

PHOSPHORUS REMOVAL FROM WATER USING FLY ASH AND MODIFIED FLY ASH - COMPARISON OF X-RAY FLUORESCENCE SPECTROMETRY AND STANDARD SPECTROPHOTOMETRIC METHODS

ODSTRAŇOVÁNÍ FOSFORU Z VOD POMOCÍ POPÍLKŮ A MODIFIKOVANÝCH POPÍLKŮ – SROVNÁNÍ RENTGENFLUORESCENČNÍ SPEKTROMETRIE A STANDARDNÍ SPEKTROFOTOMETRICKÉ METODY

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Abstract

This study investigates sorptive properties of three different types of fly ash and one fly ash type treated with a 1M, 3M, and 6M solution of NaOH to remove phosphates from water. In addition, this work examines possibilities of using the Wavelength Dispersive X-ray Fluorescence (WD XRF) spectrometry method for the analysis of phosphorus in liquid samples. The WD XRF method was compared with the standard spectrophotometric method (molybdate method). The results showed that the fly ash containing the highest initial CaO concentration sorbed the greatest amount of phosphorus. The treatment of fly ash with NaOH solutions did not significantly enhanced the sorption capacity of fly ash. The determination of P in fly ash indicated a good correlation between both methods, thus validated the application of XRF for the analysis of P in water.

Abstrakt

Tato práce zkoumá sorpční vlastnosti tří různých druhů popílků a jednoho popílku modifikovaného 1M, 3M a 6M roztokem NaOH pro odstraňování fosforu z vod. Tato práce navíc ověřuje možnosti využití rychlých nekonvenčních analýz, konkrétně metody vlnově disperzní rentgenfluorescenční spektrometrie (WD XRF) pro analýzu fosforu v kapalných vzorcích. Analýza kapalných vzorků byla pro srovnání prováděna i tradiční spektrofotometrií (molybdenanová metoda). Výsledky experimentu ukázaly, že největší množství fosforu bylo odstraněno použitím popílku s největším obsahem CaO. Modifikace popílku roztokem NaOH výrazně nezlepšila sorpční vlastnosti popílku. Stanovení obsahu fosforu v kapalných vzorcích ukázalo srovnatelné výsledky za použití obou metod a tím byla ověřena možnost použití XRF spektrometrie pro analýzu fosforu ve vodách.

Key words: Phosphorus, fly ash, modification, XRF, standard spectrophotometric method.

1 INTRODUCTION

In the recent years we can see the great effort to reuse energetic by-products whose amount increases with increasing demand for energy throughout the world. Fly ash – a by-product of coal combustion offers due to its physical-chemical properties various possibilities of utilization. In spite of a relatively wide range of applications, still a great amount of fly ash is directly landfilled without any regards for environmental and economic concerns tending to find an alternative way to utilise the fly ash. Due to the high percentage of alumina and silica, it is a good candidate for use as an economic adsorbent for various pollutants. It has been found out that the fly ash can remove both inorganics [1] and organics [2] from aqueous solution. Any utilization of secondary by-products instead of primary materials leads to savings and reduction of natural resources and contributes to sustainable development.

Phosphorus in water causes considerable problems related primarily to the quality of surface waters. Excessive phosphorus encumbering various sources is the major reason for eutrophication in natural water reservoirs. Basic methods of phosphorus removal involve chemical precipitation, biological removal, ionic exchange and sorption [3, 4]. Sorption was found to be an attractive option in removing P from the effluent. Naturally, recent studies have focused on economically feasible and effective sorbents. From this point of view – fly ash as an industrial by-product seems to be the most suitable sorbent for this purpose. The principle of phosphorus removal from wastewater consists in the formation of insoluble metal phosphates, namely calcium, magnesium, iron and aluminium that influence concentrations of phosphorus in water [5]. The fly ash contains oxides of all of the above mentioned elements and therefore it is a suitable candidate for phosphate removal. Few works have investigated phosphorus removal from wastewaters using the fly ash. They usually compared the effectiveness of fly ash and other adsorbents for P removal [6, 7, 8].

This study was made to investigate the feasibility of using three different types of fly ash (a by-product of coal combustion for electric power generation and a by-product of blast furnace). The solution of 1M, 3M and 6M NaOH was used to enhance the sorption capacity of a selected fly ash. In literature can be found that the alkali modification of fly ash can increase the active sites for sorption [9].

This study also compares two different methods of the analysis of phosphorus in water, namely the standard molybdenate spectrophotometric method and the X-ray fluorescence spectrometry method (XRF). The X-ray fluorescence analysis is mainly used to analyse solid materials in forms of powders, pellets, or fused disks. Its usage for the liquid analysis is seldom. It is mainly because of high detection limits due to a great dispersion on light matrix, vaporization of some types of solutions, etc [10]. On the other hand, the application of XRF in comparison with other methods for liquid samples is quick, easily automated and without the necessity of dilution. That is why we decided to compare this method with the standard spectrophotometric method for the determination of phosphorus in waters. The X-ray fluorescence spectrophotometry method was also used for the detection of P in solid samples – fly ashes before and after reactions.

2 MATERIALS AND METHODS

2.1 Fly ashes

In this work three different types of fly ash were used. Two of them were coal fly ashes from two different coal-fired power plants in the northern part of Moravia, marked as FA1 and FA2. The used coal fly ashes were supplied by the Dětmarovice and Mittal electric power stations. The third was a fluid fly ash marked as FF1, supplied by the thermal power station Olomouc. The samples were oven-dried at 105°C for 4 hours before use. Its chemical composition was determined by the WD XRF spectrometer and is shown in Tab. 1.

The major components of all fly ashes were calculated in an oxide form. Major components are represented by Si, Al oxides, followed by Fe. The compositions of the used ashes were proved by a diffraction analysis. The results of FA2 based on the analysis of crystal phases show the composition, as follows: Hematite-1.84%, Magnetite-3.06%, Mulite-49.00%, Periclase-4.85%, Quartz-41.20%.

Tab. 1 Chemical composition of untreated fly ashes

Constituent	FA1	FA2	FF1
	weight (%)		
MgO	1,46	2,15	1,29
Al ₂ O ₃	19,54	19,40	26,33
SiO ₂	54,95	41,02	42,06
P ₂ O ₅	0,11	0,15	0,33
SO ₃	0,17	0,63	3,59
K ₂ O	2,35	2,02	1,00
CaO	3,54	5,48	14,30
TiO ₂	0,95	0,97	1,67
Fe ₂ O ₃	6,37	7,88	6,65

2.2 Modification of fly ash

The fly ash FA2 was subjected to the modification, because of its feasible grain composition. For the modification the solution of NaOH in 1M, 3M and 6M concentrations was used. The fly ash was treated in NaOH solutions in the ratio of 1:10 by weight. The mixture was then incubated at 55°C for 24 hours. At the end of the treatment, the mixture was filtered, washed thoroughly with distilled water and dried in the oven at 75°C. The final solid products were then subjected to chemical and physical analyses.

2.3 Phosphorus adsorption

Five grams of each sample (initial and treated fly ashes) were added to 50 ml of KH₂PO₄ solutions in concentrations of 0,5; 1; 1,5; 3 and 6 g P/l. The samples were equilibrated by continuous shaking on a rotating shaker (195 rpm) at a constant temperature (21°C). The suspensions were then filtrated. P remaining in the supernatant was determined by two methods; standard ammonium molybdate method and XRF spectrometry. The pH in the equilibrium solution was measured using the WTW pH 330i meter. The fly ash after the sorption process was dried at 105°C and phosphorus was determined by the XRF spectrometry technique. The data obtained from all three methods were expressed as an amount of phosphorus adsorbed on 1 g of fly ash and were compared.

For the calculation of the adsorbed amount of phosphorus the equation was used, as follows:

$$a = V \cdot \frac{(c_0 - c_r)}{m} \quad (1)$$

where:

- a – adsorbed amount [mg/g],
- c₀ – initial concentration of P in solution [mg/l],
- c_r – concentration of P after sorption [mg/l],
- V – volume of solution [l],
- m – weight of fly ash [g].

2.4 Apparatus

WD XRF

The Wavelength Dispersive X-ray fluorescence (WD XRF) spectrometer MAKC GVII Spectron –Russia was used to analyse liquid and solid samples.

The liquid samples were measured in polypropylene cuvettes covered with a Mylar foil. Pellets were made for measurement of phosphorus and other oxides in the solid samples. The pellets were made by blending the fly ash and a binder (Hoechst wax for X-ray fluorescence analysis) in the ratio of 4:1 using a pellet die and hydraulic manual press.

The calibration curve for phosphorus determination in water was performed using a standard solution of KH_2PO_4 in H_2O . The working standards of 0, 1, 5, 10, 20, 50, 100, 250, 500, 1000, 2500 and 5000 mg P/l were used. The correlation coefficient was $R^2 = 0,9999$, that indicates the high precision of the linear calibration curve. The accuracy of the solid sample measurements was verified by using standard reference materials NIST 1633b, 2689, 2690 and 2691.

The quantification and estimation of combined standard uncertainty (u_c) was performed by determining the uncertainty A (u_A) calculated as a standard deviation of 20 measurements of one sample and the experimentally estimated uncertainty B (u_B).

Standard spectrophotometric method

Orthophosphates react under the H_2SO_4 condition and catalytic effect of antimony (III) ions with ammonium molybdate and form yellow phosphomolybdenum acid. The yellow complex turns in a blue complex (feasible for the absorption by the spectrophotometry) of phosphomolybdenum after the reduction with ascorbic acid. The concentration of diluted orthophosphates is determined from the calibration graph, which is prepared from a stock solution of PO_4^{3-} in an concentration of 0,05 – 1,00 mg P per litre.

The sample after sorption process was diluted to the required concentration and transferred to a 50 ml volumetric flask followed by the addition of 5 ml of the ammonium molybdate reagent. The obtained solution was left for 15 min to ensure colour development. The absorbance was measured at 690 nm with the HACH DR/2000 spectrophotometer. The same procedures were used for blank and the result was subtracted from the value of the examined sample.

Microscopy

As a supplemental method for the description of structural properties of fly ashes the observation in the laser confocal scanning microscope Olympus LEXT OLS 3100 was used. It is possible to observe surface characteristics of particular materials by utilization of a laser beam of 408 nm wavelength with subsequent optimization of the display quality.

2.5 Comparison of analytical methods

The calculation of the sorbed amount of phosphorus was determined in three different ways and the results were compared. First two methods were based on the analysis of phosphorus remaining in the solution after filtration of the sorbent after set time. The sorbed amount was calculated using the equation (1). Two different methods - standard spectrophotometric method and XRF spectrometry were used for this purpose. The third method was based on the analysis of phosphorus directly in the sorbent after the reaction. The XRF analysis of these samples gave results in weight % which could be directly converted to mg/g and than compared with other two methods.

3 RESULTS AND DISCUSSION

3.1 Phosphorus immobilization of untreated fly ashes

The ability of three different fly ashes to sorb phosphorus from solution was studied. From the Fig. 1 it is clear that the fly ash FF1 showed the greatest ability to bind the phosphorus.

This ability increased with increasing initial concentration of phosphorus in the solution. This result is in agreement with the composition of untreated fly ashes. It is known that a main factor influencing the mobility of phosphorus in solution is the calcium concentration in the sorbent. The fluid fly ash which showed the greatest P sorption ability contained the highest concentration of calcium oxides (see Tab.1) which is considered to play the dominant role in phosphorus immobilization. Previous studies suggested that P immobilization on fly ash was attributed to the binding of phosphorus and calcium resulting in the precipitation of calcium phosphate and to the adsorption on the surface of fly ash [11]. It is known that also other oxides mainly the oxides of Fe and Al have the ability to bind phosphorus [12]. The sum of all these three oxides is the lowest in the fly ash FA1. That results in the lowest capacity of this fly ash to remove phosphorus from the solution.

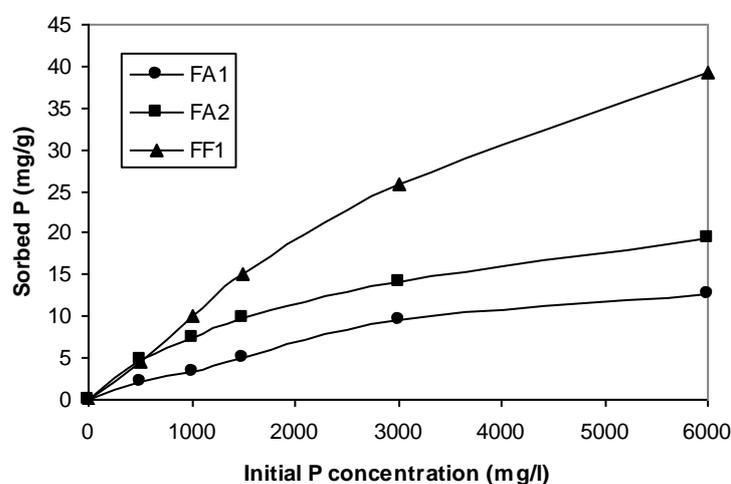


Fig. 1 Sorption of P on untreated fly ashes

The fly ash FA2 was chosen for further studies since we tried to enhance its sorption capacity by its modification to the level of fluid one.

3.2 Modification of fly ash FA2

The fly ash FA2 was modified by three various solutions of NaOH in a way described in chapter 2.2. The chemical composition of the modified samples (determined by WD XRF spectrometry) is shown in Tab. 2.

Tab. 2 The chemical composition of the untreated fly ash FA2 and the fly ash treated by the 1M, 3M and 6M solution of NaOH.

Constituent	FA2	FA2-M1	FA2-M3	FA2-M6
	weight %			
MgO	2,15	2,17	2,02	2,17
Al ₂ O ₃	19,40	19,23	18,47	20,05
SiO ₂	41,02	40,78	37,15	36,51
P ₂ O ₅	0,15	0,13	0,10	0,07
SO ₃	0,63	0,26	0,27	0,25
K ₂ O	2,02	1,82	1,33	0,96
CaO	5,48	5,78	6,10	6,40
TiO ₂	0,97	1,01	0,84	0,87
Fe ₂ O ₃	7,88	8,60	8,75	9,12

Changes in CaO and Al₂O₃ contents can be described by the dissolution and precipitation mechanisms connected with the formation of portlandite, ettringite and hydrated Ca-aluminate phases. The dissolution effect and further precipitation of CaO could be supported by the decreasing content of SO₃. The content of Fe oxides also rose with the strength of solution. In contrast to that, the oxides of Si, P and K decreased with increasing concentration of NaOH. The decreasing content of SiO₂ is in accordance with the results presented by A. Mikuni [13]. The increasing concentration of NaOH has a significant influence on the dissolution of SiO₂. All those components like K₂O, Al₂O₃, CaO and SiO₂ and their changes are involved in the formation of zeolite phases such as hydroxy-sodalite and clinoptiolite. This transformation mechanism on the liquid and solid interfaces results from alkaline conditions within the fly ash conversion.

The images of the treated fly ashes made on the confocal microscope are shown in Fig.2. The figure showed the presence of micro-particles in the shape of smooth balls in the untreated fly ash. After treating with NaOH, the ball-shaped particles of FA2 were transformed into agglomerations of an undefined shape of plates, rods, etc. From these figures it is obvious that the surface area of the treated fly ash was greater than that of the untreated fly ash. The results of the increased surface areas are in accordance with the results obtained from methylene blue - spot test method. Within the modification of the fly ash surface a rising trend of the methylene blue sorption was observed. The greater surface area of NAOH treated FA2 may be attributed to the transformation of the fly ash into a zeolite-like structure which was found to form under alkali conditions by other studies [9,11,14].

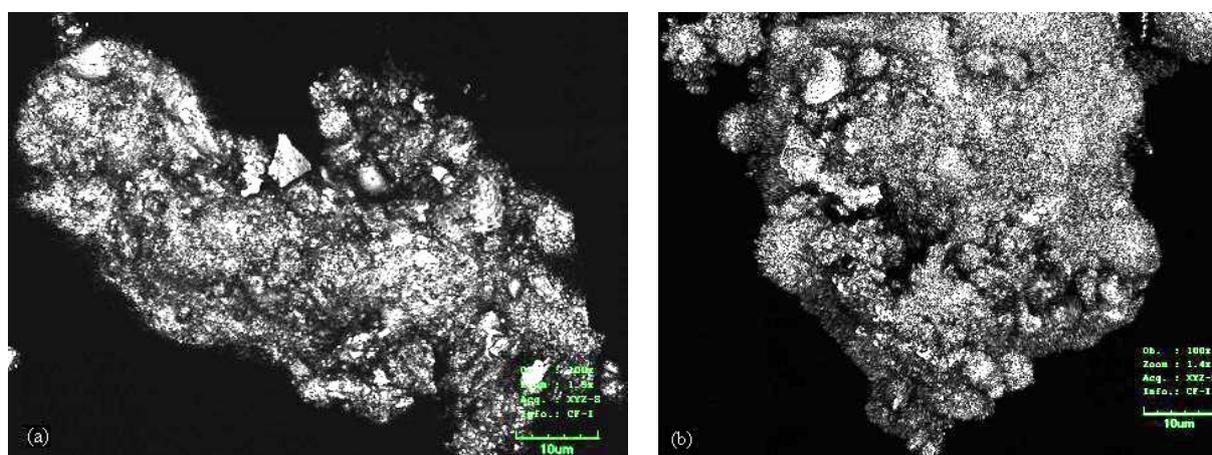


Fig. 2 Images of the untreated fly ash FA2 (a) and the 6M NaOH treated fly ash FA2-M6 (b)

3.3 Phosphorus immobilization on FA2 treated with NaOH

The experiment of testing sorption of phosphorus on the treated fly ashes was done in the same way as the experiment with the untreated fly ash. Fig. 3 shows the amount of sorbed P measured by the WD XRF spectrometry of solution.

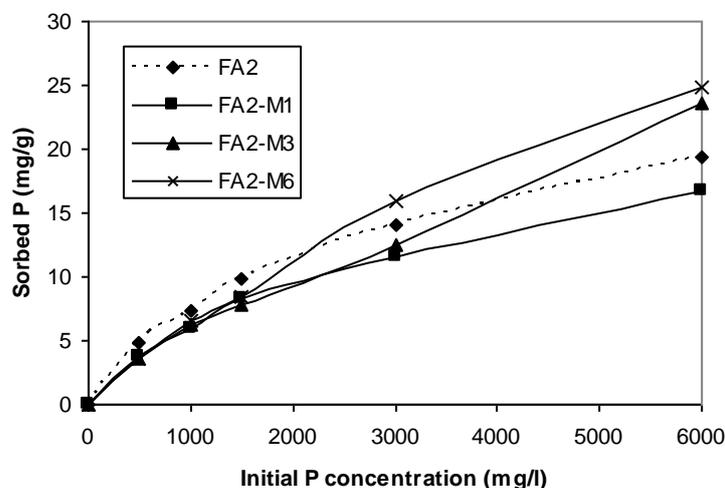


Fig. 3 The sorption of P on the fly ashes treated with NaOH

The phosphorus sorption capacity of the untreated FA2 and the fly ashes treated with NaOH sharply increased with an increase in P concentration with the greatest sorption at c_0 of 6000 mg/l. From Fig. 3 it is obvious that up to the initial concentration of P of 3000 mg/l for the untreated fly ash showed a better sorption capacity of phosphorus than the modified ones. At c_0 of 3000 mg/l and 6000 mg/l the fly ash treated with 6M NaOH sorbed the highest amount of phosphorus. The fly ash treated with 1M of NaOH had the worst P sorption capacity of all samples used and therefore the modification of fly ash with 1M NaOH has no advantage. Experiment showed that the modification of fly ash with the 6M NaOH solution for improving the sorption capacity for phosphorus was evident only for the solutions with an initial concentration of phosphorus over 2000 mg/l, with the 3M NaOH solution even over 4000 mg/l. However, none of the treated fly ashes showed enough distinct difference from the untreated one to make the treating process reasonable.

3.4 Comparison of analytical methods

Three different analyses and consequent calculations of the adsorbed amount of phosphorus were done and compared. The measurements of liquid samples (l) are marked as Spf (l), if the concentration of P was measured by the standard spectrophotometric method. The WD XRF analysis of supernatant is marked as XRF (l). The determination of P in solid samples by the XRF method is marked as XRF (s). The sorption of phosphorus by untreated fly ashes as well as modified fly ashes was measured by all three methods. The analyses were done in triplets and average concentrations are displayed in Fig. 4. The estimated combined standard uncertainties (u_c) for the results obtained by XRF in solid samples are 11%, in liquid samples 10% and by the spectrophotometric method better than those by XRF (7%).

The comparison of all three methods indicated a lower concentration of P measured directly in fly ash by the XRF spectrometry technique. Both methods measuring the concentration of P in solution showed comparable results with a bit higher concentration determined by the standard spectrophotometry method. However, the comparison of the results showed a good correlation among all three methods, because all results were within the range of estimated uncertainties. Therefore, it is clear that any of the investigated method can be used for the calculation of P sorbed by adsorbent.

From this point of view the analysis of phosphorus in solution by the XRF spectrometry method seems to be the optimal choice. If a wide-range calibration curve is drawn, the samples can be analysed directly without any preparation. Moreover, no chemicals are needed for the determination of phosphorus in such samples. The standard spectrophotometry technique is an approved and widely used method for this purpose, but the analysis procedure takes longer time, chemicals for reactions are necessary and the samples often need to be diluted. The analysis of phosphorus directly in a solid sample is quick, but the sample preparation is time consuming. The

sample needs to be dried at least for two hours at 105°C, homogenized with an additive and a pellet has to be made. Moreover, the accuracy of this method is lower in comparison with other two methods.

But it must be pointed out that the initial concentrations of phosphorus in water were potentially high (from 500 – 6000 mg/l) which gave a related high residual concentration in solution. There is a high probability that the XRF analysis will not be sufficient for the detection of a low concentration of P in water due to its potentially high detection. Other experiments have to be done to confirm or deny this theory.

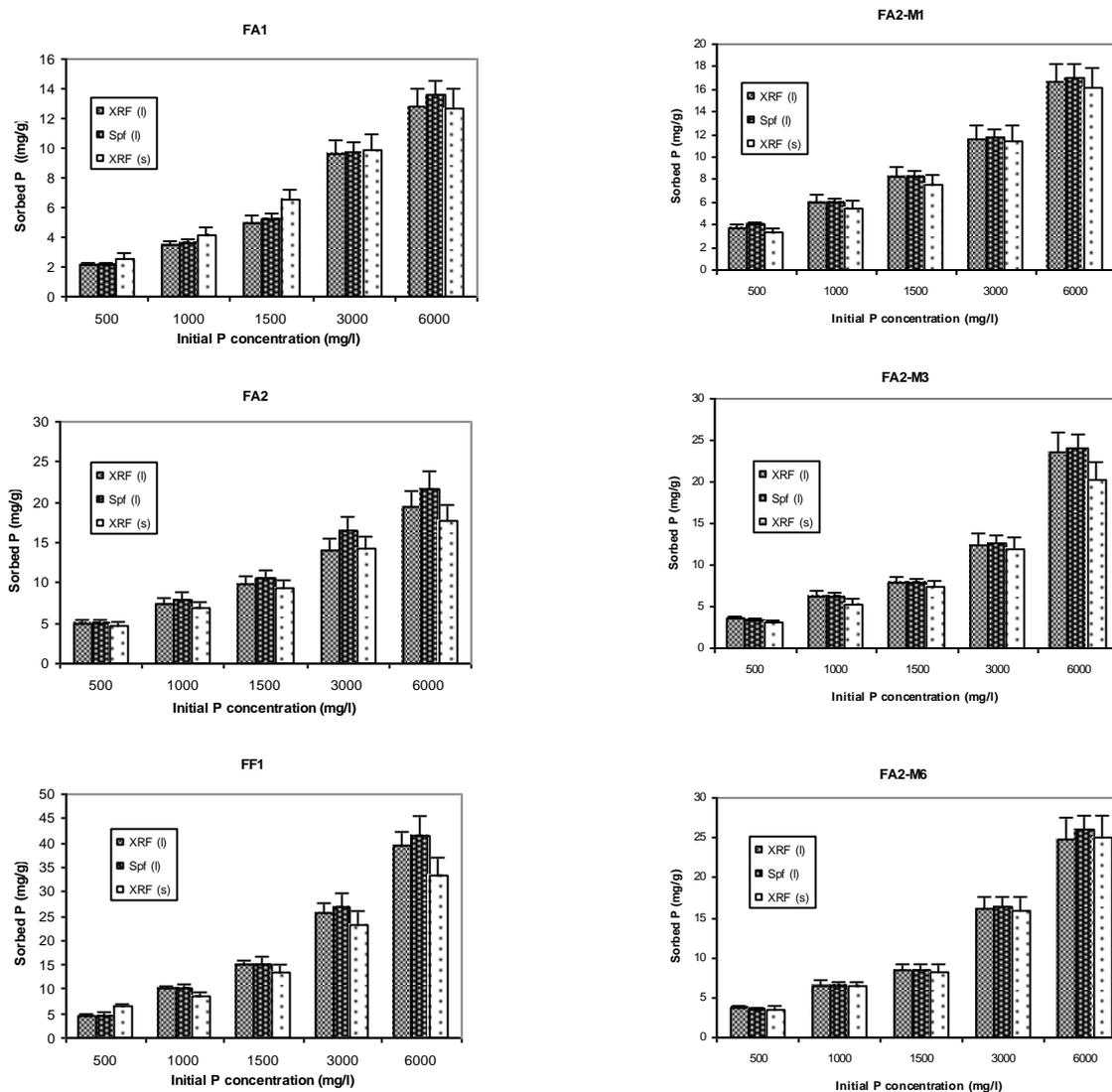


Fig. 4 Concentrations of P (in mg/kg) by the Spectrophotometric method Spf (l) and by XRF in liquid (l) and solid (s) samples

4 CONCLUSIONS

Three fly ashes with a different initial composition showed different behaviours in the phosphate uptake. The fluid fly ash showed the higher sorption capacity of phosphorus mainly due to its higher concentration of CaO which is known to play the most important role in the phosphorus immobilization.

In order to enhance the sorption capacity of a non-fluid fly ash the treatment with NaOH was performed. The chemical treatment of coal fly ash changed the surface of the treated fly ash, which was clearly seen by using the confocal microscope; however the values of the adsorbed amount of P did not indicate significant increase. The fly ash treated with 1M NaOH showed even a lower adsorption capacity than the untreated sample. The best results were showed by the fly ash treated with 6M NaOH, but only with the initial concentration of P over 3000 mg/l. At 6000 mg/l the fly ash treated with 6M NaOH adsorbed 24,91 mg/g of phosphorus, while the untreated fly ash 19,36 mg/g of phosphorus. However, the value of fluid fly ash for the same initial concentration was 39,35 mg/g. Overall, the alkali modification of the fly ash did not enhance the capacity of phosphate immobilization to such level to ensure the return of energy and materials put into the treatment process.

In this work we also studied a comparability of the analytical results of different techniques, but with identical samples. The objective was to find out, whether XRF, characterized by a simple sample preparation and fast analysis can be considered as appropriate for the analysis of P in water. For this purpose all samples used for the sorption test (30) were analyzed by the XRF and standard spectrophotometric methods. The comparison of the results showed a good correlation between the methods and so validated the application of WD XRF for the analysis of P in water. However, the measurements were accomplished under a relatively high concentration of phosphorus in water and therefore other measurements verifying the applicability of the proposed method under the conditions of a low concentration of P are needed.

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RESUMÉ

Celosvětově zvyšující se energetické nároky vedou ke větší spotřebě uhlí a následovně k produkci velkého množství popílků. Přestože velkou část těchto odpadních produktů využívá například stavební průmysl, stále zůstává významný podíl uložen na skládky bez dalšího využití. Alternativní způsoby využívání popílků jsou proto stále předmětem zájmu. Jejich předností je hlavně snadná dostupnost ve velkých objemech a současně příznivá cena. Výhoda použití popílků jako sorbentu spočívá v jeho vhodném chemickém složení. Popílků obsahují vysoké koncentrace oxidů hliníku, železa, vápníku nebo křemíku, které hrají podstatnou roli v odstraňování fosforu z vod.

V této práci byly zkoumány sorpční vlastnosti tří typů popílků různého složení (dva úletové a jeden fluidní) a jednoho popílku modifikovaného pro odstraňování fosforu z vod. Vybraný úletový popílek byl modifikován 1M, 3M a 6M roztokem NaOH. Cílem bylo zlepšení jeho sorpčních vlastností změnou specifického povrchu a fázového složení.

Výsledky publikované v této práci ukazují na pozitivní vliv obsahu volného CaO na výsledky sorpčních testů. V porovnání tří standardních a tří modifikovaných forem, fluidní popílek obsahující nejvyšší koncentrace CaO vykazoval nejvyšší účinnost sorpce fosforu z vod. Modifikačních přeměny vstupního popílku zvýšily účinnost sorpce jen při vysokých počátečních koncentracích fosforu ve vodě, u modifikace 6M roztokem NaOH při 2000 mg P/l a vyšších u 3M až při počátečních koncentracích 4000 mg P/l. Popílek modifikovaný 1M roztokem NaOH vykazoval dokonce nižší hodnoty absorbovaného fosforu než jeho neupravený ekvivalent.

Výsledky sorpčních testů byly sledovány pomocí metody vlnově disperzní rentgenfluorescenční spektrometrie (WD XRF) a běžné spektrofotometrické metody (molybdenanová metoda). Z porovnání výsledků analýz jednotlivých metod vyplývá dobrá korelace stanovení, což umožňuje úspěšně využívat metodiky WD XRF pro kapalné vzorky. Výhodou této metody je rychlé nenáročné stanovení bez použití chemikálií a nutnosti ředění.