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RESEARCH ARTICLE

DETERMINATION OF URANIUM IN PHOSPHATE INDUSTRY EFFLUENTS USINGWDXRF: CONCENTRATION ASSESSMENT AND ENVIRONMENTAL IMPLICATIONS

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Abstract

Uranium in phosphate industry effluents poses significant environment al risks due to its chemical toxicity and radiological properties. This study evaluates wavelength-dispersive X-ray fluorescence (WDXRF) as a rapid and non-destructive method for the direct determination of uranium in liquid effluents without complex sample preparation. The method demonstrated good precision, with a coefficient of variation of 1.6%, confirming its suitability for routine monitoring. Uranium concentrations were found to be approximately 10 mg/L, significantly exceeding international guideline values established by the World Health Organization and the United States Environmental Protection Agency (0.03 mg/L). Such elevated levels highlight the potential for serious environmental impacts, including contamination of aquatic systems, sediment accumulation, and bioaccumulation in the food chain.These findings emphasize the necessity of implementing effective treatment strategies prior to discharge. Overall, WDXRF is confirmed as a reliable and cost-effective technique for environmental monitoring of uranium in industrial wastewater.

Introduction:-

The phosphate industry plays a key role in global fertilizer production. Wet-process phosphoric acid production generates liquid effluents containing dissolved salts, heavy metals, and naturally occurring radionuclides [1,2]. Among these, uranium is particularly concerning due to its chemical toxicity, radiological hazard, and mobility in water and soil [3,4]. During the acidulation of phosphate rock, uranium partially dissolves into the liquid phase, leading to its accumulation in industrial effluents [5]. These effluents, if released without treatment, can contaminate surface and groundwater, bioaccumulate in aquatic organisms, and impact soil and crop quality [6,7]. Uranium also represents a strategic resource, and its recovery from industrial effluents is gaining attention [8].Accurate determination of uranium is critical for environmental monitoring and resource management. Techniques include spectrophotometry, alpha spectrometry, and plasma-based methods such as Inductively Coupled Plasma Optical Emission Spectrometry [9]. While ICP-OES offers multi-element detection with good precision, it requires complex sample preparation and may suffer from matrix effects.X-ray fluorescence spectrometry, especially WDXRF, offers

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rapid, non-destructive analysis with minimal sample preparation, making it suitable for industrial monitoring [10,11]. Previous studies have successfully applied WDXRF to trace element analysis in complex matrices [12,13]. Optimizing sample preparation and calibration ensures accurate uranium measurements in phosphate effluents [14,15,16]. This study aims to develop a WDXRF method for uranium quantification in phosphate effluents and discuss the environmental implications of uranium at detected levels.

Experimental:-

Materials:-

- **Spectrometer:-**

An X-ray fluorescence spectrometer (Magix 3kW, PW2403, PANalytical) was used for the determination of the uranium; it is a sequential spectrometer with wavelength-dispersive with a channel of measure based on a single goniometer covering the complete range of measure. It isequipped with an X-ray tube which is the X-ray source, the anode of the X-ray tube is in rhodium.

- **scintillation detector:-**

It is constituted by a crystal of iodide of sodium in which the atoms of thallium are distributed in a homogeneous way (NaI; Tl), by a photocathode and by a tube photomultiplier. It works by converting the X-ray in light which is then measured with a photomultiplier.

Sample preparation:-

The phosphate industry effluent samples were analyzed directly, without any prior chemical treatment, only filtration through 0.45 µm membranes and homogenization. Each sample was placed in a specialized liquid sample holder with a polymer film at the bottom, allowing direct exposure to the incident X-rays. In our setup, an inverted optical configuration was used, with the X-ray tube positioned beneath the sample. A critical factor in this arrangement is the mechanical strength of the film. Film breakage during analysis could result in liquid leakage and potential damage to the instrument. Therefore, it is essential to select a film that is sufficiently resistant while minimizing X-ray absorption and avoiding additives that might interfere with the measurement. For this study, we used MYLAR® X-ray film (63.5 mm diameter), which has minimal X-ray absorption and no effect on the analytical matrix. Each sample of phosphate industry effluent (3 g, measured with 0.1 mg precision) was placed in the cup. Measurements were conducted under a helium atmosphere at 900 hPa to prevent boiling under vacuum conditions.

Measurement conditions:-

Measurement conditionsusing WDXRF aresummarized as follows;
•Atmosphere: Helium (900 hPa pressure)
•Power: 60kV, 50 mA
•Crystal:LiF220
•Collimator: 150microns
• X-ray: U-Lα1 line of 13.631 keV energy.

The WDXRF analysis of uraniumis shown in Figure 1.

Figure1.Detectionof uraniumpeak usingthecrysta lLiF220

- The experimentalangleof the lineof uranium: U-Lα1 (2θ°) [noted U-LA1]:37.247°

Results and Discussion: -

Linearity, detection limit and quantification limit: -

The linearity of the proposed method was investigated by repeating the analysis (n = 6) at six concentration levels of 0, 5, 10, 15, 20 and 30 ppm. The intensity obtained at each concentration was plotted against the initial concentration of uraniumthe linear regression equation was evaluated in the statistical treatment of calibration data. The characteristics of the regression were calculated using Microsoft Excel™.

We have the following curve in figure 2,

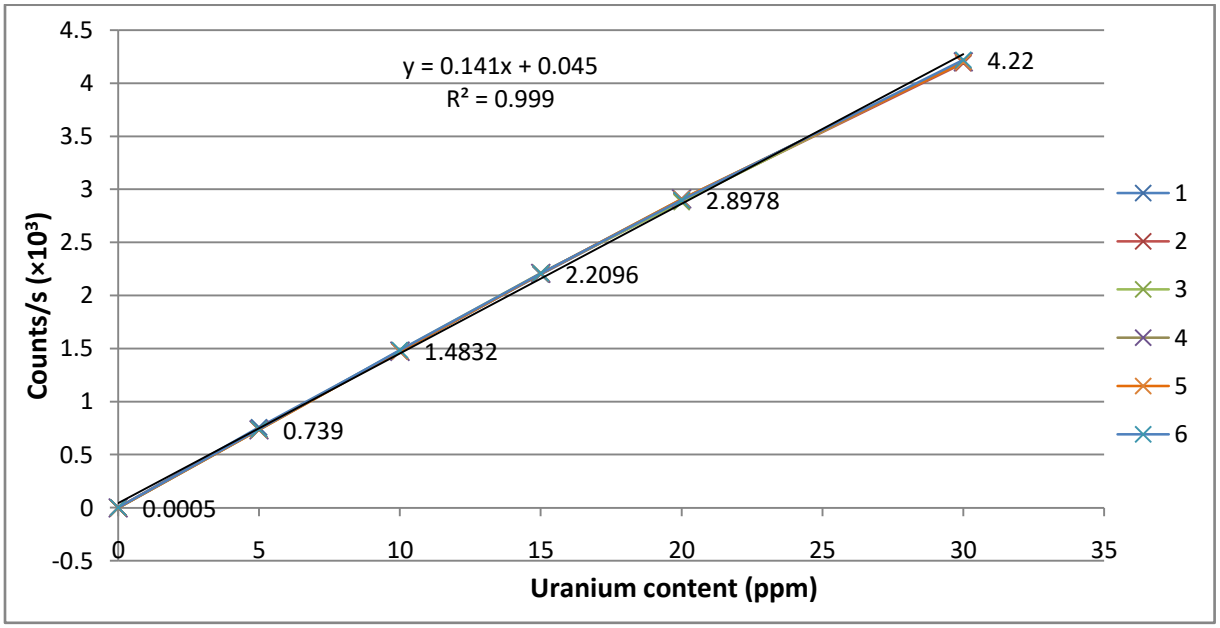

Figure2. Calibration of the methodfor the determination ofuranium.

The measurement collectedto performthe linearitycan be used tocalculate thesensitivity (b_1) andits standard deviation $S(b_1)$ and theblank value$b_0$andits standard deviation $S(b_0)$.

These standard deviations ($S(b_1)$ and $S(b_0)$) define as follows, the limits ofdetection and quantification, based on the twodefinitions:

The limit of detection: $LD = \frac{b_0+3.S(b_0)}{b_1}$ (1)

The limit of quantification: $LQ = \frac{b_0+10.S(b_0)}{b_1}$(2)

$b_1 = \frac{SPE\,(x,y)}{SCE\,(x)}$(3)

$b_0 = \bar{\bar{y}} - b_1.\bar{\bar{x}}$ (4)

$S(b_1) = \sqrt{\frac{s^2(e)}{SCE\,(x)}}$standard deviation of the sensitivityb_1 (5)

$S(b_0) = \sqrt{s^2(e)\left(\frac{1}{np} + \frac{\bar{\bar{x}}^2}{SCE\,(x)}\right)}$standard deviation of the blankb_0 (6)

$s^2(e)$: experimental variance of the regression.

$SCE(x)$: Sum of squared deviations for the variable x.

$SPE(x,y)$: Sum of the products of deviations for the variable x and y.

The results of thestudylinearity testare summarized in the following Table 1:

Table 1. Statistical results of the calibration

Designation	Observed value for uranium
Number oflevels(p)	6
Total number of measurements	36
Sensitivityb_1	0.141 ppm
Blank value b_0	0.0456 ppm
Equationoflinearregression	Y=0.141 X+0.0456
Correlation coefficient	0.9992
Standard deviation ofsensitivity$S(b_1)$	7.53×10^{-4}
Standard deviation of theblank value $S(b_0)$	0.0124
Detection limit (DL)	0.26 ppm
Quantification limit (QL)	0.88 ppm

- The results of the test of evaluation of the linearity showed that the model of regression is considered acceptable for uranium and that the domain of linearity is validated.

Reproducibility: -

This test is used to calculate the intra-laboratory reproducibility of the method studied, that is to say his fidelity when repetitions are made by several operators or longer time intervals with respect to the method. A sample was analyzed for 10 different days (p = 10) with 3 replicates (n = 3). The results are summarized in Table 2.

Table 2. Results of reproducibility test

Day	Replicates			Average	Variance	$S_r^2(x)$	$S_L^2(x)$	$S_R(x)$	$CV_R(\%)$
	1	2	3						
1	9.875	10.112	9.8	9.929	0.026	0.019	0.006	0.1615	1.602
2	10.215	10.234	9.997	10.148	0.017				
3	10.187	10.204	10.321	10.237	0.005				
4	9.978	9.859	10.12	9.985	0.017				
5	10.298	10.301	10.297	10.298	4.33333E-06				
6	10.025	9.947	10.201	10.057	0.017				
7	10.157	9.964	9.87	9.997	0.021				
8	10.1	10.237	9.879	10.072	0.033				
9	9.859	10.108	10.29	10.085	0.047				
10	10.008	9.978	10.194	10.06	0.014				

To calculate the variance of internal reproducibility, we relyon these formulas:

$N' = N - \frac{\sum_{i=1}^{p} n_i^2}{N}$ Corrected average numberof repetitions (7)

$S_L^2(x) = \frac{(p-1)\left(\frac{SCE_L(x)}{p-1} - S_r^2(x)\right)}{N'}$ Variance inter-sample (8)

$S_R^2(x) = S_L^2(x) + S_r^2(x)$ Variance of internal reproducibility (9)

$CV_R(\%) = \frac{S_R(x)}{\bar{\bar{x}}} \times 100$ Coefficient of internal reproducibility (10)

n_i : repetition number per day (n=3)

N : total number of measurements

$SCE_L(x)$: Sum of squared inter-sample differences

$S_r^2(x)$: variance internal repeatability

$S_R(x)$: standard deviation of internal reproducibility

- We obtain a value of coefficient of variation of reproducibility intra-laboratory CV_R:

$$CV_R = 1.6\ \%$$

Fidelity in terms of internal reproducibility is acceptable.¬

Discussion: -

The measured uranium concentration of approximately 10 ppm (10 mg/L) in the analyzed effluents is consistent with values previously reported for phosphate industry wastewaters [17,18]. The low coefficient of variation (CV = 1.6%) further confirms that wavelength-dispersive X-ray fluorescence (WDXRF) is a reliable and reproducible technique, suitable for the direct analysis of liquid effluents without prior complex sample preparation process.

From an environmental perspective, a uranium concentration of 10 mg/L is considerably higher than internationally recommended limits. The World Health Organization establishes a guideline value of 30 µg/L (0.03 mg/L) for uranium in drinking water [19], while the United States Environmental Protection Agency sets a maximum contaminant level (MCL) of 30 µg/L [20]. This indicates that the uranium concentration measured in the effluents exceeds these limits by more than two orders of magnitude (over 300 times higher), highlighting its potential hazard. Although these standards are designed for drinking water, they provide a useful reference for evaluating environmental risk. Uranium toxicity in aquatic environments is primarily chemical rather than radiological, with documented nephrotoxic effects in living organisms [21]. The International Atomic Energy Agency emphasizes that uranium released into aquatic systems can persist, undergo speciation changes, and accumulate in sediments and biota [22].

Ecotoxicological studies suggest that harmful effects on aquatic organisms may occur at concentrations as low as a few µg/L to several hundred µg/L, depending on species sensitivity and environmental conditions such as pH, salinity, and carbonate concentration [23,24]. In this context, the measured concentration of 10 mg/L (10,000 µg/L) is several orders of magnitude higher than thresholds associated with chronic and acute toxicity. Potential impacts include reduced growth, reproductive impairment, and increased mortality in fish, invertebrates, and microorganisms, which is observed in the studied region. In marine environments, dilution may initially reduce uranium concentrations following discharge. However, uranium tends to form stable carbonate complexes in seawater, enhancing its mobility and persistence [22]. Furthermore, uranium can adsorb onto suspended particles and eventually accumulate in marine sediments, creating a long-term contamination source. This sediment-bound uranium may later be remobilized under changing physicochemical conditions, posing prolonged risks to benthic organisms and the broader marine food web [24].

Given these considerations, the discharge of untreated effluents containing uranium at concentrations around 10 ppm represents a significant environmental concern. To mitigate these risks, appropriate treatment technologies—such as adsorption, ion exchange, membrane filtration, or chemical precipitation—should be applied prior to discharge to reduce uranium concentrations to environmentally acceptable levels.Overall, these findings confirm that WDXRF is an effective, precise, and non-destructive method for monitoring uranium in phosphate industry effluents. At the same time, the elevated uranium concentrations detected underscore the necessity of implementing strict environmental controls and treatment strategies to protect aquatic ecosystems.

Conclusion:-

In conclusion, the present study demonstrates that WDXRF is a robust, reliable, and efficient technique for the direct determination of uranium in phosphate industry effluents, eliminating the need for complex sample preparation. The

low coefficient of variation (1.6%) confirms the high precision and reproducibility of the method, supporting its suitability for routine monitoring applications.However, beyond the analytical performance, the results reveal a critical environmental concern. The measured uranium concentration of approximately 10 ppm (10 mg/L) is markedly higher than internationally accepted limits, such as the 30 µg/L guideline established by the World Health Organization and the United States Environmental Protection Agency. This significant exceedance highlights the potential for severe environmental contamination if such effluents are discharged without adequate treatment.

At these elevated concentrations, uranium poses both chemical and radiological risks to aquatic ecosystems. Its persistence in water, tendency to form stable complexes in marine environments, and capacity to accumulate in sediments and biota increase the likelihood of long-term ecological impacts, including toxicity to aquatic organisms, bioaccumulation, and potential transfer through the food chain. These risks are further emphasized by international assessments, including those of the International Atomic Energy Agency, which underline the environmental mobility and persistence of uranium.Therefore, this study not only validates WDXRF as an effective analytical tool but also underscores the urgent need for strict environmental management of phosphate industry effluents. The implementation of appropriate treatment technologies and continuous monitoring strategies is essential to reduce uranium concentrations to safe levels and to prevent long-term contamination of aquatic and terrestrial ecosystems.Overall, the findings highlight the dual importance of accurate analytical techniques and proactive environmental protection measures in addressing uranium pollution and safeguarding ecosystem and public health.

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