

Multi-model integration for watershed nitrate source apportionment: Methods and applications

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Received: October 19, 2026; Received in revised form: October 25, 2026; Accepted: October 25, 2026; Available online: October 26, 2026

Abstract: Overlapping source signatures and nonlinear hydrochemical relationships complicate nitrate source apportionment at the watershed scale. This letter presents an integrated framework linking hydrochemical source identification, quantitative apportionment, and data-driven prediction, with nitrate isotopes providing complementary constraints on sources and transformations. The framework's core analytical components were demonstrated using Spearman correlation analysis, principal component analysis (PCA), positive matrix factorization (PMF), and XGBoost. Two principal components explained 75.9% of the total hydrochemical variance. PMF resolved mixed natural–anthropogenic, sewage-related, and mineral-dissolution factors, accounting for 54.4%, 23.9%, and 21.7% of the modeled hydrochemical contributions, respectively. XGBoost model achieved an overall classification accuracy of 0.833. K^+ , Ca^{2+} , Cl^- , and SO_4^{2-} collectively accounted for 59.7% of the feature importance, indicating the predictive relevance of major-ion signatures. Increasing the sample size could further improve predictive accuracy. These findings displayed the potential of integrating receptor modeling and machine learning for rapid source screening.

Keywords: Nitrate contamination; Source apportionment; Hydrochemistry; Dual nitrate isotopes; Machine learning

1. Introduction

Water resources are fundamental to sustaining human society and ecosystems [1]. However, human disruption of the nitrogen cycle has made nitrate one of the most widespread inorganic contaminants in aquatic environments [2, 3]. By 2020, shallow aquifers worldwide had accumulated approximately 162 ± 6 Tg N as nitrate, with shallow groundwater concentrations exceeding the World Health Organization drinking-water guideline (11.3 mg N L^{-1}) across approximately 10% of the global land area [4]. An additional estimated 4037 ± 214 Tg N remains stored as nitrate in the vadose zone. Even if nitrogen surpluses were eliminated immediately, projections indicate that concentrations would remain above the guideline beyond 2100 in approximately 4% of affected areas [4]. Elevated nitrate concentrations in drinking water can cause infant methemoglobinemia, and a nationwide cohort study links long-term exposure to concentrations as low as 3.87 mg N L^{-1} to increased colorectal cancer risk [5]. Reliable source identification and quantitative apportionment are therefore essential to watershed management and drinking-water protection.

Watershed nitrate contamination reflects multiple sources whose contributions vary across space and time. Major sources include agricultural fertilizers, soil organic nitrogen mineralization,

domestic sewage, livestock manure, atmospheric deposition, and industrial effluents [6]. Their relative importance and delivery pathways depend on land use and hydrological conditions [7]. Fertilizer inputs and soil nitrification dominate in agricultural areas, where nitrate transport is coupled to storm runoff and irrigation return flows [8]. In urban areas, sewage, industrial inputs, and runoff from impervious surfaces dominate [9]. Source overlap and nonlinear hydrological controls complicate source identification and contribution estimation.

Methods for nitrate source apportionment have evolved from qualitative screening to quantitative estimation, and from individual indicators to multiple complementary constraints. Hydrochemical diagrams and multivariate statistics initially supported source identification [10]. Receptor models, including absolute principal component scores–multiple linear regression (APCS-MLR) and positive matrix factorization (PMF), subsequently enabled quantitative estimates of source contributions [11, 12]. Dual nitrate isotopes ($\delta^{15}\text{N}\text{-NO}_3^-$ and $\delta^{18}\text{O}\text{-NO}_3^-$) provided additional constraints through tracer-based mass-balance approaches [12]. More recently, machine learning has expanded opportunities to integrate heterogeneous data and generate rapid predictions. These approaches nevertheless differ in their data requirements, assumptions, and inferential capabilities. No single method consistently combines accuracy, computational efficiency, and mechanistic interpretability across watershed settings, motivating their integration.

The central challenge, therefore, is to combine complementary methods within a coherent framework that respects their distinct assumptions and constraints. Here, we synthesize five methodological approaches (hydrochemical diagrams, receptor models, isotope tracing, and machine learning) to propose an integrated framework that links source identification, quantitative apportionment, and prediction by integrating data and models. The framework is intended to improve the efficiency and interpretability of watershed nitrate source apportionment and support targeted pollution control.

2. From source identification to prediction

2.1. Hydrochemical characterization and nitrate-source identification

Researchers commonly evaluate nitrate contamination characteristics using hydrochemical diagrams and ion-ratio analyses. Piper and Gibbs diagrams can reveal the dominant hydrochemical facies and clarify the relative importance of mineral dissolution, evaporation, precipitation, and anthropogenic inputs. Bivariate relationships between NO_3^- and major ions, along with characteristic ion ratios, further help distinguish potential nitrate sources such as domestic sewage, agricultural fertilizers, soil nitrogen, livestock manure, and mineral dissolution [10, 13]. These graphical approaches are particularly useful for preliminary source screening because they provide an intuitive representation of hydrochemical evolution and source-related signatures. However, different sources may produce overlapping chemical signals, while hydrological transport and biogeochemical transformations can modify the original composition. Hydrochemical diagrams therefore provide qualitative or semi-quantitative evidence and are most effective when combined with quantitative source-apportionment techniques.

2.2. Quantitative apportionment of nitrate sources

Correlation analysis was used to characterize associations among nitrogen pollution and major ions, thereby identifying potential relationships between nitrogen species and hydrogeochemical

variables [14]. Spearman's correlation coefficient was calculated to assess the direction and strength of monotonic relationships without assuming normal distributions. Statistical significance was evaluated at $p < 0.05$. Principal component analysis (PCA) was employed to reduce data dimensionality and identify dominant patterns of hydrochemical variation [15]. The Kaiser–Meyer–Olkin (KMO) test was used to assess sampling adequacy, and principal components were extracted based on the eigenvalue criterion and cumulative variance explained. Component loadings were examined to identify variables contributing most strongly to each component. Potential sources were interpreted by integrating the component loadings with hydrogeological conditions, lithological characteristics, and known anthropogenic activities in the study area.

Positive matrix factorization (PMF) was applied to quantitatively resolve potential sources and estimate their contributions to the observed hydrochemical variables [16]. The model represents the concentration matrix as the product of source contributions and source profiles, together with a residual matrix:

$$X_{ij} = \sum_{k=1}^p g_{ik} f_{kj} + e_{ij} \quad (1)$$

where X_{ij} is the measured concentration of species j in sample i , g_{ik} represents the contribution of factor k to sample i , f_{kj} denotes the profile of species j in factor k , and e_{ij} is the residual. PMF minimizes the uncertainty-weighted objective function:

$$Q = \sum_{i=1}^n \sum_{j=1}^m \left(\frac{e_{ij}}{u_{ij}} \right)^2 \quad (2)$$

where u_{ij} is the uncertainty associated with the corresponding concentration measurement. The number of factors was selected by considering model performance and the interpretability of source profiles. Model robustness was evaluated using residual distributions, observed–predicted agreement, Q statistics, and bootstrap and displacement diagnostics. Source identification was based on factor profiles and their consistency with the correlation analysis, PCA results, and local hydrogeochemical conditions.

2.3. Isotope analysis

Dual nitrate isotopes, $\delta^{15}\text{N}\text{-NO}_3^-$ and $\delta^{18}\text{O}\text{-NO}_3^-$, provide valuable constraints for distinguishing nitrate derived from synthetic fertilizers, soil organic nitrogen, manure, and sewage. Their combined application can also help identify transformations such as nitrification and denitrification, which alter nitrate concentrations and isotopic compositions during transport [17]. By integrating isotope signatures with hydrochemical indicators, hydrological connectivity, land-use patterns, and spatial sampling, source contributions can be traced along the watershed continuum from source areas to rivers, aquifers, reservoirs, and other receiving environments.

2.4. Data-driven prediction of nitrate sources

Data-driven methods extend source apportionment from retrospective analysis to rapid prediction. Machine-learning algorithms can capture nonlinear relationships between hydrochemical indicators and nitrate-source categories, integrating information from multiple variables without requiring explicit specification of all relationships. In this framework, hydrochemical measurements serve as predictor variables, while source-identification or source-apportionment results serve as response labels. Algorithms such as random forest (RF), support vector machine (SVM), and extreme

gradient boosting (XGBoost) can be trained to classify potential nitrate sources or predict source contributions [18, 19]. The road map was presented in the Fig. 1.

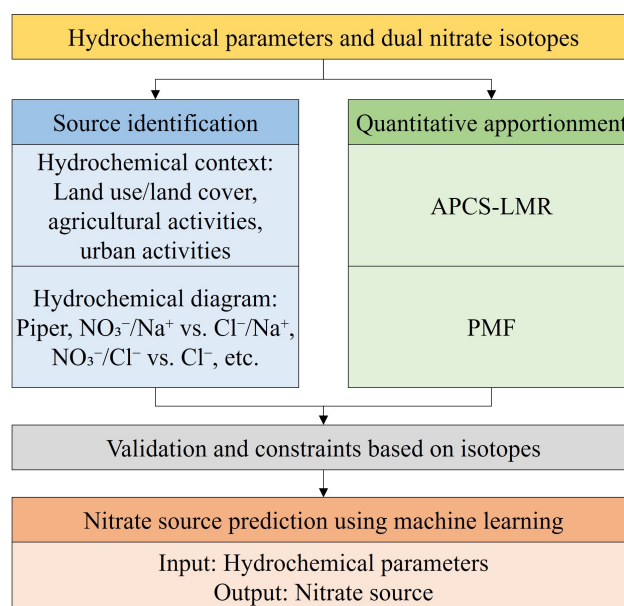


Figure 1. A collaborative multi-model technical roadmap for nitrate pollution source tracing.

3. Applications of nitrate source apportionment

3.1. Sample collection and laboratory experiment

The case study was conducted in a plain watershed in southwestern China during the dry season of 2023, where 134 surface-water samples were collected across the watershed for hydrochemical analysis and 18 samples for dual nitrate isotope analysis. Sampling sites represented upstream, midstream, and downstream sections and major land-use types, including urban, agricultural, and forested areas, while avoiding wastewater outlets, tributary confluences, and other disturbance-prone locations. All samples were collected from the central river channel and stored in 500 mL polyethylene bottles; bottles for cation analysis were pre-rinsed with 10% HCl, whereas those for anion analysis were not acidified. DO and TDS were measured in situ using a WTW multi 3400i, major cations and anions were determined by ICP-AES, NO_2^- and NH_4^+ by molecular spectrophotometry, and HCO_3^- by acid–base titration. Charge-balance errors ranged from –5% to +5%. Nitrate isotopes were analyzed using the bacterial denitrification method, in which NO_3^- was quantitatively converted to N_2O and measured by IRMS. Because NH_4^+ was undetectable in nearly all samples, it was excluded from subsequent hydrochemical analyses.

$$\% \text{CBE} = \frac{\sum \text{cations} - \sum \text{anions}}{\sum \text{cations} + \sum \text{anions}} \times 100\% \quad (3)$$

To ensure analytical accuracy and reproducibility, 20% of randomly selected samples in each batch were analyzed in duplicate. All results met quality-control requirements, with a 100% pass rate. Recoveries based on certified reference materials and blanks ranged from 92% to 108%, within the acceptable range.

3.2. Quantifying nitrate contributions under multi-source mixing

Spearman’s rank correlation analysis revealed systematic relationships between inorganic nitrogen species and major ions (Fig. 2a). NO_3^- was significantly correlated with several major ions,

whereas NO_2^- exhibited strong associations with Cl^- , Na^+ , and K^+ . PCA yielded a Kaiser–Meyer–Olkin (KMO) value of 0.759, and two principal components explained 75.9% of the total variance. PC1 accounted for 50.5% and exhibited high loadings of TDS , SO_4^{2-} , Cl^- , K^+ , Mg^{2+} , Ca^{2+} , HCO_3^- , NO_3^- , and NO_2^- , indicating the combined influence of lithological weathering and anthropogenic inputs [20]. PC2 explained 25.4% of the variance and was dominated by Cl^- , Na^+ , HCO_3^- , and NO_2^- . Given the low likelihood of halite formation under local hydrogeological conditions, this association primarily reflects sewage-derived inputs.

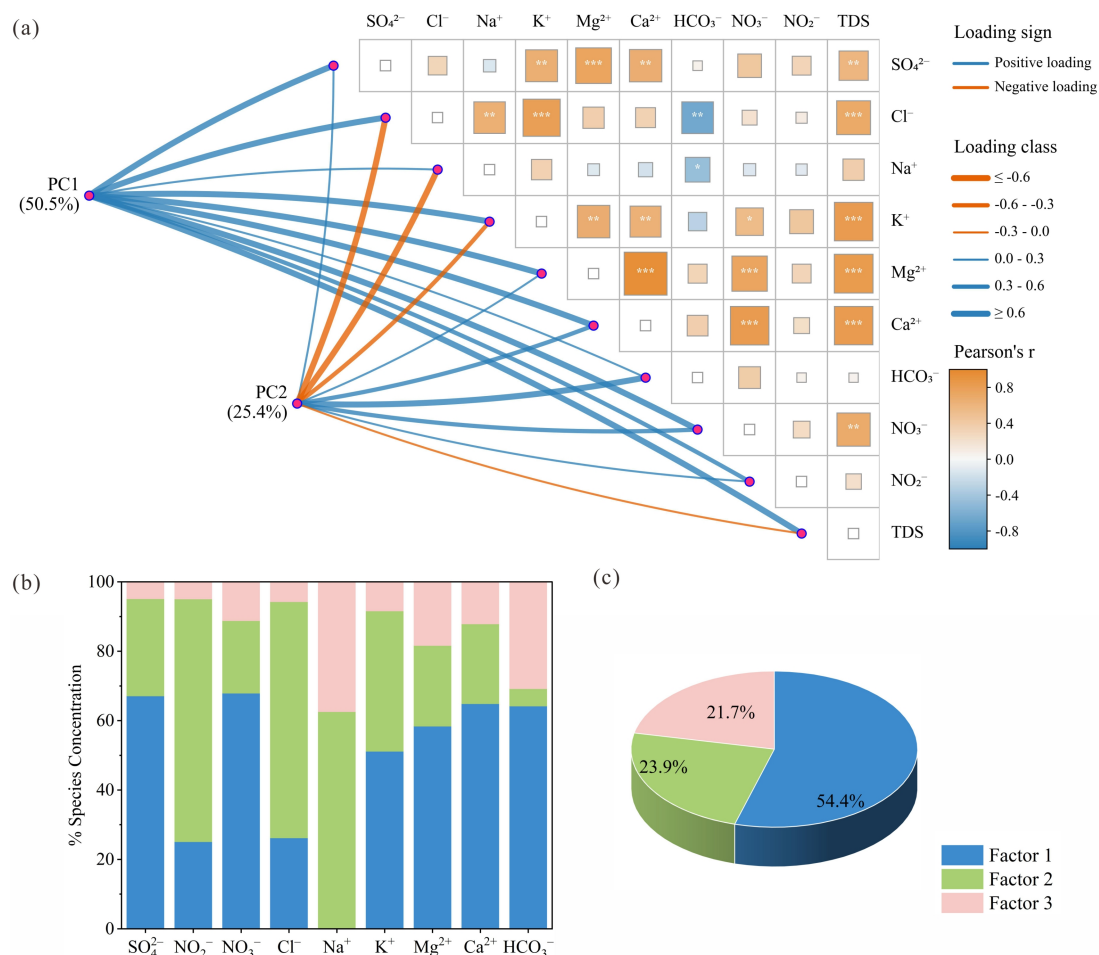


Figure 2. Source apportionment using PCA and PMF. (a) The results of correlation analysis and PCA. (b) The contributions of various factors. (c) The contribution percentages of various sources.

Three factors were identified based on the PMF model (Fig. 2b, c). Factor 1 accounted for 54.4% of the total modeled contribution and made substantial contributions to SO_4^{2-} , NO_3^- , K^+ , Mg^{2+} , Ca^{2+} , and HCO_3^- . The simultaneous contributions to weathering-related ions and NO_3^- indicate a mixed natural–anthropogenic source, reflecting the superposition of mineral dissolution and nutrient inputs from urban and agricultural areas [21]. Factor 2 accounted for 23.9% of the total contribution and was dominated by Na^+ , NO_2^- , and Cl^- . The strong association among these constituents, together with the geological constraints on evaporite formation, identifies sewage discharge as the most plausible source [22]. This factor highlights the importance of wastewater-derived inputs in controlling nitrogen enrichment and Cl^- accumulation. $\delta^{15}\text{N}-\text{NO}_3^-$ and $\delta^{18}\text{O}-\text{NO}_3^-$ also confirmed that the source was sewage. Factor 3 contributed 21.7% of the total modeled contribution and was primarily associated with Na^+ and HCO_3^- . The absence of substantial contributions from Cl^- , SO_4^{2-} , Ca^{2+} , and Mg^{2+} distinguishes this factor from sewage discharge, evaporite dissolution, and carbonate-related

sources [23]. Considering the widespread distribution of silicate rocks, Factor 3 is best attributed to silicate weathering and associated cation-exchange processes.

3.3. From source identification to nitrate-source prediction

This research developed a data-driven nitrate-source classification model using hydrochemical variables as predictor features and source-apportionment results as response labels. The XGBoost algorithm was optimized using the Optuna hyperparameter-optimization framework in conjunction with cross-validation. The optimized XGBoost model achieved an overall classification accuracy of 0.833 (Table 1, Fig. 3). The mineral-dissolution category was identified without classification error, with precision, recall, and F1-score of 1.000. The mixed-source category exhibited perfect recall (1.000) and a high F1-score (0.867), but lower precision (0.765), indicating some false-positive assignments. By contrast, the sewage category showed perfect precision (1.000) but limited recall (0.200), indicating that the model correctly identified the samples assigned to this category but failed to detect a substantial fraction of the actual sewage-related samples.

Feature-importance analysis indicated that K^+ was the most influential predictor, with an importance score of 0.208, followed by Ca^{2+} (0.150), Cl^- (0.123), and SO_4^{2-} (0.116) (Figure 3). These four major-ion variables jointly accounted for 0.597 of the total feature importance, demonstrating the substantial contribution of the overall hydrochemical signature to nitrate-source discrimination. Mg^{2+} , Na^+ , HCO_3^- , NO_2^- , and NO_3^- had importance scores of 0.087, 0.086, 0.080, 0.078, and 0.073, respectively. The relatively modest importance assigned to NO_3^- suggests the model discriminated sources primarily through integrated information from multiple hydrochemical indicators rather than nitrate concentration alone.

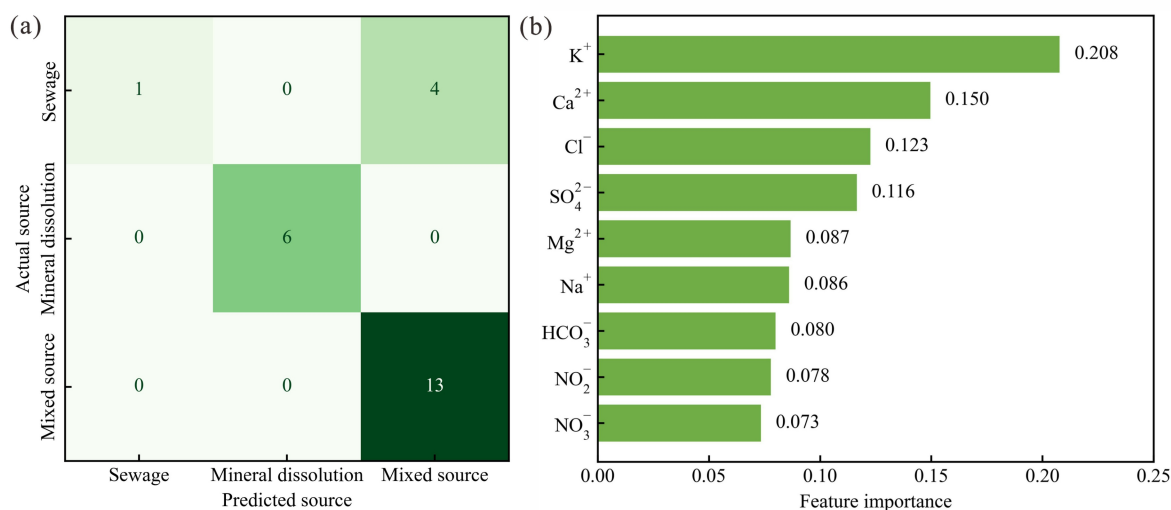


Figure 3. (a) Confusion matrix; (b) Feature importance analysis of XGBoost.

Table 1. Evaluation of XGBoost prediction accuracy for nitrate source prediction.

Source	Precision	Recall	F1-score	Sample count
Sewage	1	0.2	0.333	5
Mineral dissolution	1	1	1	6
Mixed source	0.765	1	0.867	13
Accuracy		0.833		

4. Limitations and prospects

Several limitations should be considered when interpreting the proposed framework. Hydrochemical signatures are influenced by both nitrate inputs and water–rock interactions, which can complicate attributing individual factors to specific pollution sources. PMF-derived contributions characterize the overall hydrochemical mixture and do not necessarily represent nitrate-specific source fractions, while uncertainties in source labels may propagate into machine-learning predictions. The number of samples from the sewage source was limited, which affected predictive performance. Future work should incorporate more representative sewage-affected samples and evaluate approaches for improving class-specific performance.

Overall, this study presents an integrated framework connecting hydrochemical characterization, quantitative apportionment, and data-driven nitrate-source prediction. The application showed the potential of routine hydrochemical measurements to support rapid source screening. With improved source-label validation and broader field evaluation, the framework could inform targeted nitrate monitoring and pollution control at the watershed scale.

Contributions: R. Y. designed the research and wrote the manuscript. S. Y. processed the data and wrote the manuscript. Z. S., L. C., and Q. H. conducted the survey and provided the data. X. T. reviewed the manuscript. All authors reviewed the manuscript.

Funding: This research was funded by the Water Pollution and Technology Foundation Project of Chengdu Municipal Bureau of Ecological Environment (KY2025005). The study was, in part, supported by the Natural Science Foundation of Sichuan Province (2024NSFSC0133).

Conflicts of Interest: The authors declare no conflicts of interest.

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