the sorbing phase and the exchange of sorbed cations with those from solution influenced by ultrasound.

The results of determination of fluoride, chloride and sulphate anions in water leachate from coal using the microwave-assisted technique are shown in Fig. 3. The concentration of extracted fluoride, increased with increasing extraction temperature. The MAE was the most efficient for fluoride extraction, giving at 100 °C an extracted amount that was 1.50 times higher than the ones obtained by RAE and UAE. The MAE at 150 °C resulted in the highest amount of extracted fluoride compared to any of the other extraction techniques used: the amount of extracted fluoride was 2.40 times higher than the average amounts obtained by employing an ultrasonic bath and rotary mixer for all extraction times. The interaction of microwave energy with samples and reagents causes both ionic migration and dipole rotation, resulting in fast heating of the suspension with the consequent acceleration of the physico-chemical processes.¹⁸ The use of microwave energy for coal sample preparation and extraction of elements from coal has increased in recent years mainly due to the heating mechanism, resulting in a shortening of the extraction and digestion times, as well as in a decreased consumption of the extraction agent.¹⁹

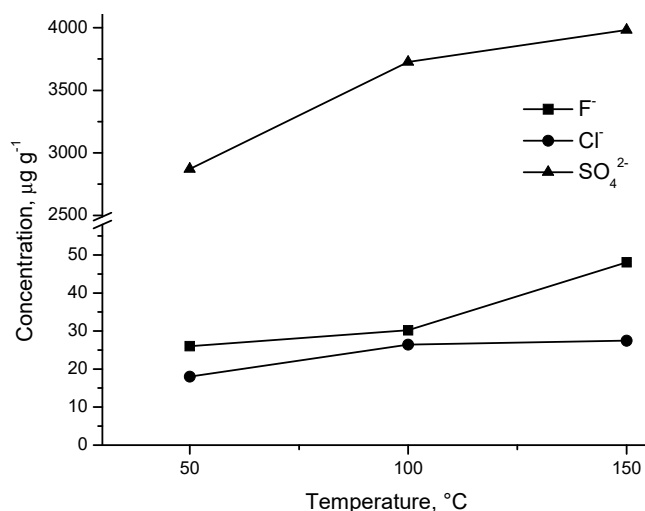


Fig. 3. Change in the extracted amounts of fluoride, chloride and sulphate ($\mu\text{g g}^{-1}$) obtained by MAE as a function of extraction temperature.

The concentration of chloride extracted by the MAE at 100 °C was about 1.1 times higher than the highest concentration obtained by the other two techniques. When the temperature was increased to 150 °C, the obtained concentration was about 1.2 times higher than the highest concentration resulting from the use of

RAE or UAE. The extraction behaviour of chloride depends on temperature and increasing leaching temperatures causes more chlorine to be leached.

The results for MAE (Fig. 3) demonstrated that significantly increased amounts of extracted ions occurred with increasing extraction temperature. The use of MAE at a temperature of 150 °C gave an almost 1.3 time higher amount of sulphate than the average amount obtained by UAE. Compared to all the other investigated techniques, MAE at 150 °C was more efficient for the extraction of sulphate ions. The average value obtained by the MAE was higher than the one obtained by the UAE after the extraction time of 30 min, which gave the highest extracted amount.

Considering the MAE technique, there was, as expected, an obviously positive correlation between the number of extracted ions and the extraction temperature (Fig. 3). The variations in the extracted amount resulting from the use of different extraction techniques were minimized in the case of fluoride.

The results of extracted anions from the coal sample using the MAE technique showed that an increase in temperature produced higher amounts of extracted anions. The microwave-assisted procedures were faster than the rotary mixer and ultrasonic bath procedures. The precision of all techniques was very good with the majority of the *RSD* values below 5 %, thereby demonstrating the good reproducibility of all techniques and indicating that the microwave-assisted procedures offer comparable data quality in terms of reproducibility to the REA and UAE techniques.

The amounts of extracted fluoride and chloride by the MAE at 150 °C were compared to anion content determined according to standard methods.^{21,22} The results are given in Table II as recovery values, calculated as the found to declared concentration ratio. In the case of fluoride, the recovery was satisfactory, but not good. Fluoride has been identified as an ecologically important trace element, and the extracted quantity corresponded to water soluble and exchangeable fluorine. The leaching behaviour of fluorine is pH-dependent: a lower pH value led to fluorine release from the mineral matrix; a higher ion strength or pH value of leaching solution tended to free more fluorine into the aqueous solution.²² The obtained results correspond to the leaching of fluoride under mild pH conditions. On the other hand, fluorine in coal is mostly present as stable inorganic compounds, which means that the proposed extraction method is not suitable for stable inorganic forms of fluorine, such as CaF_2 , MgF_2 or $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})\text{F}$.¹¹ The good recovery for chlorine indicated that the total chlorine content in the analyzed coal was extracted by MAE at 150 °C. The results of leaching showed that chlorine occurs in the investigated coal in various soluble mostly mineral forms. The sulphate content in coal in the absorption solution was also determined after oxygen bomb combustion. Low recovery for sulphate was expected, since the combustion method provides total sulphur analysis, regard-

less the form of the sulphur in coal. The sulphate concentration determined by MAE at 150 °C corresponds to the soluble sulphate in coal. These results indicated that extracted sulphate was present in the analysed coal in less soluble and/or accessible forms than was chloride.

TABLE II. Recovery of fluoride, chloride and sulphate determined by MAE at 150 °C

Anion	Declared ^a , µg g ⁻¹	Found, µg g ⁻¹	Recovery, %
F ⁻	67.60	48.08	71.0
Cl ⁻	26.62	27.44	103
SO ₄ ²⁻	62043	3982	6.40

^aDetermined by the ASTM standard methods

When results for total fluoride, chloride and sulphate are compared to those obtained by UAE for 30 min, the recoveries were 31.0, 90 and 4.96 %, respectively. These indicated that recovery was good for chloride, meaning that UAE could be successfully applied for chloride determination. Previous leaching experiments for chloride showed that about 50 % of chloride could be removed from particles of grain size less than 45 µm, and 30 % from 100–500 µm granulations.²³ The effect of ultrasound on chloride leaching from coal was proven since the efficiency was three times higher for the particles used in the present experiments (smaller than 212 µm). The recovery of UAE leaching of sulphate was in accordance with the results of other authors who investigated the effect of ultrasound on sulphur removal from coal.²⁴

The availability and mobility of anions occurring in coal depended on the physicochemical form of the elements. All three procedures showed relatively good levels of accuracy. Applying these techniques to the extraction of anions from coal samples combined with different extraction solvents, *e.g.* dilute acids or salts solutions, could be considered and requires further investigation.

Determination of anions in fly ash

The results of the determinations of fluoride, chloride and sulphate in the coal fly ash extracts obtained using RAE and UAE are presented in Table III.

Fluoride extraction using RAE showed slight differences between obtained concentrations with time. The maximum extracted quantity was obtained after 60 min of extraction, and further increase of extraction time led to decreases in the concentration. In the case of UAE, the concentration of fluoride after 20 min of extraction was 10 % lower than after 10 min of extraction, probably due to resorption, but subsequently increased and after 50 min, it was the same as after 10 min of extraction. The extraction of fluoride by UAE was almost efficient as RAE for all investigated times except for 60 minutes of RAE.

Extraction of chloride by RAE and UAE followed a similar trend. After the initial extraction, with increasing time, the chloride concentration fell, and then

rose to a constant level. The only difference was for the highest investigated time of extraction. For the rotary-assisted extraction, the maximum chloride concentration was achieved after 180 min. For the ultrasound-assisted extraction, the concentration of chloride extracted after 50 min was the same as that after 10 min extraction. In general, RAE was more efficient for chloride extraction from fly ash than UAE.

TABLE III. The results of the determination of anions in fly ash ($\mu\text{g g}^{-1}$) by RAE/UAE ($n = 5$, uncertainty as standard deviation)

Time, min	Fluoride	Chloride	Sulphate
RAE			
30	29.12±0.04	13.85±0.04	6250±40
60	33.26±0.09	12.48±0.07	7234±100
90	29.42±0.90	14.85±0.30	6893±70
120	26.48±1.50	15.58±0.70	6813±50
180	28.66±0.80	18.14±0.90	6965±55
UAE			
10	30.38±1.20	14.52±0.55	6017±62
20	27.31±0.80	12.76±0.35	6467±97
30	29.62±0.40	13.16±0.20	5690±85
40	29.60±0.85	13.95±0.50	7762±80
50	30.70±0.58	14.12±0.20	7851±44

The concentration of sulphate in the extract increased until 60 min of RAE, and then reached a maximum. Further prolongation of the extraction time led to a decrease in the sulphate concentration, which then remained almost constant. The amount of sulphate anions extracted by UAE gradually increased, but after 30 min, it dropped sharply. Subsequently, the amount of sulphate sharply increased with extraction time and was then almost constant.

Comparing to the extraction of the anions from coal, the concentrations obtained by RAE and UAE indicated that fluoride condensed on fly ash.^{1,25} This was expected since fluorine volatilizes during coal combustion. On the other hand, after combustion, the fluoride in fly ash was present in soluble forms. The amounts of chlorine extracted from fly ash using RAE and UAE were lower than those obtained from coal. During combustion, depending on the conditions, chlorine in coal can form compound with various metals, which are less soluble.²⁵ The sulphate extraction indicated that after combustion, sulphur in fly ash was in soluble forms since the pyrite in coal converts to sulphate. Due to its dominant surface association in fly ash and the marked solubility of most sulphate-bearing compounds, sulphur is the major soluble element in fly ash, along with Ca. Under environmental conditions, reduced sulphur species are generally present in negligible quantities and sulphate is the dominant species.²⁵

The amount of fluoride and chloride extracted using MAE (Table IV) decreased with increasing temperature. This was contrary to results obtained for the MAE of coal. The reason could be the volatility of fluorine and chlorine compounds

TABLE IV. The results of the determination of anions in fly ash ($\mu\text{g g}^{-1}$) after MAE ($n = 3$, uncertainty as standard deviation)

Temperature, °C	Fluoride	Chloride	Sulphate
50	41.36±0.56	35.48±3.70	7731±135
100	35.60±0.90	22.94±3.60	8043±158
150	33.80±2.10	19.54±1.70	8164±114

present in fly ash. The microwave-assisted extraction of fluoride and chloride at 50 °C was more efficient than RAE and UAE. The concentration of sulphate in the extracts obtained by MAE increased with increasing temperature. The extraction of sulphate from coal and fly ash had a similar trend. When temperature changes from 50 to 100 °C, more sulphate was extracted than for a further temperature increase of 50 °C.

CONCLUSIONS

From the obtained results, it can be concluded that the techniques employed in this research as preparation steps of coal and fly ash samples are significant for estimating the concentration of trace elements and pollutants that could possibly be released into the environment.

The results of ion measurements in the extraction solutions of coal by IC were reliable with standard deviations of less than 5 %. The fluoride content extracted using a rotary mixer and an ultrasonic bath were very similar and much lower than the quantity extracted in the microwave-assisted extraction at the highest investigated temperature (150 °C). The ultrasound-assisted extraction of chloride was more efficient than the rotary mixing extraction, and comparable with the microwave-assisted extraction. Sulphate extraction using the ultrasound bath was slightly more efficient than by the rotary mixer, and the assistance of the microwave system did not influence the sulphate extraction significantly. The results of ultrasonic testing indicated that highest extracted quantities of chloride and sulphate were obtained after 30 min. The measurements of pH value and conductivity in the extracts suspensions showed that these parameters changed in the opposite manner after 30 min of ultrasound-assisted extraction. It was concluded that not only the extraction of anions influence these changes but cations extraction as well. Comparing the microwave-assisted extraction at 150 °C to standard ASTM methods, it was found that recovery for fluoride was good, but not satisfactory, for chloride good and for sulphate very low. This led to the following conclusion: a) chloride in coal is present in various soluble mostly mineral forms; b) the applied temperature was not suitable for stable inorganic forms

of fluoride, and c) sulphate in the analyzed coal was present in sparingly soluble inorganic forms and insoluble organic forms. In general, the microwave-assisted procedure showed good performance for coal extraction, safety and high throughput, but further investigations are required to improve the accuracy and precision of the analysis. In general, the rotary-assisted extraction was more efficient for extraction of fluoride and chloride from fly ash than ultrasound-assisted extraction. The optimum extraction time for fluoride and sulphate using rotary-assisted extraction was 60 min, and for chloride, 180 min. Increasing the temperature for microwave-assisted extraction led to increasing sulphate concentrations in the extract, while concentration of fluoride and chloride decreased. Comparing to the extraction of anions from coal, the extraction from fly ash indicated that fluoride, chloride and sulphate condensed on the fly ash.

SUPPLEMENTARY MATERIAL

Additional data and details related to coal, its combustion and extraction processes for solid samples and reagents, as well as validation of IC measurements, are available electronically at the pages of journal website: <http://www.shd.org.rs/JSCS/>, or from the corresponding author on request.

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ИЗВОД

ЕКСТРАКЦИЈА АНЈОНА ИЗ УГЉА И ЛЕБДЕЋЕГ ПЕПЕЛА ВОДОМ И ЊИХОВО ЈОН-ХРОМАТОГРАФСКО ОДРЕЂИВАЊЕ

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Представљене су три технике екстракције анјона из угља и лебдећег пепела помоћу дејонизоване воде: употребом ротационо-механичке мешалице и ултразвука при различитим екстракционим временима, и микроталасне екстракције која је извођена при различитим температурама. Валидацијом је утврђено да је јонска хроматографија метода која је погодна за одређивање анјона у екстрактима испитиваних узорака. Промене количине екстрахованих анјона из угља и пепела, у току времена екстракције, су биле незнатне при примени ротационо-механичког мућкања. Ефикасност ултразвучне екстракције је временски зависна и највећа је за 30 min. Ултразвучна екстракција анјона из лебдећег пепела се показала мање ефикасном од ротационо-механичке. Пораст температуре микроталасне екстракције има позитиван ефекат на количину екстрахованих свих анјона из угља као и на сулфате из пепела, док се количина флуорида и хлорида екстрахованих из пепела са порастом температуре смањује. Резултати микроталасне екстракције анјона из угља на 150 °C су упоређени са добијеним ASTM стандардним методама и у доброј су сагласности само за одређивање хлорида. Промене рН вредности и проводљивости током ултразвучне екстракције су мерене у циљу објашњења промена

на површини честица угља у контакту са водом и различитих процеса који се дешавају у природним условима.

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