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DETERMINATION OF ARSENIC IN COMPLEX MATRICES BY UV-Vis SPECTROSCOPY: IMPORTANCE OF pH FACTOR

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ABSTRACT

Arsenic is a widespread metalloid found in soils, sediments and waste materials. The main sources of contamination are metallurgy, energy production from thermal power station and mining activities, which release stable forms of arsenic with different mobility depending on the pH conditions. This creates a health risk for workers exposed to contaminated materials and dust. The aim of the study is to determine the arsenic content in solid samples of industrial and natural origin by UV-Vis method with Variamine blue indicator. The results show good linearity (0.0389 - 0.649 mg L^{-1}, $R^2 = 0.99$, LOD = 0.0114 mg L^{-1} and LOQ = 0.0379 mg L^{-1}) and dependence on the pH values of the tested samples. The method is suitable for arsenic analysis, providing a short test time at a low cost and a sufficient degree of reliability of the result.

Keywords: arsenic, pH factor, Variamine blue, contamination, safety, UV-Vis spectroscopy.

INTRODUCTION

Arsenic causes significant health problems for almost all living organisms, including plants, animals and humans [1]. It is considered one of the most toxic elements in the environment and a human carcinogen, reported in many countries and regions [2]. According to the European Directive on Environmental Quality Standards, arsenic is listed as a priority toxic element [3]. Today, humanity is exposed to the highest levels of pollution in addition to arsenic and other heavy metals: lead, mercury, aluminium, copper, nickel, tin, antimony, bromine, bismuth and vanadium, and according to Rehman et al. the levels are up to several thousand times higher than in primitive man [1].

Due to its high toxicity and proven carcinogenicity, the accumulation of arsenic in various solid matrices such as soils, sludges from the steel [4] and cement industries [5], ashes from thermal power plants [6] and waste soils [4] poses a serious risk to the environment and human health. The main mechanism of exposure is through secondary contamination of water, air and the food chain, as mobile forms of arsenic can easily pass into groundwater and surface waters [7].

Several studies have been conducted in the past to elucidate the mechanisms of arsenic accumulation in the food chain and its impact on the nature [8]. According to Siddiqui et al., there are three main natural sources of arsenic contamination: hydrothermal activity, minerals/ores, and aquifer sediments [2]. In soil systems, the toxicity threshold and mobility of arsenic depend on soil properties: particle size, texture, mineral nutrient content, pH, presence of other ions, and the chemical form of arsenic. These soil properties are very important for assessing the impact of arsenic on its accumulation and distribution in the environment [9]. In soil samples, arsenic is often associated with mineral fractions, but industrial activities such as metallurgy, cement production, and coal combustion significantly increase its concentration and bioavailability, which creates prerequisites for long-term human exposure through agricultural production and dust emissions [10].

In addition to natural sources, industrial activities also contribute significantly to arsenic pollution. In the metallurgical industry, non-ferrous metal smelting processes release arsenic vapours and dust particles, which pose a serious risk to the health of workers and the environment [11]. In steelmaking sludge, arsenic is a by - product of the use of ores and additives, and if not adequately controlled, it can be released into the environment through storage or reuse of waste [12]. Coal combustion in thermal power plants also generates fly ash containing arsenic, which, if not properly managed, can lead to large-scale pollution [13]. Ash from thermal power plants, especially when burning coal with a high arsenic content, can concentrate significant amounts of the element, and if improperly disposed of, leaching and secondary contamination of groundwater can occur [14]. In the cement industry, the addition of arsenic-contaminated raw materials or the use of secondary fuels leads to the accumulation of As in the clinker and in the released dust particles, which pose a hazard to workers [12]. Waste soils generated during mining activities are also a potential source of arsenic compounds, which, upon atmospheric weathering, can be transported by wind erosion or infiltration [15].

The accumulation of arsenic in ecosystems has serious consequences, including toxicity to aquatic organisms, contamination of drinking water sources, and increased risk of chronic diseases when ingested by humans [16]. Long-term exposure to even low concentrations is associated with increased incidence of cancer, cardiovascular, and neurological diseases [17]. Arsenic can enter the human body through various routes - by ingestion, inhalation, and contact through the skin and mucous membranes. Sources of such exposure in the natural and occupational environment include particulate matter, soil, industrial waste, contaminated air and water, and foods from affected areas [18]. Therefore, the development and implementation of effective policies to protect the health of workers in hazardous industries is essential for the prevention of arsenic intoxication.

A variety of analytical approaches are used to assess these risks. Methods such as Ion Selective Photometry with Mass Spectrometry (ICP-MS) and Hydride Generation Atomic Absorption Spectrometry (HG-AAS) allow highly sensitive determination of total arsenic content and its valence state in soil and sediment extracts [7]. UV-Vis spectroscopy is widely used for routine analysis due to its low cost and rapid performance, especially in laboratories with limited resources [19]. In recent years, developments with nanomaterials and new reagents have increased the selectivity and sensitivity of UV-Vis methods, making their application possible for the detection of arsenic in complex matrices [20]. UV-Vis spectroscopy is an analytical method based on the measurement of light absorption by molecules or complexes in the ultraviolet and visible regions, which allows for the rapid and sensitive quantification of various elements and compounds [21]. The measurement and monitoring of arsenic in these solid matrices is not only an analytical task, but also a key aspect of human health risk management [22]. Therefore, the development of an accessible and rapid method for arsenic determination is essential to prevent its accumulation in the food chain and to ensure a safe environment for humans.

The aim of this article is to demonstrate, through rapid UV-Vis analysis, using the Variamine blue indicator, the possibility of determining the arsenic content in complex matrices, by investigating the influence of the pH factor on its absorbance.

EXPERIMENTAL

The study is based on the analysis of seven samples from different industrial and natural sources: slag from a steel plant (sample No. 1), sludge from a cement plant treatment plant (sample No. 2); waste soil mass from a non-ferrous metal mine (sample No. 3); soil sample (sandy soil - sample No. 4) and ashes from thermal power plants (samples No. 5, 6, 7) [23]. The samples were dried at room temperature to remove free moisture by spreading out in a layer about 1 cm thick and pieces larger than 5 mm were removed from the dried samples. The samples were crushed and sieved through a sieve with a mesh size of 2 mm to obtain the fraction required for the study. One gram (± 0.0001) from the obtained laboratory samples, is weighed and transferred to an Erlenmeyer flask and poured with 3 - 4 cm^3 H_2SO_4 (Merck, p.a.) until wet, 10 cm^3 HNO_3 (Sigma-Aldrich, p.a.) is added and heated until evaporation. Then, if necessary, another portion of nitric acid is added until the organic matter is completely burned. After cooling, the sample is poured with 10 cm^3 0.1 M HCl (Sigma-Aldrich, p.a.), heated until vapours are released and filtered through a double blue ribbon filter into a 50 cm^3 volumetric flask. It is washed with hot, acidified with a few drops of hydrochloric acid water and made up to the mark with distilled water.

NaOH (Sigma-Aldrich, p.a.) (0.1 M and 0.2 M solutions) was used to adjust the pH of the working samples. A pH meter with a glass electrode (Milwaukee MW805, USA) was used to measure the pH, calibrated with buffer solutions with pH 4, 7 and 10 (Scharlab).

For the purpose of spectrophotometric determination, a suitable aliquot was pipetted from each sample and transferred to a 10 cm^3 volumetric flask. 1 cm^3 of 2 % KIO_3 solution (Teokom, p.a.) and 1 cm^3 of 0.4 M HCl were successively added to it, after which the mixture was homogenized by gentle shaking. 1 cm^3 of 0.05 % Variamine blue solution (Sigma-Aldrich, p.a.) and 2 cm^3 of 2 % CH_3COONa solution (Merck, p.a.) were added. The resulting solution was left to stand for 5 minutes, after which spectrophotometric measurement was performed at λ = 556 nm against a blank sample [23]. A DLAB spectrophotometer (China) was used for UV-Vis analysis.

To prepare the standard line, aliquots of 0.1 M arsenic standard solution ($NaAsO_2$, Merck, p.a.) were pipetted and solutions were prepared in the concentration range 0.0389 - 0.649 mg L^{-1}. Each solution was subjected to five consecutive measurements, and the average value obtained from these repetitions was used to assess the repeatability of the absorbance. The wavelength at which the measurement was performed was 556 nm, relative to a blank sample.

For the spectrophotometric determination of arsenic in the laboratory samples, it is necessary to pipette 2 cm^3, after which the procedure for spectrophotometric analysis is followed. The absorbance results of the working samples are presented in the results and discussion section, and for each sample, measurements were performed at pH = 4, pH = 5.5, pH = 6 and pH = 7 on five parallel samples, to monitor its influence on the spectrophotometric measurement.

RESULTS AND DISCUSSION

The determination of arsenic is based on its reaction with Variamine blue after oxidative treatment. As(III) is oxidized to As(V) with potassium iodate in hydrochloric acid, releasing iodine (I_2). Iodine oxidizes Variamine blue to a blue-violet colour (Fig. 1).

Regarding arsenic, as reported by Mohan et al. [25] and Komonweeraket et al. [26] it is sensitive over the entire pH range. According to a previous study, the working pH range of the indicator was found to be 4 - 7 [23].

To ensure the suitability of the applied UV-Vis method for arsenic quantification, key analytical parameters were investigated: linear range, linearity, coefficient of determination, precision and limits of detection, and quantification.

The uncertainty of the method was calculated in the concentration range from 0.0389 to 0.649 mg L^{-1}. The linear relationship between absorbance and arsenic concentration was demonstrated by calculating the coefficient of determination (R^2). The obtained value of 0.99 (Fig. 2) shows a strong linear relationship and confirms that the method obeys the Beer-Lambert law in the specified range, and the equation of the calibration curve is: $y = (0.4714 \pm 0.0559)x + (0.0832 \pm 0.0191)$, where the values are presented as estimate ± standard deviation.

The precision of the method, expressed as relative standard deviation (RSD %), was evaluated at different

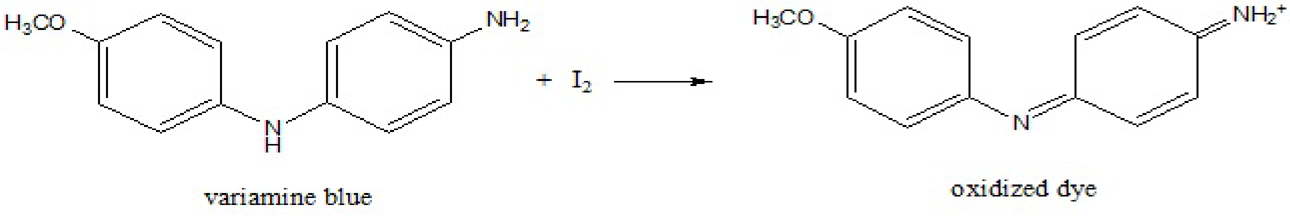

Fig. 1. Variamine blue colour reaction [24].

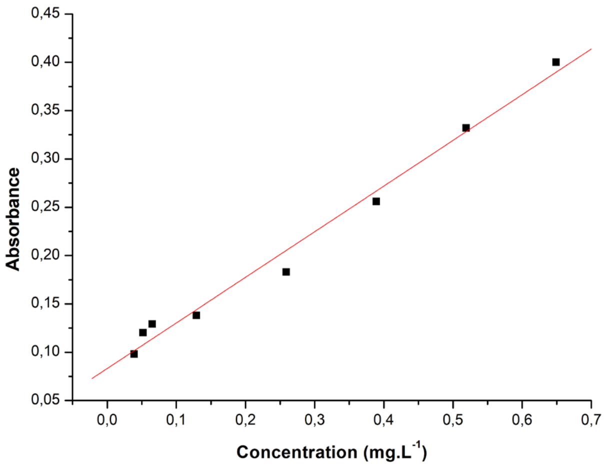

Fig. 2. Calibration curve.

concentrations. At low concentration (0.0389 mg L^{-1}) the calculated RSD % value was 7.34 %, which is within the acceptable limits for quantification regulated by international guidelines ICH Q2(R1) [27] and AOAC [28], which allow a limit of 10 - 15 % especially at low concentrations. At high concentration (0.649 mg L^{-1}) the method demonstrated excellent precision with an RSD value of 0.39 %, which is below 1 % and indicates excellent precision of the method [29].

The limit of detection (LOD) and the limit of quantification (LOQ) were experimentally determined to be 3.3 and 10 times the standard deviation of the blank sample, respectively, converted to concentration by the slope of the calibration curve [30]. The values found were LOD = 0.0114 mg L^{-1} and LOQ = 0.0379 mg L^{-1}. These values indicate that the developed method is suitable for the determination of arsenic in low-content samples.

The results of the analytical calculations, presented in Table 1, confirm that the developed method is reliable and precise, as well as suitable for quantitative determination of arsenic in the studied concentration range. The established good analytical characteristics - linearity, low standard deviation and high precision – confirm its applicability for routine laboratory analyses. To demonstrate its practical effectiveness, the method was applied to study the arsenic content in different matrices with diverse origins and compositions. The obtained results are presented in the following table.

The data presented in Table 2 reveal the complex influence of pH on the mobility (and therefore measurable concentration) of arsenic contained in matrices of different origin and composition. The results clearly show that the efficiency of arsenic leaching does not follow a uniform trend but is strongly dependent on the specific chemical origine of each sample. Predicting the environmental risk and the risk to workers requires a thorough knowledge not only of the total arsenic content, but also of the mineralogy of the contaminated matrices and the specific pH conditions.

From an occupational safety perspective, these data are of great importance. The extremely high mobility of arsenic from the steel mill sludge (sample No 1) under neutral conditions (pH = 7) suggests a serious risk to workers. Studies have shown that such sludges can release significant amounts of soluble arsenic upon contact with water [31], especially at pH values close to neutral [32]. Activities involving wetting of this material with water (cleaning, dust control, precipitation) can

Table 1. Calibration data and statistical indicators for the UV-Vis method.

Concentration (x) mg L^{-1}	Mean ($\bar{y}$)	Standard deviation (S)	Relative standard deviation (RSD), %	Fitted values, $\hat{y}$	Confidence interval, ±
0.0389	0.0978	0.0072	7.35 %	0.1015	0.0175
0.0519	0.1204	0.0068	5.68 %	0.1077	0.0168
0.0649	0.1286	0.0024	1.87 %	0.1138	0.0161
0.129	0.1382	0.0055	4.01 %	0.1440	0.0127
0.259	0.1826	0.0049	2.67 %	0.2054	0.0104
0.389	0.2560	0.0224	1.24 %	0.2666	0.012
0.519	0.3322	0.0243	7.31 %	0.3278	0.0163
0.649	0.4006	0.0015	0.39 %	0.3891	0.0219

Table 2. Results for absorbance.

Sample No	Absorbance at pH = 4	Absorbance at pH = 5.5	Absorbance at pH = 6	Absorbance at pH = 7
1	0.173	0.442	0.466	0.904
2	0.201	0.155	0.403	0.234
3	0.432	0.474	0.625	0.330
4	0.164	0.355	0.359	0.310
5	0.213	0.393	0.719	0.415
6	0.299	0.221	0.400	0.300
7	0.287	0.433	0.663	0.621

lead to the formation of highly concentrated arsenic solutions. This creates a direct hazard of skin contact or inhalation of aerosols containing dissolved arsenic, which significantly increases the risk of occupational diseases [33].

The lower reported arsenic absorbance in cement sludge (sample No 2) upon pH change indicates that in this case the risk of arsenic exposure to workers is lower. Studies on the stabilization of hazardous waste by cementitious materials have shown that arsenic can be effectively immobilized in a matrice [34], which limits its solubility [35]. However, there is still a risk of inhalation of dust particles containing immobilized arsenic, especially under dry conditions and intensive mechanical processing.

The results summarized so far emphasize that occupational safety measures must be specifically adapted to the type of material being handled. Universal precautions may not be sufficient. For industries such as steelmaking (sample No 1), strict control of working conditions is necessary to prevent workers from coming into contact with generated sludge and wastewater [31], and the mandatory use of personal protective equipment that protects both the respiratory tract and the skin [33].

Arsenic concentrations in the samples (see Table 3) were calculated based on a calibration curve for the concentration interval 0.0389-0.649 mg L^{-1}, which guarantees quantification within the studied range. This procedure allows for reliable comparison between samples and assessment of the influence of pH on the solubility and mobility of arsenic.

Table 3. Concentrations at pH = 6 and pH = 7.

Sample No	Concentration at pH = 6 mg L^{-1}	Concentration at pH = 7 mg L^{-1}
1	0.812 ± 0.08	1.742 ± 0.18
2	0.673 ± 0.065	0.320 ± 0.042
3	1.149 ± 0.11	0.523 ± 0.052
4	0.585 ± 0.057	0.481 ± 0.05
5	1.349 ± 0.13	0.704 ± 0.067
6	0.672 ± 0.064	0.460 ± 0.048
7	1.229 ± 0.12	1.141 ± 0.12

From the data presented in Table 3, the highest concentration was recorded at pH = 7, in sample No. 1, namely 1.742 mg L^{-1}, while for the other samples the highest concentrations were found at pH = 6. These results suggest greater mobility and solubility of arsenic in neutral and slightly acidic conditions. At the same time, the highest value found in sample No. 1 may be due to the specific composition of the matrice used, where significant amounts of metals and metalloids, including arsenic, accumulate because of industrial processes. This sharp jump suggests a process of arsenic mobilization in the specific matrice [25], probably due to dissolution of iron-arsenic complexes or desorption at appropriate pH values [36].

A similar trend was observed in sample No. 5 (ash from thermal power plant), where at pH = 6 a concentration of 1.349 mg L^{-1} was recorded. This type of samples shows potential behavior of arsenic as a mobile element in an origin with variable pH, which emphasizes the need for additional attention in their treatment and management. According to Liu et al. [37] and Deonarine et al. [38], in coal ash, even a small part of the total amount of arsenic is mobile and depends on the pH.

The results of the study provide an insight into the influence of pH on the absorbance of arsenic in the studied matrices, differing in their origin and composition. This is consistent with literature data on the behaviour of arsenic in industrial residues [39], where it occurs mainly as As(V), and in smaller amounts as the more toxic form As(III) [40]. The arsenic concentrations measured in the working samples confirm the effectiveness of the applied preparation and analysis methods within the studied concentration range. These data clearly emphasize that the determination of the total arsenic content in complex industrial matrices is highly dependent on the leaching and analysis conditions.

CONCLUSIONS

Based on the conducted studies, it was found that the applied method is an effective and reliable analytical tool for rapid, accurate and low-cost effective assessment of arsenic contamination in real samples from different matrices. Its practical application showed that the pH parameter has a significant impact on the analytical signal and should be strictly controlled during the analysis.

The results revealed that the human health risk of arsenic is not determined by its total content, but by its potential for mobilization under specific conditions. The studied slag poses a significant hazard at neutral pH, while the cement sludge limits the mobility of arsenic and reduces that hazard.

The method could serve as a basis for a differentiated risk assessment for both worker health and environmental impacts, making it a valuable tool for the informed selection of appropriate safety measures tailored to the specific characteristics of the analysed matrices.

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