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Application of inductively coupled plasma optical emission spectrometer in water quality monitoring

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ABSTRACT

Agriculture has high requirements for both the quality and quantity of water resources. However, the traditional monitoring methods used in the field of water quality monitoring for agricultural irrigation suffer from issues such as complex operation, low automation, and low efficiency. To address these problems, this study proposes an agricultural irrigation water quality monitoring system based on an inductively coupled plasma optical emission spectrometer and designs the usage process and strategies of the spectrometer according to the characteristics of agricultural irrigation water. The water quality monitoring system can precisely monitor heavy metal elements that may contaminate agricultural irrigation water and eliminate the interference of other impurities. Experimental results show that the system achieves a correlation above 0.999 for the standard curves of five major heavy metal elements, and the signal-to-noise ratio of heavy metal elements reaches 12.56 when the system carrier gas flow rate is 0.8 L/min. Compared to traditional detection methods, the system exhibits higher detection speed, automation, and comparable accuracy, thus demonstrating potential for practical application in the field of water quality monitoring for agricultural irrigation.

Keywords: Inductively coupled plasma; Water quality monitoring; Irrigation water; Heavy metal

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1. Introduction

For modern agriculture, irrigation water requires a significant amount and has high standards for water quality [1]. According to relevant national regulations, the pH value of agricultural irrigation water must be within the range of 5.5–8.5, and the content of heavy metal elements must be strictly controlled. Specifically, the cadmium and its compounds should not exceed 0.005 mg/L, copper and its compounds should not exceed 1 mg/L, and zinc and its compounds should not exceed 3 mg/L. If the concentration of heavy metal elements in irrigation water is too high, it can have irreversible negative effects on crops and soil [2]. In the fields of agricultural water rescue and water quality monitoring, some researchers have conducted meaningful experimental studies. For instance, Ji et al. [3] studied the changes in water quality for rural water supply in Luzhou City over a span of 5 y, and pointed out that the water quality scores have been improving in recent years. Gowda et al. [4] focused on the dynamic changes of water quality in surface water and groundwater sources in the kiwifruit production area at the northern foot of Qinling Mountains, and conducted fixed-point toilet cleaning to analyze the nitrate and salt ion levels in different water sources under different land-use patterns. The results indicated that the nitrate levels in the natural vegetation-covered area in the region did not exceed the standard, while there were excessive nitrate levels in the concentrated kiwifruit production area.

In traditional water quality monitoring methods, dealing with heavy metal elements relies heavily on manpower, and

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single-run testing capabilities are limited, resulting in low efficiency [5]. In light of this, a water quality monitoring system for agricultural irrigation water based on an inductively coupled plasma optical emission spectrometer is proposed to address the monitoring of heavy metal elements in agricultural irrigation water. Inductively coupled plasma-optical emission spectrometry (ICP-OES) boasts high sensitivity and accuracy, enabling simultaneous determination of multiple elements, and has been widely applied in fields such as materials science and geology [6]. Teramoto and Kim [6] utilized ICP-OES to design a method for the determination of metal elements in marine materials, and the calibration curves for each element exhibited good linear relationships with correlation coefficients not less than 0. Mortada and Alharthi [7] developed an ICP-OES method based on microwave digestion for accurate measurement of metal elements in soil, demonstrating low detection limits. Surksum and Fisher [8] developed an ICP mass spectrometry method for the determination of indium in flue ash, and the results showed high correlation coefficients of calibration curves (0.9994) and recovery rates ranging from 98% to 102%. Serrano et al. [9] led his team to establish a method for vortex-assisted extraction coupled with inductively coupled plasma optical emission spectrometry, which was applied to the precise determination of metal elements in olive oil. The results demonstrated high accuracy of this approach. Dospatliev and Ivanova [10] investigated the correct measurement of sulfur in soil under different digestion methods using inductively coupled plasma optical emission spectrometry. They constructed working curves using different standard substance digestion solutions and studied the impact of working curves and spectral interference on the analysis results. The research findings showed good agreement between the theoretical derivation of spectral interference and the experimental results in terms of trend. Currently, ICP-OES has been widely applied in element monitoring in various fields. In this study, the application of ICP-OES in water quality monitoring for agricultural irrigation was explored, and the monitoring process and details were designed based on the characteristics and requirements of agricultural irrigation, with the aim of making practical contributions to water quality monitoring in agriculture and forestry.

2. Application design of ICP-OES in water quality monitoring of agricultural irrigation water

2.1. Structural design and analysis of ICP-OES

The structure of the ICP-OES system for water quality monitoring in agricultural irrigation is shown in Fig. 1. The main components of this system include the sample introduction system, data and control system, grating system, and light source system. The main components of the light source system are the plasma torch and RF generator. The plasma torch consists of a Fassel torch, which is composed of three concentric quartz tubes, and the RF generator utilizes a 26.97 MHz frequency oscillator circuit with a maximum output power of 1.6 kW. The grating system has a spectral response range of 160–900 nm, allowing for coverage of the ultraviolet to visible light spectrum. As an essential component, the control system is responsible for functions such as signal acquisition and communication with the host computer. It includes the I/O control center, software, and data workstation. The sample introduction system comprises the nebulization system, gas path system, and others, which primarily handle the measurement samples for analysis by the ICP-OES.

In the ICP-OES system, the inductively coupled plasma is generated from ionized argon gas, and the raw material for the argon gas is high-purity argon. In practical operations, liquid argon should be preferably used as the raw material because the operation of the ICP-OES instrument requires a high amount of argon gas [11]. Due to the high excitation efficiency of the inductively coupled plasma, multiple elements can exhibit low limits of detection, making the determination of multi-metal elements feasible [12]. On the other hand, in the ICP-OES system, samples are introduced and analyzed in the form of aerosols in the plasma, where the temperature of the samples is in the range of 6,000–8,000 K. In this state, the samples are almost completely dissociated, and

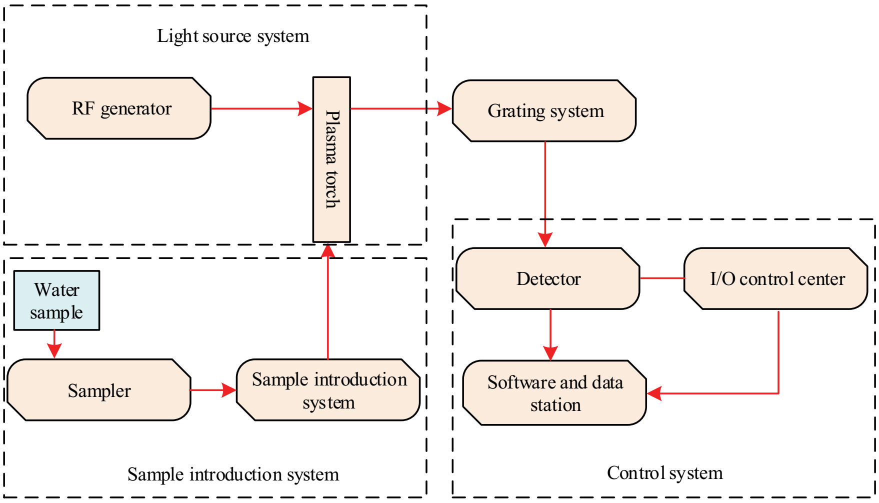

Fig. 1. ICP-OES structure for monitoring water quality in agricultural irrigation.

the adverse effects of chemical interferences in the ICP-OES are significantly reduced [13]. After completing the structural design of the ICP-OES system, it is necessary to further calculate its limit of detection, precision, and accuracy. The calculation method for the limit of detection is as shown in Eq. (1).

$$t\left(N-1,0.99\right)\cdot S=D \tag{1}$$

where D represents the limit of detection; N denotes the number of parallel determinations for blank samples; t represents the t-distribution value; and S is the standard deviation of the parallel determinations. In practical operations, the number of parallel determinations is typically set as 7. The limit of detection is an important indicator in water quality monitoring, which evaluates the minimum concentration of an element required for the monitoring system to detect and analyze. A smaller value indicates a more sensitive water quality monitoring system. The calculation of instrument precision primarily utilizes the relative standard deviation as the main indicator, which evaluates the absolute value of data dispersion. The value of the relative standard deviation is influenced by both the average value and the degree of dispersion of the detected object [6]. A higher relative standard deviation indicates a larger measure of the object's dispersion. The mathematical expression of the relative standard deviation is shown in Eq. (2).

$$v=\frac{\frac{\sqrt{\sum_{i=1}^{N}\left(x_i-\bar{x}\right)^2}}{n-1}}{\bar{x}} \tag{2}$$

where $\bar{x}$ represents the average value of the object; x_i denotes the value of the i-th measurement; n represents the total number of measurements; and v is the relative standard deviation. When testing the accuracy of the instrument, standard substances are mixed into the sample for determination, and the recovery rate is calculated through multiple recovery experiments and the average value is obtained. In the recovery rate test, it is crucial to ensure that the form of the standard substance and the substance under test are consistent. Additionally, the amount of the standard substance should not exceed three times that of the substance under test, as a higher amount can lead to decreased accuracy of the recovery rate test results. The accuracy of the ICP-OES instrument is considered appropriate within the range of 95%–105%. The calculation method for instrument accuracy is shown in Eq. (3).

$$R=\frac{T_A-T}{A} \tag{3}$$

where R represents the recovery rate of the instrument; T_A denotes the measurement value of the spiked sample; T represents the measurement value of the sample; and A is the spiked value.

2.2. Water sample processing and machine analysis strategies for monitoring the quality of agricultural irrigation water

The water sources for agricultural irrigation are relatively complex and mainly include river runoff, surface runoff, and subsurface runoff. In some areas, reclaimed water from urban sources is also utilized for agricultural irrigation [14]. Runoff water sources and reclaimed water sources often have complex compositions and may contain impurities. Direct analysis without proper sample pretreatment may result in erroneous analysis results and, in severe cases, could lead to instrument clogging and contamination. Therefore, pretreatment of the test water samples is necessary [15]. In the ICP-OES system, nitric acid digestion is employed for impurity removal during water sample pretreatment. The principle is to utilize the oxidizing effects of nitric acid and hydrogen peroxide to convert the metal elements in the test water sample into easily separable inorganic compounds at high temperatures. Organic impurities in the sample are destroyed, thus no longer affecting water quality monitoring [16]. The specific steps of nitric acid digestion pretreatment are shown in Fig. 2.

Nitric acid digestion treatment can effectively eliminate the effects of most sediment and organic matter in practical applications. It requires very low manual operation on the basis of automation, greatly reducing the health hazards of chemical substances to operators. The operators only need to monitor for potential issues such as leakage. In comparison, the traditional treatment method uses a manually-acidified hot plate digestion method, which is difficult to automate and relies heavily on the operator's experience, observation skills, and operational level. It not only consumes more time but also has lower accuracy and higher error rates. Moreover, prolonged exposure of the operator to corrosive chemicals can harm their health [17]. Overall, nitric acid digestion treatment is superior to the traditional treatment method in all aspects.

Once the water sample pretreatment is completed, the next step is the on-machine analysis of the test water samples. The on-machine analysis of the test water sample is

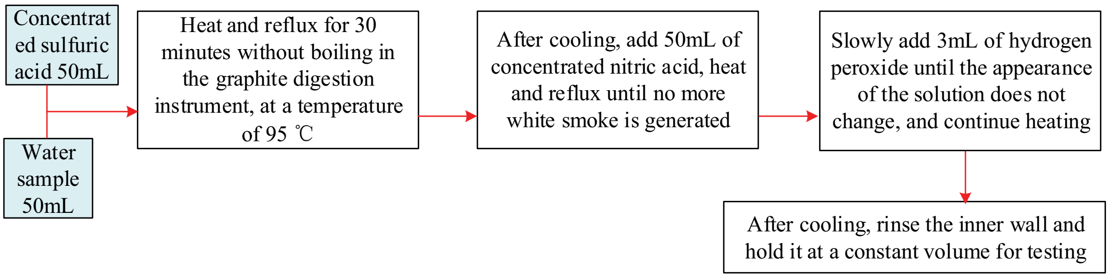

Fig. 2. Nitric acid digestion pretreatment of water samples.

one of the most crucial steps in ICP-OES water quality monitoring, with a primary focus on two aspects: the selection of analysis element spectral lines and the identification and correction of interference factors. The selection of analysis element spectral lines is primarily based on the content of the target element. Major elements use less sensitive lines, while trace and ultra-trace elements generally require more sensitive lines. In case the selected spectral line is interfered by coexisting elements or other factors, alternative spectral lines can be considered. The identification and correction of interference factors is also critical. Common interference factors in water quality analysis include physical interference, spectral interference, chemical interference, and inter-element interference. Physical and chemical interference can be conveniently addressed by diluting the test water sample, adding ion buffer, internal standards, and matrix matching. Spectral interference can be mitigated through techniques like wavelength variations and sub-array plot optimization. Inter-element interference significantly impacts water quality monitoring results. For example, Table 1 describes the influence of zinc at different concentrations on other metal elements in water samples. Zinc exhibits substantial interference with low concentrations of lead, cadmium, and copper. However, at high concentrations, the interference is relatively minimal for these three elements, while other interferences between elements may manifest differently [18]. To address inter-element interference, ICP-OES systems employ background subtraction and interference factor correction methods, which offer similar effectiveness compared to common separation and matrix matching approaches but are more convenient in practice.

3. Performance analysis of ICP-OES and application experiments in water quality monitoring

3.1. Performance analysis of ICP-OES

The evaluation of ICP-OES will be conducted from two perspectives: the performance and associated data of the ICP-OES system, and the performance of ICP-OES in practical water quality monitoring activities, as well as comparisons with other similar systems. Regarding the analysis of performance and associated data, the first step is to calculate the standard curves for the major metal elements in water quality monitoring. The standard curve describes the functional relationship between the concentration of the analytes and the response values of the instrument used for measurement. The accuracy of the standard curve affects the accuracy of the measurement results and also reflects the characteristics of the instrument. To create the standard curves, a mixed standard solution is added to six volumetric flasks, and nitric acid is used to dilute each flask to standard solutions with concentrations of 0, 0.2, 0.5, 1, 2, 3, and 5 mg/L, respectively. The measurements are then taken, and the results are shown in Table 2.

Table 1
Interference of zinc on other metal elements

Elements (mg/L)		Zinc (0.01 mg/L)	Zinc (1.0 mg/L)	Zinc (10 mg/L)
Error rate (%)	Cadmium (0.01 mg/L)	–100.00	–100.00	26.00
	Cadmium (1.0 mg/L)	0.08	1.9	2.36
	Cadmium (10 mg/L)	0.12	0.03	0.10
	Lead (0.01 mg/L)	100.00	514.00	903.00
	Lead (1.0 mg/L)	–48.99	–43.99	–14.72
	Lead (10 mg/L)	–3.18	–2.18	–2.28
	Copper (0.01 mg/L)	36.00	56.00	72.00
	Copper (1.0 mg/L)	–4.64	–5.69	–7.53
	Copper (10 mg/L)	–0.29	–0.18	0.12

Table 2
Calculation results of metal element standard curve

Solution concentration (mg/L)		0	0.2	0.5	1.0	2.0	3.0	5.0
Ionic strength	Cadmium	4.921	1,917	4,833	9,412	18,240	28,210	46,960
	Copper	209	6,285	15,492	29,808	60,460	89,859	149,413
	Nickel	–0.4422	1,019	2,574	4,987	9,637	14,782	24,583
	Lead	1.223	32.46	80.22	156.9	304.5	468.1	782.8
	Zinc	9.086	2,810	6,979	13,414	26,013	39,727	65,604
Element standard curve	Cadmium	$Y = 9373.1X - 12.66$; $R = 0.9999$						
	Copper	$Y = 29838X + 346.59$; $R = 1$						
	Nickel	$Y = 4903.6X + 30.05$; $R = 1$						
	Lead	$Y = 155.99X + 0.153$; $R = 1$						
	Zinc	$Y = 13092X + 196.95$; $R = 1$						

In the standard curve equation, *Y* represents the ionic intensity of the target element, and the coefficients and constants are the slope and intercept of the standard curve, respectively. After measuring common elements such as cadmium, copper, nickel, lead, and zinc, it was found that all five elements exhibit good curve linearity, with high correlation coefficients meeting the required standards. It is generally accepted that the *R*-value in water quality monitoring should be greater than 0.999. In the experiment, the *R*-values for copper, nickel, lead, and zinc were all 1, while for cadmium, it was 0.9999. This result indicates that the ICP-OES meets the application standards for element standard curves. After completing the calculation of the standard curves, the signal-to-background ratio (SBR) relationship of the ICP-OES system at different nebulizer flow rates was analyzed. The nebulizer gas flow rate significantly influences the measurement results of the ICP-OES system, as both the signal intensity and background signal intensity vary with changes in the nebulizer gas flow rate and pressure. By studying the effect of the nebulizer gas flow rate as a variable on the SBR of major elements in the ICP-OES system, the results are shown in Fig. 3.

From Fig. 3 the SBR of the ICP-OES system is sensitive to changes in the nebulizer gas flow rate. Zinc and cadmium show the highest sensitivity to the nebulizer gas flow rate, while nickel and lead exhibit relatively lower sensitivity. Additionally, most elements show an increasing trend in SBR with an increasing nebulizer gas flow rate within a certain range, but beyond that range, the SBR decreases with an increasing nebulizer gas flow rate. In the ICP-OES system, the critical point is at 0.8 L/min. When the nebulizer gas flow rate is below this value, the SBR increases with an increasing gas flow rate, and when the gas flow rate exceeds this value, the SBR decreases. Copper, on the other hand, demonstrates a unique characteristic in the gas flow rate-SBR relationship, with its highest value not at 0.8 L/min but at 1.0 L/min, where the SBR is 5.73. Since all other elements reach their maximum SBR at a gas flow rate of 0.8 L/min, it is recommended to use that value as the nebulizer gas flow rate in the ICP-OES system. The next step is to analyze the detection limits and precision of the ICP-OES system in practical applications. For comparison, an atomic absorption spectrophotometry system is used as a control group. Atomic absorption spectrophotometry is a widely used method in the field of metal element measurement. The comparison of detection limits between the two methods is shown in Fig. 4.

Fig. 4 depicts the detection limits of the ICP-OES system and atomic absorption spectrophotometry system for several major elements in water quality monitoring, including nickel, cadmium, zinc, lead, and copper. It can be observed that the detection limits for nickel, zinc, and lead are lower in the ICP-OES system compared to the atomic absorption spectrophotometry system. Among these, the difference in detection limits is most significant for lead, with the ICP-OES system having a lead detection limit of 0.01 mg/L, whereas the atomic absorption spectrophotometry system has a lead detection limit of 0.02 mg/L, which is twice that of the ICP-OES system. Both systems demonstrate consistent detection limits for cadmium and copper, with a detection limit of 0.001 mg/L for cadmium and 0.004 mg/L for copper. Overall, the ICP-OES system exhibits lower detection limits compared to the atomic absorption spectrophotometry system, indicating that ICP-OES is more sensitive to metal elements in water quality analysis and provides more accurate detection results. Precision testing of the ICP-OES system was performed by conducting 12 parallel measurements and calculating the mean, relative standard deviation, and coefficient of variation. The results are summarized in Table 3.

Table 3 describes the precision performance of the ICP-OES system and atomic absorption spectrophotometry

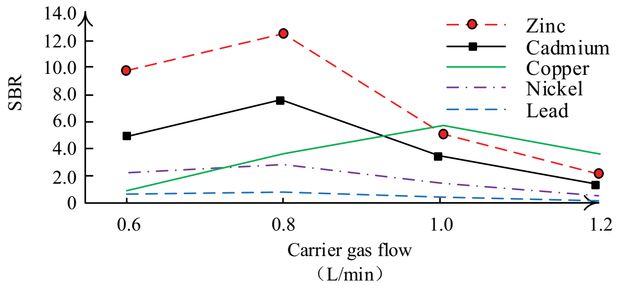

Fig. 3. Effect of different atomizer carrier gas flow rates on SBR of ICP-OES.

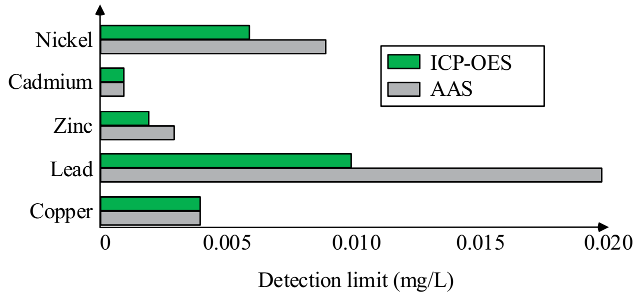

Fig. 4. Comparison of water quality monitoring system detection limits.

Table 3
Precision testing and comparison results of the ICP-OES system

Elements		Copper	Lead	Zinc	Cadmium	Nickel
Mean measurement (mg/L)	AAS	0.4861	0.5902	0.1481	0.673	0.4533
	ICP-OES	0.4883	0.5827	0.1466	0.6668	0.4202
Relative standard deviation (%)	AAS	8.0	0.7	2.0	1.2	16.0
	ICP-OES	4.0	0.5	0.5	0.4	11.2

system for various major elements. The precision of the systems is described based on the mean value and relative standard deviation. From the mean value perspective, the measurement values from both systems are relatively close. However, there are significant differences in the relative standard deviations. The relative standard deviation of the ICP-OES system is significantly lower than that of the atomic absorption spectrophotometry system for all five major water quality monitoring elements. For example, the relative standard deviation of zinc in the ICP-OES system is only 0.5%, while it is 2% for the atomic absorption spectrophotometry system, which is four times higher than that of the ICP-OES system. Similarly, the relative standard deviation for nickel in the ICP-OES system is 11.2%, whereas it is 16% for the atomic absorption spectrophotometry system, resulting in a significant difference of 4.8%.

3.2. ICP-OES water quality monitoring application experiment

In the field of water quality monitoring, graphite furnace atomic absorption spectrophotometry (GFAAS) is one of the most widely used instruments. Therefore, in the application experiment section, graphite furnace atomic absorption spectrophotometry is used as a comparison to evaluate the practicality of the ICP-OES system. First, a standard solution of nickel with a concentration of 15 μg/L is prepared. Then, the ICP-OES system and the graphite furnace atomic absorption spectrophotometry system are used to perform 11 replicate measurements, and the results are observed. The measurement results are shown in Fig. 5.

Fig. 5 depicts the measurement performance of the GFAAS and ICP-OES systems in 11 replicate measurements using a scatter plot. It can be observed that the deviations between the two testing methods are not significant. The highest measurement values for both methods in the 11 measurements are 15.7 μg/L. When observing the lowest measurement values, the ICP-OES system has a minimum of 14.6 μg/L, while GFAAS has a minimum of 14.2 μg/L. Based on the lowest measurement values, the ICP-OES system is closer to the true value. Overall, the measurement capabilities of both methods are similar. The mean measurement value for GFAAS is 15.1, with a calculated standard deviation of 0.491 and a relative standard deviation of 3.1%. The ICP-OES system has a mean calculated value of 15.2, a standard deviation of 0.371, and a relative standard deviation of 2.3%. The *t*-test shows that there is no significant difference between the two methods. This result indicates that the ICP-OES system is very close to the GFAAS system in terms of elemental measurement results. After the measurement experiment, further tests were conducted to evaluate the recovery rates and the correlation of calibration curves for both measurement methods, as shown in Table 4.

Table 4 presents the recovery rates and related indicators, which were obtained by adding standard solution to water samples and conducting multiple replicate measurements. The correlation of calibration curves was determined by linear regression analysis after diluting nitric acid solutions. It can be observed that the ICP-OES system exhibits better linearity in calibration curves, with a correlation coefficient of 0.9995, compared to 0.9988 for GFAAS. In terms of recovery rates, GFAAS shows recovery rates between 95.2–107 μg/L, while the ICP-OES system demonstrates recovery rates between 96.6–104 μg/L. In comparison, the recovery rates of the ICP-OES system are closer to 100%. This result indicates that both monitoring methods achieve good recovery rates, indicating good accuracy, with a slight advantage for the ICP-OES system. Similar conclusions can be drawn when examining the measured values of both methods. The measured values for the ICP-OES system fall between 9.67–10.5 μg/L, while GFAAS ranges from 9.37–10.7 μg/L. It

Table 4
Comparison of the recovery rates and calibration curve correlation of two methods

Method	GFAAS	ICP-OES
Water sample concentration (μg/L)	10	10
Adding standard matter amount (μg/L)	5	5
Measured value (μg/L)	9.37–10.7	9.67–10.5
Total measured amount (μg/L)	14.27–16.09	14.47–15.77
Recovery rate (μg/L)	95.2–107	96.6–104
Calibration curve correlation	$R = 0.9988$	$R = 0.9995$

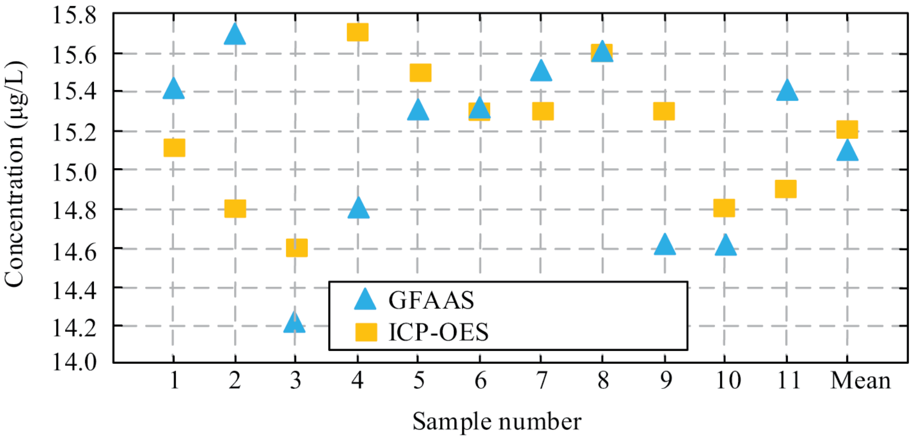

Fig. 5. Comparison of measurement results of standard nickel solution.

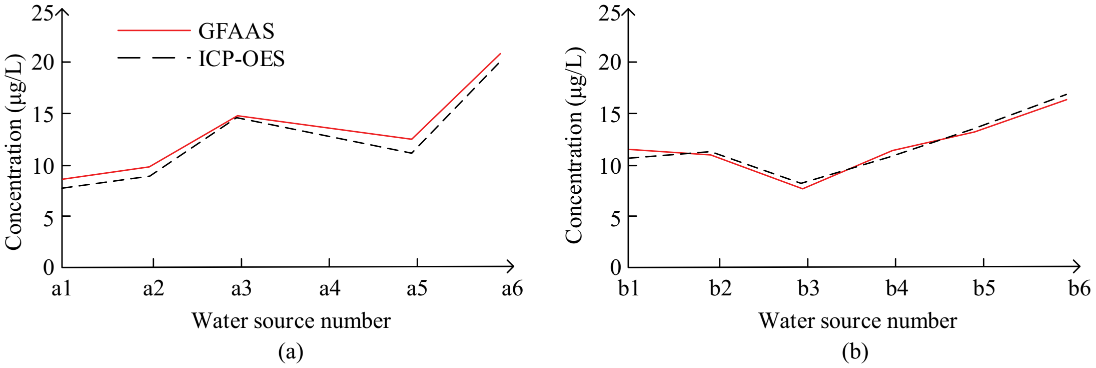

Fig. 6. Experimental results of agricultural irrigation water source monitoring.

can be observed that the ICP-OES system provides results closer to the true value, although the difference between the two methods is small. The main application field of this ICP-OES system is water quality monitoring for agricultural irrigation. To evaluate the actual water quality monitoring capabilities and practical value of the ICP-OES system accurately, multiple tests using different water sources as samples are required. In this study, six agricultural irrigation water sources were selected from each of two regions, and monitoring experiments were conducted using the ICP-OES system and GFAAS. The results are shown in Fig. 6.

Fig. 6a shows the water quality monitoring results for both methods in the first region, while Fig. 6b shows the results for the second region. It can be observed that the monitoring results for both methods in both regions are very close, indicating that the practical performance of the two methods is very similar, with almost no difference in accuracy. Furthermore, in actual water quality monitoring work, GFAAS can only measure one heavy metal element at a time, and its operational process is more cumbersome compared to ICP-OES. Additionally, GFAAS has characteristics such as low correlation coefficients of calibration curves and higher atomization temperature requirements. Therefore, in practical applications, the ICP-OES system has lower costs compared to GFAAS, while maintaining a similar level of accuracy.

4. Conclusion

Agricultural irrigation water sources are diverse, including natural water resources, sewage, and reclaimed water. To ensure the safety of irrigation water and the healthy growth and yield of crops, the water quality for agricultural irrigation needs to meet strict standards. Among the potential factors that can cause contamination to crops and soil, heavy metal elements are common pollutants. In addressing the issue of heavy metal contamination in agricultural irrigation water, a strategy of applying ICP-OES for water quality monitoring in agricultural irrigation has been proposed. The detection process and details have been designed based on the characteristics and requirements of agricultural irrigation water. Experimental results demonstrate that the ICP-OES water quality monitoring system based on ICP-OES achieves a calibration curve correlation of 0.9995 for nickel, which is higher than the commonly used detection methods in the field of water quality monitoring. Furthermore, the average measured value of the ICP-OES system in the elemental measurement experiments differs from the true value by only 0.1 μg/L. The experimental results for the detection limit of the ICP-OES system indicate that it is generally superior to the widely used atomic absorption spectrophotometry system. For example, the detection limit for lead with the ICP-OES system is 0.01 mg/L, while the atomic absorption spectrophotometry system has a detection limit of 0.02 mg/L for lead. The experimental results demonstrate that, compared to the commonly used detection methods in the field of water quality monitoring for agricultural irrigation, the ICP-OES system has higher practicality. This study has achieved relatively successful results. However, there are still certain limitations. The system is primarily designed to address heavy metal contamination in agricultural irrigation water, but there are other pollution sources that need to be considered. In future research, membrane filters and filter cartridges could be added to the system to expand the range of detectable pollution sources.

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