

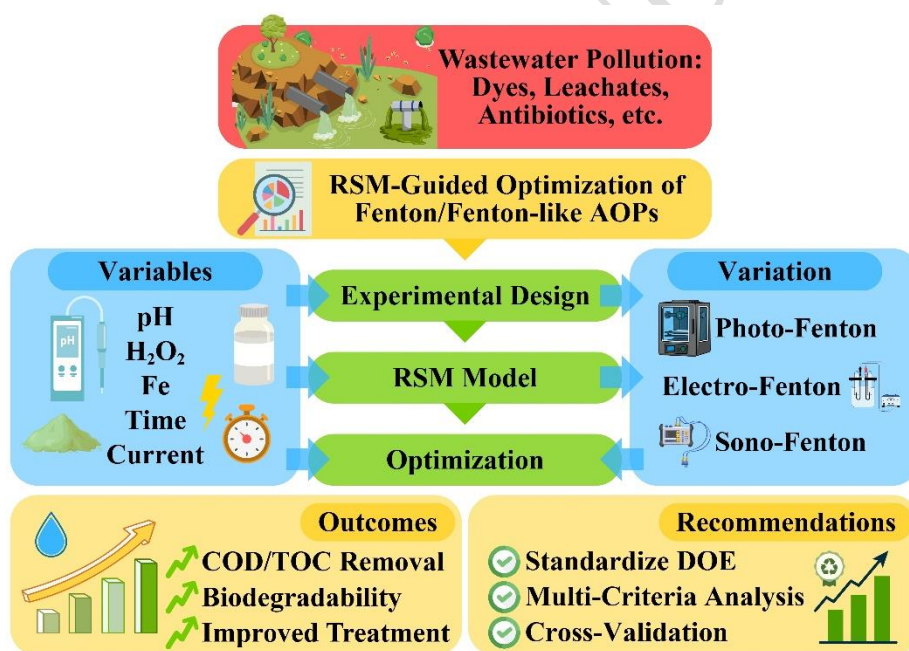
Critical Methodological Review of RSM for Fenton/Fenton-like Process Optimization: SLNA-Based Bibliometric and Systematic Review

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Graphical Abstract

12 **Abstract**

13 This study critically reviews the use of response surface methodology (RSM) and design of
14 experiments (DOE) in optimizing Fenton and Fenton-like processes for wastewater treatment from
15 2015 to 2025. Through systematic literature network analysis (SLNA), bibliometric analysis, and
16 systematic review, 78 articles were assessed to address the following research questions. For RQ1,
17 the current landscape is characterized by low collaboration but high productivity in Turkey, Iran,
18 India, and China, where research has shifted from method development toward pilot-scale
19 implementation. The findings for RQ2 identify three influential thematic groups: methodological
20 aspects, advanced oxidation variations, and performance/economic investigations. The ten most-
21 cited publications focus primarily on methodological and applied aspects. For RQ3, researchers
22 have mainly focused on COD/TOC and color reduction in wastewater. However, several issues
23 remain, including insufficient reporting of DOE methodology, limited model validation, restricted
24 factor ranges, and a lack of multi-objective investigations. As suggested guidelines, future studies
25 should consider mandatory DOE methodology reporting, multivariate optimization, multistage
26 pilot-scale validation, and the use of hybrid RSM–ANN/mechanistic modeling approaches.

27 **Keywords**

28 Advanced Oxidation Processes; Design of Experiments; Wastewater Treatment Performance; Co-
29 authorship Networks; Co-occurrence Analysis; Latent Dirichlet Allocation; Scalability and
30 Robustness

1. Introduction

Urbanization and industrialization accelerate water pollution, thereby hindering sustainable development. The decline in water quality due to various contaminants, coupled with inadequate infrastructure, has resulted in water scarcity and is therefore considered a pressing issue requiring immediate attention from governments (Nazir et al., 2025; Yang et al., 2025). Effective wastewater treatment enables water reclamation, improves water availability, promotes conservation and rational use, and reduces costs while contributing to the achievement of the Sustainable Development Goals (SDGs) (Azupogo et al., 2025; Yang et al., 2025). Among various wastewater treatment methods, such as physical adsorption (Faizal et al., 2024), membrane filtration (Asif et al., 2024), photocatalysis (Chen et al., 2024), and biodegradation (S. Zhou et al., 2024), advanced oxidation processes (AOPs) are gaining popularity because of their exceptional oxidation capacity (Lu et al., 2025). AOPs refer to chemical processes in which reactive radicals are generated to oxidize pollutants present in water (Tchobanoglous et al., 2014). In general, AOPs use radical species to decompose pollutants through oxidation (Hübner et al., 2024). Figure 1 illustrates the classification of AOPs based on previous research (Gonzaga et al., 2023; Lupu et al., 2023).

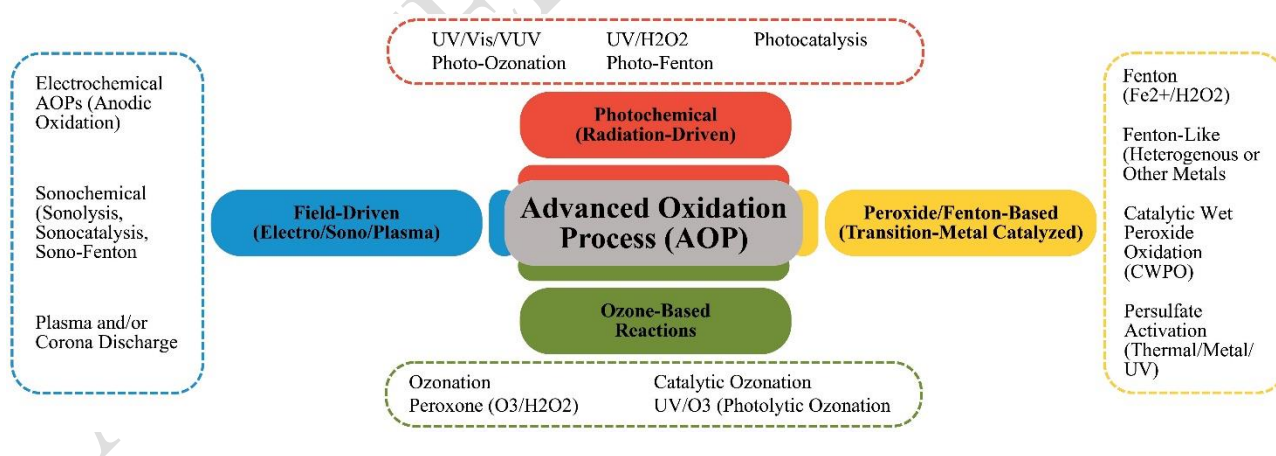


Figure 1 AOP classification (Adapted from Gonzaga et al. (2023) and Lupu et al. (2023), CC BY 4.0. Changes made: categories merged, layout redesigned, and labels updated.)

Examples of AOPs include Fenton oxidation and Fenton-like techniques, in which hydroxyl radicals are generated through the oxidation of peroxides with transition metal catalysts to degrade organic compounds (Miklos et al., 2018; Ribeiro et al., 2024). Fenton-like AOPs simulate traditional AOPs

by combining photo-Fenton, ozonation, UV, photochemical reactions, and electro-Fenton oxidation (Alturki et al., 2024). These methods involve multiple oxidants and metals, resulting in increased chemical and metal-ion concentrations in solution, in addition to the production of secondary by-products such as metal-ion sludge (Bello et al., 2019; Ribeiro et al., 2024; M. Zhang et al., 2019). The use of statistical optimization techniques enables the adjustment of dosages and resource distribution. One such technique is response surface methodology (RSM), which is characterized by high precision, reduced experimentation, and lower costs because of its quadratic nature (Alturki et al., 2024; Veza et al., 2023). RSM, as a design of experiments (DOE) technique, involves factorial, central composite, and Box–Behnken designs (Ragunath et al., 2017).

Table 1 Comparison of Previous Review Studies

Aspect/Context	Previous Reviews	This Study (Novelty)
Main focus	Emphasize energy indicators, lamp types, EEO-TRL, and AOP process efficiency (Hübner et al., 2024; Miklos et al., 2018).	Integrates bibliometrics with RSM/DOE methodological analysis for Fenton processes, highlighting statistical validation, AI/ML, and practical aspects.
Scope of Fenton reviews	Discuss trends in solar/photo-Fenton, heterogeneous catalysts, and electro-Fenton with a focus on H ₂ O ₂ formation and operational limitations (Pimentel Prates et al., 2023; M. Zhang et al., 2019).	Provides a comprehensive framework linking experimental design, variable interactions, validation, and multi-objective recommendations.
Bibliometric perspective	Highlights publication trends, collaborations, and research themes (Brdarić et al., 2024).	Combines bibliometrics with methodological evaluation and produces a practical checklist for future research.
Optimization approach	Generally focuses on single objectives, such as energy efficiency or removal performance, or on external aspects of AOPs (Nazlabadi et al., 2021; Reza et al., 2024).	Emphasizes multiple objectives, such as pollutant removal, energy aspects, reagents, sludge, toxicity, economic perspectives, and step-by-step validation.
Contribution style	Provides technology trends, indicators, and frameworks (Hübner et al., 2024; Miklos et al., 2018).	Offers a compact checklist and comparison table as an operational guide for future RSM–Fenton research.

Recent reviews of AOPs have emphasized energy efficiency indicators, such as electrical energy order (EEO), lamp type, treatment volume, and detoxification reagents, while providing a framework for scalability and technology readiness level (TRL) (Hübner et al., 2024; Miklos et al., 2018). Other reviews of Fenton reactions, including solar Fenton/photo-Fenton and heterogeneous

66 catalysts, have highlighted operational trends and limitations (Pimentel Prates et al., 2023; M.
67 Zhang et al., 2019). Bibliometric analyses have mapped publication and collaboration trends
68 (Brdarić et al., 2024), whereas systematic reviews have examined the application of DOE and RSM
69 in oxidation and related treatment processes (Nazlabadi et al., 2021; Reza et al., 2024). Collectively,
70 these studies provide valuable insights but remain focused on mechanistic aspects, energy indices,
71 or external factors. In contrast, this review integrates bibliometric mapping with methodological
72 evaluation of DOE and RSM in Fenton systems, emphasizing experimental design choices, factorial
73 ranges, statistical diagnostics, and cross-validation. By explicitly addressing the lack of systematic
74 evaluation of DOE/RSM practices, statistical validation, and integration with emerging artificial
75 intelligence (AI) and machine learning (ML) approaches in Fenton-based wastewater treatment, this
76 review highlights the scientific gap overlooked by previous studies. Unlike earlier reviews that
77 mainly compared pollutant removal efficiencies, our analysis links these methodological
78 shortcomings to the broader challenges of reproducibility, scalability, and sustainability. To provide
79 a comprehensive review, this study summarizes recent findings through a critical methodological
80 review and applies bibliometric analysis to examine research trends, key articles, and major
81 research gaps, followed by further discussion through a systematic literature review.

2. Methodology

2.1. Study Design and Corpus

In this study, systematic literature network analysis (SLNA) was used to examine trends and gaps in the statistical analysis of RSM for wastewater purification using the Fenton method. SLNA combines systematic literature review (SLR) and bibliometric analysis (BA). In this study, SLNA was used as the basis for a further critical methodological review (CMR) (Ikhlas & Ramadan, 2024; Wibowo et al., 2024). The research methodology followed the Preferred Reporting Items for Systematic Reviews and Meta-Analyses (PRISMA) 2020 framework and is presented in Figure 2.

The methodological process involved several steps, the first of which was formulating research questions (RQs) to guide the analysis. These RQs are presented below.

- RQ1: What does the research landscape on RSM for Fenton-type optimization look like between 2015 and 2025? This includes publication trends, collaborations among authors and countries, variations in processes, and a preliminary thematic map.
- RQ2: Which themes or groups have had the greatest impact, and how are they expressed through the most influential articles?
- RQ3: How do these studies apply RSM/DOE, what performance outcomes are prioritized, what methodological or reporting gaps are identified, and what future research agenda is suggested?

The next step involved identifying metadata from scientific databases in accordance with the PRISMA 2020 recommendations (Page et al., 2021). A separate subsection details the criteria for selecting, evaluating, identifying, and including studies. To answer RQ1, content analysis and identification were conducted using BA, whereas RQ2 and RQ3 were addressed through a literature review based on a selected set of references examined using SLR. Further CMR analysis was also applied to gain a deeper understanding of the methodological aspects of DOE/RSM assumptions, limitations, and modeling quality.

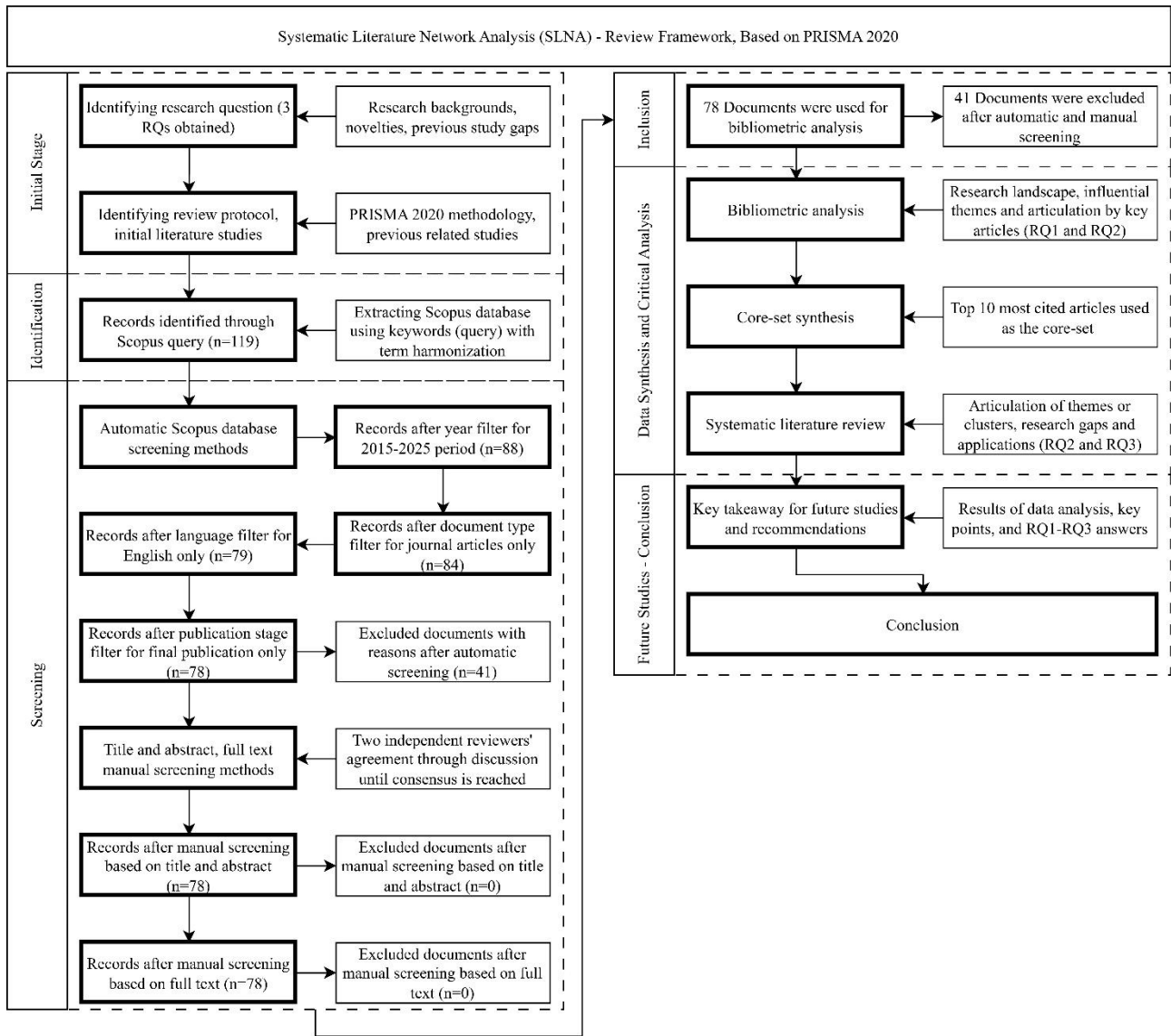


Figure 2 Methodological Framework Based on PRISMA 2020

2.2. Validity, Reproducibility, and Data Collection

The dataset was validated for the period 2015–2025 using Elsevier’s Scopus database. To ensure reproducibility, the software versions used in this process were specified: VOSviewer 1.6.20 and Orange Data Mining 3.40.0, which were used to generate and analyze the bibliometric networks. Details of data collection are briefly discussed below to support research replication. Data extraction was conducted through the Scopus database by entering several keywords into the Scopus search fields (Ikhlas & Ramadan, 2024). Scopus served as a single database for several reasons: it offers broader title coverage across disciplines and document types and provides consistent, easily exportable metadata for bibliometric analysis and data processing, thereby

118 facilitating the reproducibility of the applied bibliometric workflow (Singh et al., 2021).
119 Furthermore, comparative literature indicates that coverage differences between Scopus and other
120 databases tend to be complementary and can be mitigated through term harmonization,
121 deduplication, and full transparency of queries and article lists attached in the [Supplementary](#)
122 [Material](#) (Baas et al., 2020; Pranckutė, 2021). Therefore, the use of Scopus-only approach was
123 chosen to maintain methodological consistency and repeatability of the analysis, while all complete
124 queries, export files, and processing steps are available in the [Supplementary Material](#) to allow
125 replication or cross-checking by readers and researchers (Baas et al., 2020). Using only the Scopus
126 database for review articles is a justifiable and accepted practice, as evidenced by published
127 systematic reviews and literature reviews that rely solely on Scopus for their data sources. The
128 database's comprehensive coverage, quality assurance, and recognition in academia support its use
129 as a standalone resource for literature reviews (Alam et al., 2023; Baas et al., 2020; Rostamzadeh et
130 al., 2026). A Scopus search was performed using a predefined query (see [Supplementary Material](#))
131 in January 2026. The identification and screening process followed the PRISMA 2020 guidelines,
132 with the number of records and reasons for exclusion presented in Figure 2. Title–abstract and full-
133 text screening were performed by two independent reviewers, and no discrepancies were found; all
134 articles meeting the inclusion criteria were selected for bibliometric analysis.

135 **2.3. Scientific Mapping and Thematic Modeling**

136 VOSviewer software was used for mapping, with attention given to authorship networks based on
137 the names and countries of authors, co-occurrence networks derived from keywords and terms
138 found in the titles and abstracts of papers, and citation analysis. Orange Data Mining's text-mining
139 module was used to analyze keywords, titles, and abstracts (Ikhlas & Ramadan, 2024; Wibowo et
140 al., 2024). By combining the findings obtained from these two software tools, researchers can
141 obtain a more complete picture of the issues addressed in this study, identify knowledge gaps, and
142 propose a critical methodological analysis based on the results. Preprocessing is a crucial step in
143 bibliometric and topic-modeling workflows because it standardizes raw data, removes noise, and

ensures reproducibility, thereby increasing the reliability of co-occurrence and clustering analyses while providing a transparent basis for validating LDA topic solutions. Recent methodological studies have emphasized that preprocessing strategies directly affect perplexity scores, topic interpretation, and the robustness of research findings (Nowakowska, 2025). Data preprocessing also enabled the creation of maps, word clouds, and topics. The detailed initial data preprocessing steps are shown in [Supplementary Table 2](#).

2.3.1. VOS Analysis Technique

Current research trends and their status were described and elaborated based on the collected metadata. Several analyses were then performed, including co-authorship networks based on author names and countries, citation networks, and co-occurrence analysis based on keywords, titles, and abstracts. In this study, the visualization of similarities (VOS) technique was used because it is considered superior to other distance-based mapping methods for visualizing metadata. The VOS technique, as implemented in VOSviewer, allows relationships between nodes to be represented based on their proximity. The closer the nodes are, the stronger the relationship between the elements. Therefore, in the VOS clustering map, nodes or keywords are arranged to minimize overlap (Van Eck & Waltman, 2010). Other categories of co-authorship, citation networks, and co-occurrence are presented in Figure 3.

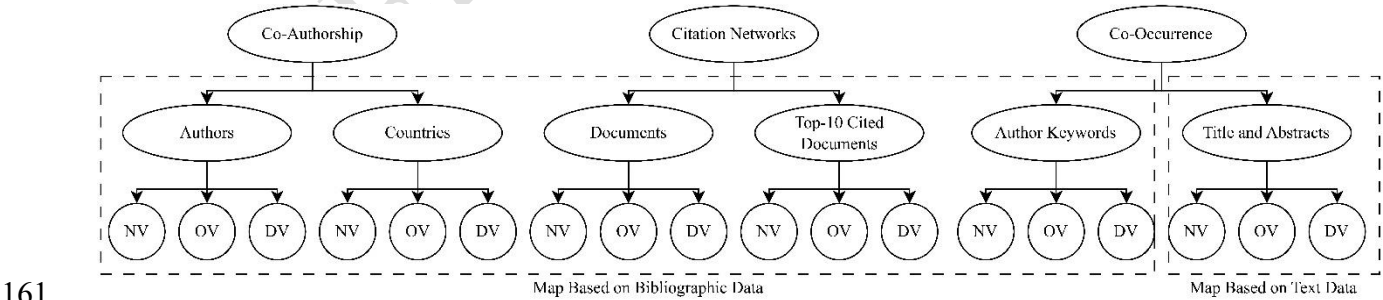


Figure 3 VOSviewer Analysis Framework

Figure 3 shows three VOS visualization modes: network visualization (NV), overlay visualization (OV), and density visualization (DV). NV applies the VOS algorithm layout to construct node and edge graphs for network analysis and clustering. OV applies the VOS layout and colors nodes based

on various attributes, such as average publication year, to indicate changes over time. DV uses colors to indicate density: blue indicates low density, green indicates medium density, and yellow indicates high density or hotspots (Van Eck & Waltman, 2010). In a VOS map, line size indicates the strength of connections between nodes; thicker lines and larger nodes indicate stronger connections (Sawassi & Khadra, 2021). Statistics related to the VOS map can be found in previous studies (Van Eck & Waltman, 2010). Authorship, country-level co-authorship, and paper-based citation networks focusing on the ten most-cited articles were used as VOSviewer analysis units, as shown in Figure 3. NV, OV, and DV were used to show the structure, dynamics, and main research flows. Regarding SLR, primary attention was given to the ten most-cited papers to identify the methodology, performance indicators, and significance of the research. This study aims to provide an overview of highly cited articles on optimization using RSM and Fenton processes and to assess their suitability for SLR. The connections between keywords and terms in the titles and abstracts were analyzed to identify research gaps and trends related to RSM and Fenton applications. Author keyword analysis and term analysis were performed on the titles and abstracts (Kholidah et al., 2022).

2.3.2. Word Cloud Analysis Technique and Thematic Modeling

To analyze the dataset, various methods available in Orange Data Mining, including word clouds, topic modeling, and multidimensional scaling (MDS), were used. In word clouds, word prominence is represented by font size, which depends on word frequency (Ikhlas & Ramadan, 2024). Therefore, large and centrally located words appear more frequently, whereas small and peripheral words appear less frequently. For theme extraction, Latent Dirichlet Allocation (LDA) was performed on the titles, abstracts, and keywords related to RSM optimization and Fenton processing. Because LDA is a natural language processing (NLP) technique, it enables data analysis through thematic modeling (Ikhlas & Ramadan, 2024; Li et al., 2021). Figure 4 shows the workflow for generating word clouds, box plots, bar charts, and scatter plots using Orange Data Mining software.

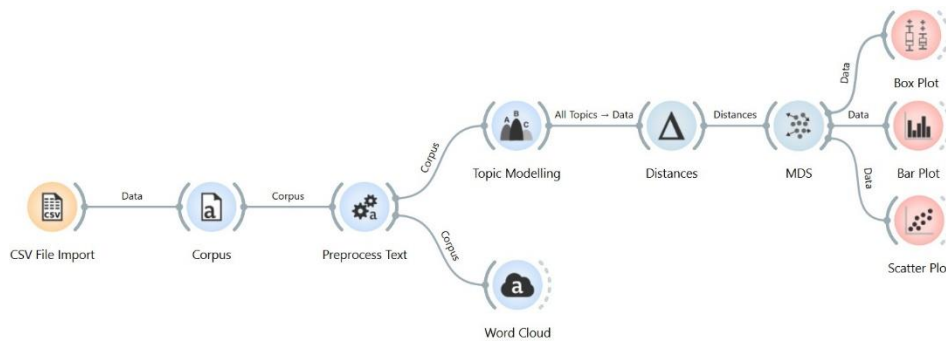


Figure 4 Orange Data Mining Software Workflow

The LDA model identifies keywords based on the co-occurrence of terms to generate thematic categories from groups of words in each article (Ikhlas & Ramadan, 2024; Li et al., 2021). Compared with VOS clustering, in which keywords are closely associated with topics, LDA keywords may not always directly correspond to themes. Therefore, latent topics may be identified using LDA but not through VOS analysis. Thematic modeling was verified using MDS and marginal probabilities derived from topic analysis. MDS determines the similarity of themes by measuring their distance: a shorter distance indicates higher similarity, whereas a longer distance indicates lower similarity. This enables data simplification without losing essential meaning (Yao et al., 2022). Thematic similarity can be converted into a two-dimensional plot while preserving the distance between documents and enabling examination of LDA theme separation. Projections were performed using stress analysis (Kruskal, 1964). For LDA, the distance between vectors with theme probabilities was calculated using the cosine distance metric; subsequently, MDS provided the coordinates for the scatter plot. The color and size of the dots correspond to the marginal probabilities of the themes, while the labels indicate the analyzed themes and demonstrate LDA theme separation. Model fit was assessed using Kruskal's stress (KS): stress = 0 indicates a perfect fit, $\text{stress} \leq 0.05$ indicates a good fit, $0.05 \leq \text{stress} \leq 0.10$ indicates an acceptable fit, and $\text{stress} > 0.15$ indicates a poor fit (Mair et al., 2016). The marginal probability distribution of themes is represented using a bar chart, which shows the extent to which certain themes dominate (Abayomi-Alli et al., 2022). Differences and extreme values across themes are shown using box plots (Lucon, 2024).

2.4.Synthesis of the Core Set

The ten most-cited articles were selected as the core corpus to answer the content-level research questions because citation impact is a pragmatic proxy for methodological influence and community acceptance in a research field (Pech & Delgado, 2021). Highly cited works often reflect methodological innovations, validated protocols, or widely adopted experimental designs; thus, concentrating on the most highly cited set allows for a focused and highly weighted review of established approaches that have shaped the RSM–Fenton literature. To mitigate the limitations of sampling based solely on citations, this study complements citation counts with additional selection criteria, such as recency, methodological relevance, and representation across bibliometric clusters, following recommended bibliometric design principles that advocate multi-criteria selection to balance objectivity and content validity (Öztürk et al., 2024). This combined strategy reduces subjective bias while maintaining analytical feasibility for in-depth SLR. Qualitative synthesis of the core corpus integrates SLR techniques with machine learning-assisted content analysis to address predefined RQs and conduct a targeted methodological assessment of DOE/RSM assumptions, validation practices, scalability, and robustness (Paul & Criado, 2020). Therefore, the resulting SLR synthesizes key concepts, trends, and methodological limitations from reference studies, providing a reliable and reproducible basis for content-level conclusions and future research directions. A formal quality assessment procedure was applied to the ten core set articles. Four domains were assessed, each scored from 0 to 6: experimental design, model validation, statistical robustness, and scale-up potential. A score of 0 indicates no or inadequate reporting, whereas a score of 6 indicates adequate or robust reporting and practices. The total score ranged from 0 to 24 and was categorized as Low (0–8), Moderate (9–16), or High (17–24). Assessments were conducted by two independent reviewers, with discrepancies resolved through discussion until consensus was reached. Complete results, including scores per domain, totals, and brief comments for each study, are available in [Supplementary Table 6](#).

239 **3. Results and Discussion**

240 **3.1. Research Landscape**

241 This study describes the application of the RSM–Fenton approach, as well as related approaches,
242 through the identification of collaboration networks and their integration into a terminology map.
243 The research methodology follows a macro–micro–thematic approach, which includes tracing
244 collaboration patterns, research activities, and terminology development.



245
246 *Figure 5 Overlay Visualization of Co-authorship Based on Country*

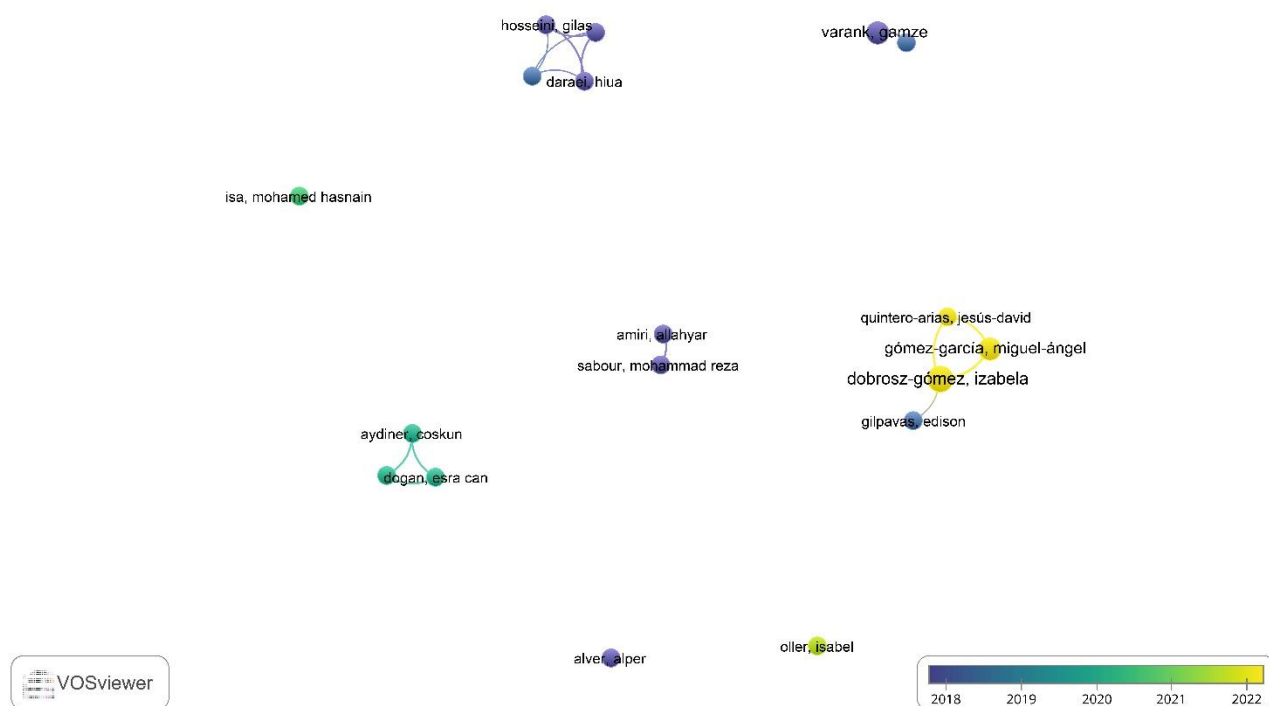


Figure 6 Overlay Visualization of Co-authorship Based on Author

The co-authorship and country maps show broad collaboration trends distributed across various regions but clearly concentrated along the Turkey–Iran–India axis. In this visualization, India serves as a hub connecting the Turkey/Iran region to China and extending its collaborative outposts toward Chile. The secondary center in this network is occupied by Brunei Darussalam. The United States, Mexico, and Pakistan show lower connectivity with the main network. The co-authorship network forms several clusters encompassing closely connected communities of authors, namely a three-author group of Turkish and Iranian authors, a unified group of Spanish-speaking authors, and several isolated authors. This distribution indicates a low level of integration among various co-author groups and suggests that certain authors tend to remain isolated in collaborative contexts. Regarding chronological overlap, previous research tended to concentrate on Turkey and Iran. However, over time, network nodes have shifted toward India and China, with recent activity observed in Chile. Thus, the collaboration process appears geographically concentrated, which may reduce representativeness and limit the possibility of cross-validation of results in the context of the RSM–Fenton model.

263 The observed Turkey–Iran–India axis and authorship groups indicate that the use of locally
264 representative training sets is a significant limitation in the application of the RSM–Fenton
265 methodology. This is evident from the list of the ten most-cited works, as regionally limited data
266 and authorship may lead to representativeness bias when working with location-specific substrates,
267 reagents, or procedures (GilPavas et al., 2019; Sabour & Amiri, 2017; Schenone et al., 2015). The
268 intermediate role of India and the increased activity in China were associated with electrochemical
269 studies at the pilot scale, which showed the effect of operational limitations on optimal laboratory
270 conditions. This suggests that single-site laboratory studies may not be generalizable until they are
271 tested independently (Salmerón et al., 2019; Toor et al., 2021; X. Zhou et al., 2020). Comparisons
272 between RSM and artificial neural networks (ANNs), as well as hybrid models, show that greater
273 methodological robustness can be achieved by applying cross-validation, external validation
274 datasets, or hybrid modeling approaches. However, these approaches remain unevenly distributed
275 geographically and across research communities (Baştürk & Alver, 2019; Sabour & Amiri, 2017).
276 The group of Spanish-language articles, for example, represents a relevant but relatively isolated
277 case study. Therefore, future meta-analytic studies should strive to report results uniformly
278 (GilPavas et al., 2019; Hassanshahi & Karimi-Jashni, 2018). To mitigate potential bias and ensure
279 efficient scale-up, multi-site experimental trials should be conducted across major national centers,
280 with open reporting of design matrices and raw responses, external or pilot-scale validation, and
281 uncertainty analysis. These measures have already been implemented in some cases (Guvenc et al.,
282 2017; Salmerón et al., 2019; H. Zhang et al., 2017; X. Zhou et al., 2020). Such steps will enhance
283 reproducibility, enable robust techno-economic comparisons, and facilitate equitable technology
284 transfer across regions (Schenone et al., 2015; Toor et al., 2021). Nevertheless, contradictions
285 emerge when comparing citation impact with co-authorship centrality, where several of the most
286 influential papers in the core set were not produced by authors or countries identified as top nodes
287 in the VOSviewer map, indicating that methodological influence can arise outside the dominant
288 collaboration clusters.

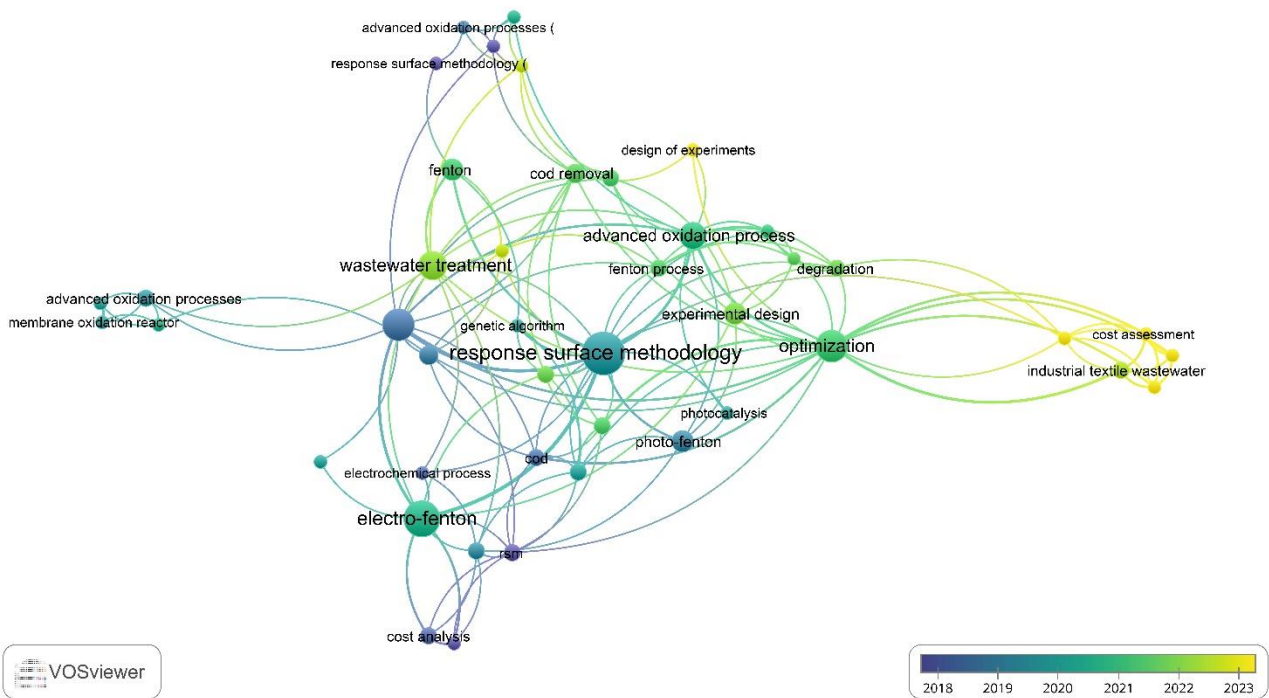


Figure 7 Overlay Visualization of Co-occurrence Based on Author Keywords

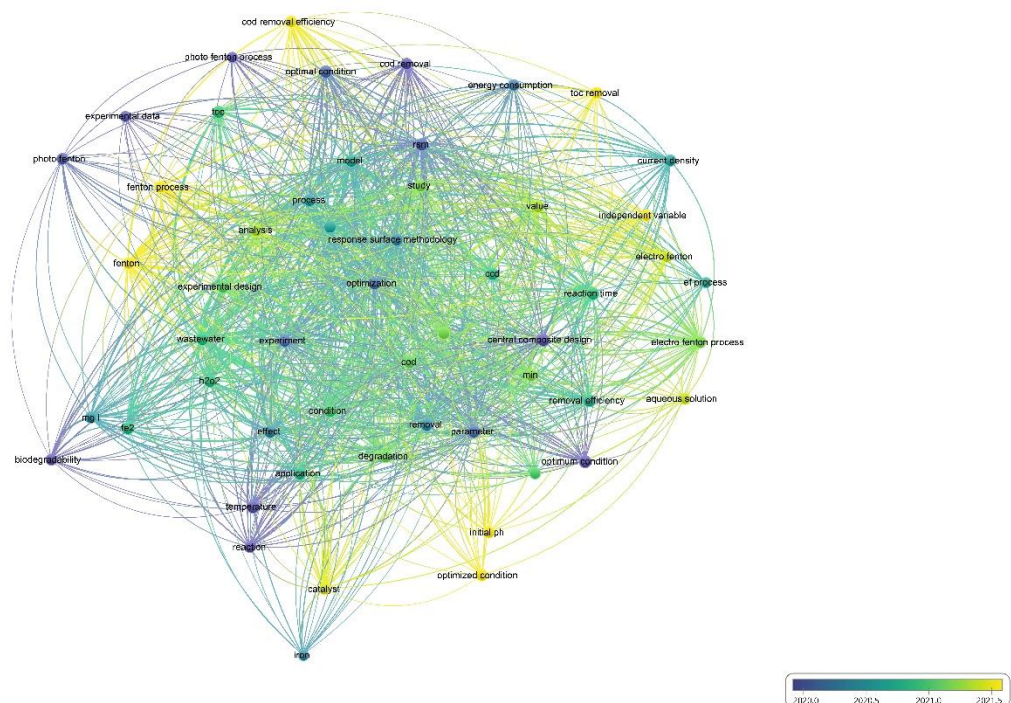


Figure 8 Overlay Visualization of Co-occurrence Based on Title and Abstract

294 The co-occurrence map highlights the centrality of RSM in the network, representing the central
295 node that connects three interrelated clusters. The first thematic cluster relates to experimentation
296 and optimization, in which methods such as central composite design (CCD) and DOE play
297 important roles. The second theme covers various AOP technologies, including Fenton, photo-
298 Fenton, electro-Fenton, and electrochemical methods, all of which are assessed based on metrics
299 such as COD/TOC removal efficiency and energy costs. The third theme addresses application and
300 economic topics, including industrial textile wastewater and cost evaluation. The temporal layer
301 shows a distinct trend, beginning with an initial emphasis on method and process development,
302 followed by optimization efforts using RSM and CCD. Recent years have shown a shift toward
303 process application and cost-performance analysis. Thus, as indicated by the connections among the
304 identified keywords, the research agenda is shifting from the development and comparison of
305 different process variants toward optimization schemes that consider multifactorial processing
306 criteria in terms of performance, energy efficiency, and economic efficiency.



307

308

Figure 9 Author Keywords Word Cloud

325 and R^2 , reveal the authors' interest in multifactor optimization, feasibility studies, and response
326 surface fitting. In contrast, response-related keywords, such as COD/TOC removal, color, and
327 biodegradability, together with common applications such as textiles, leachate, and sludge, indicate
328 the direction of applied investigations.

329 Considering the convergence of the co-occurrence and word-cloud results, recurring
330 methodological patterns are evident. Many RSM (CCD/BBD) studies report laboratory-scale
331 optimization but neglect full regression diagnostics, such as lack-of-fit and residual analysis; use a
332 narrow range of factors with sparse sampling in high-curvature regions; and focus on optimizing a
333 single metric, such as COD/TOC, without accounting for reagent or energy use. These limitations
334 increase the risk of overfitting and limit extrapolation (Baştürk & Alver, 2019; Sabour & Amiri,
335 2017; Salmerón et al., 2019; Schenone et al., 2015; Toor et al., 2021; X. Zhou et al., 2020).
336 Comparison with ML approaches shows that quadratic RSM can miss nonlinear dynamics unless
337 complemented by cross-validation or independent validation (Baştürk & Alver, 2019; Sabour &
338 Amiri, 2017). Therefore, this study recommends reporting complete design matrices and response
339 values, conducting cross-validation or pilot/independent-scale tests, quantifying uncertainty through
340 confidence intervals for optimal predictions, increasing sampling density in curvilinear regions, and,
341 where appropriate, adopting multi-objective or hybrid RSM–ANN/kinetic strategies to improve
342 predictive robustness and support scale-up (GilPavas et al., 2019; Guvenc et al., 2017; Hassanshahi
343 & Karimi-Jashni, 2018; H. Zhang et al., 2017). Nevertheless, contradictions appear across the core
344 set, while studies such as H. Zhang et al. (2017) and Salmerón et al. (2019) reported consistent
345 convergence of optimized operating conditions, others like X. Zhou et al. (2020) and GilPavas et al.
346 (2019) highlighted divergent outcomes under similar factor ranges, raising concerns about
347 reproducibility. In terms of DOE designs, CCD and BBD approaches applied in Toor et al. (2021)
348 and Schenone et al. (2015) proved efficient for factor screening, yet their limited resolution in
349 highly non-linear domains was evident in Baştürk & Alver (2019). These cases illustrate situations
350 where RSM may be inappropriate, as polynomial models fail to capture complex interactions.

Accordingly, several influential papers (Baştürk & Alver, 2019; Sabour & Amiri, 2017) contrasted RSM with machine learning methods such as ANN, showing improved predictive accuracy and multi-objective optimization, albeit requiring larger datasets and facing risks of overfitting.

3.2. Influential Themes and Structure of Key Articles

To identify important themes or phrases among the articles selected in this study, a thematic distribution technique was used. This technique automatically identifies themes in a text corpus based on the presence of co-occurring words. It exploits the frequent co-occurrence of specific words in the text and defines them as themes. In this case, the LDA algorithm was used to identify themes, with four themes considered for the author keywords, titles, and abstracts, as shown in Table 2.

Table 2 Terms Generated Through Topic Modeling

Topic	Terms in Author Keywords	Topic	Terms in Title and Abstract
Topic 1	Fenton, surface, response, methodology, design, oxidation, wastewater, electro, advanced, process	Topic 1	EF, process, COD, removal, L, electro, H ₂ O ₂ , using, treatment, pH
Topic 2	Optimization, response, surface, advanced, methodology, wastewater, treatment, oxidation, process, industrial	Topic 2	Catalyst, treatment, iron, pH, COD, removal, oxidation, Fe, time, H ₂ O ₂
Topic 3	Fenton, wastewater, process, treatment, removal, experimental, design, dye, optimization, industrial	Topic 3	Process, removal, using, design, COD, conditions, optimization, treatment, wastewater, reaction
Topic 4	Design, Fenton, process, central, composite, oxidation, degradation, electro, advanced, optimization	Topic 4	Treatment, wastewater, L, COD, process, oxidation, mg, photo, industrial, conditions

The KS value, presented in the MDS widget, is an indicator of how well the original distances can be represented in a two-dimensional map, with smaller values indicating better representation. It is generally accepted that a KS score of 0.10 or lower is acceptable, while a score below 0.05 is considered highly acceptable. In the current analysis, KS scores of 0.042 for author keywords and 0.073 for titles and abstracts indicate the reliability of these mappings. However, thematic representation based on author keywords was more consistent and accurate than that based on title–abstract pairs. Thus, the use of thematic modeling to explore theme distribution is relevant.

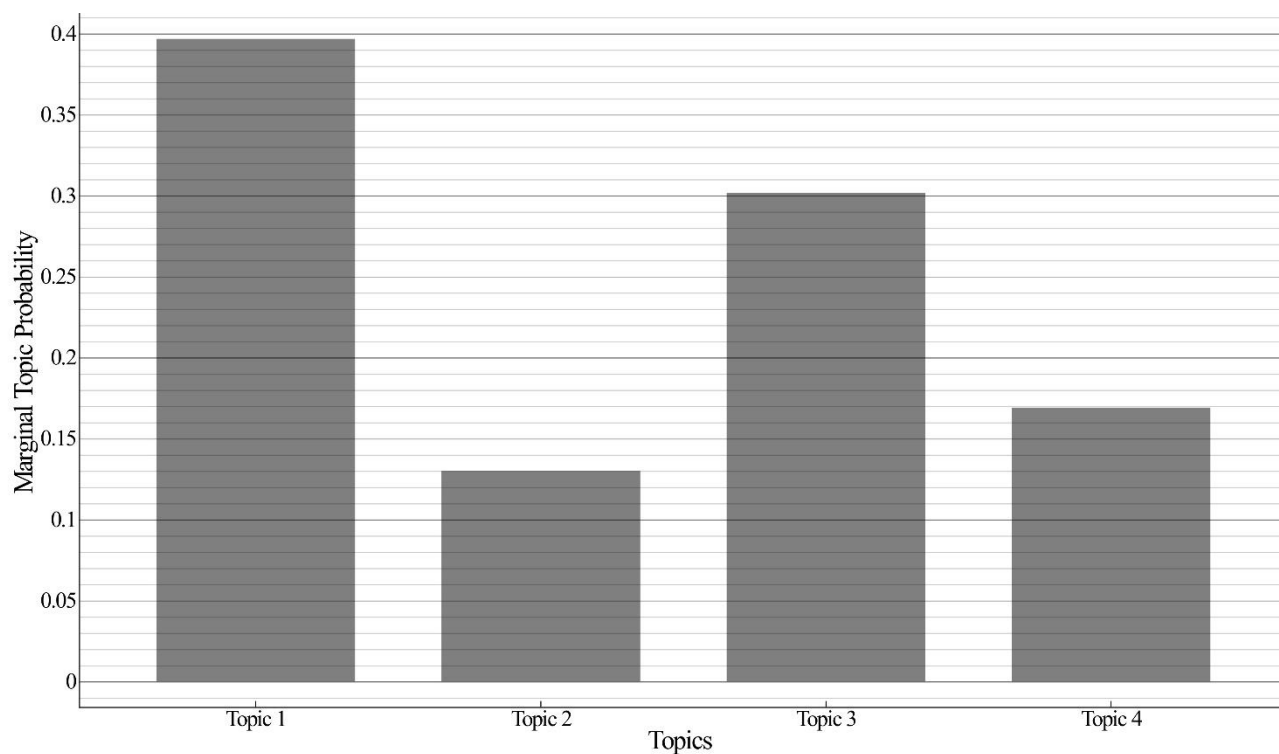


Figure 11 Bar Plot for Topic Modeling Generated from Author Keywords

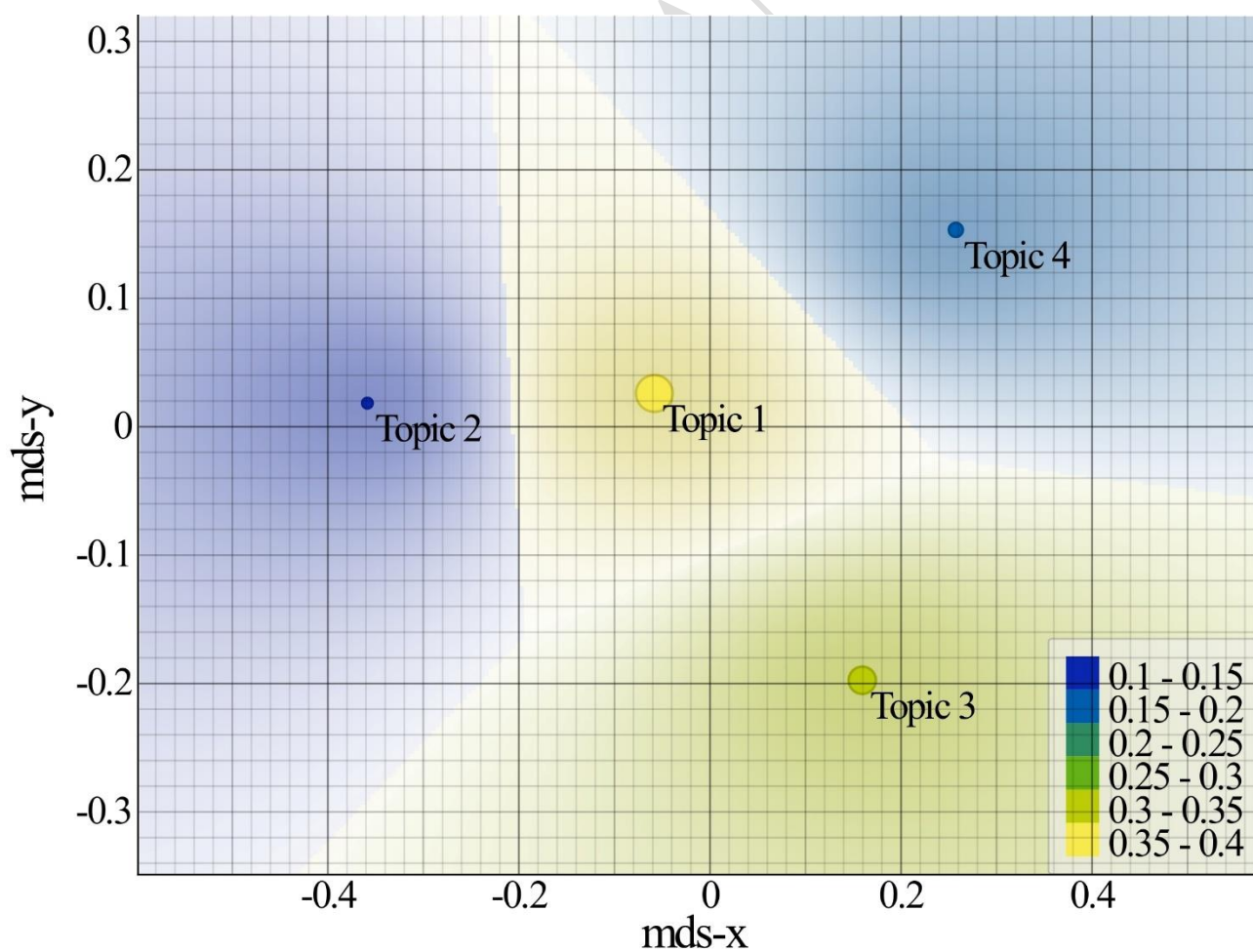


Figure 12 Scatter Plot for Topic Modeling Generated from Author Keywords

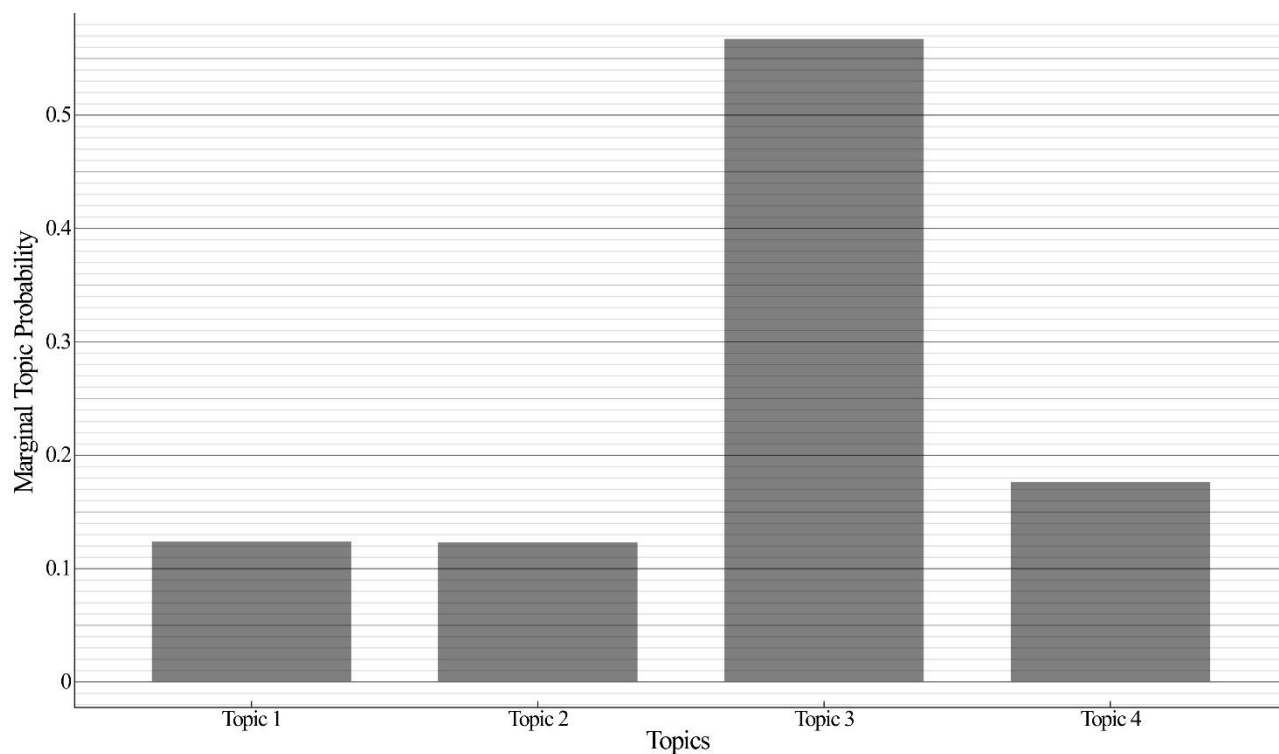


Figure 13 Bar Plot for Topic Modeling Generated from Titles and Abstracts

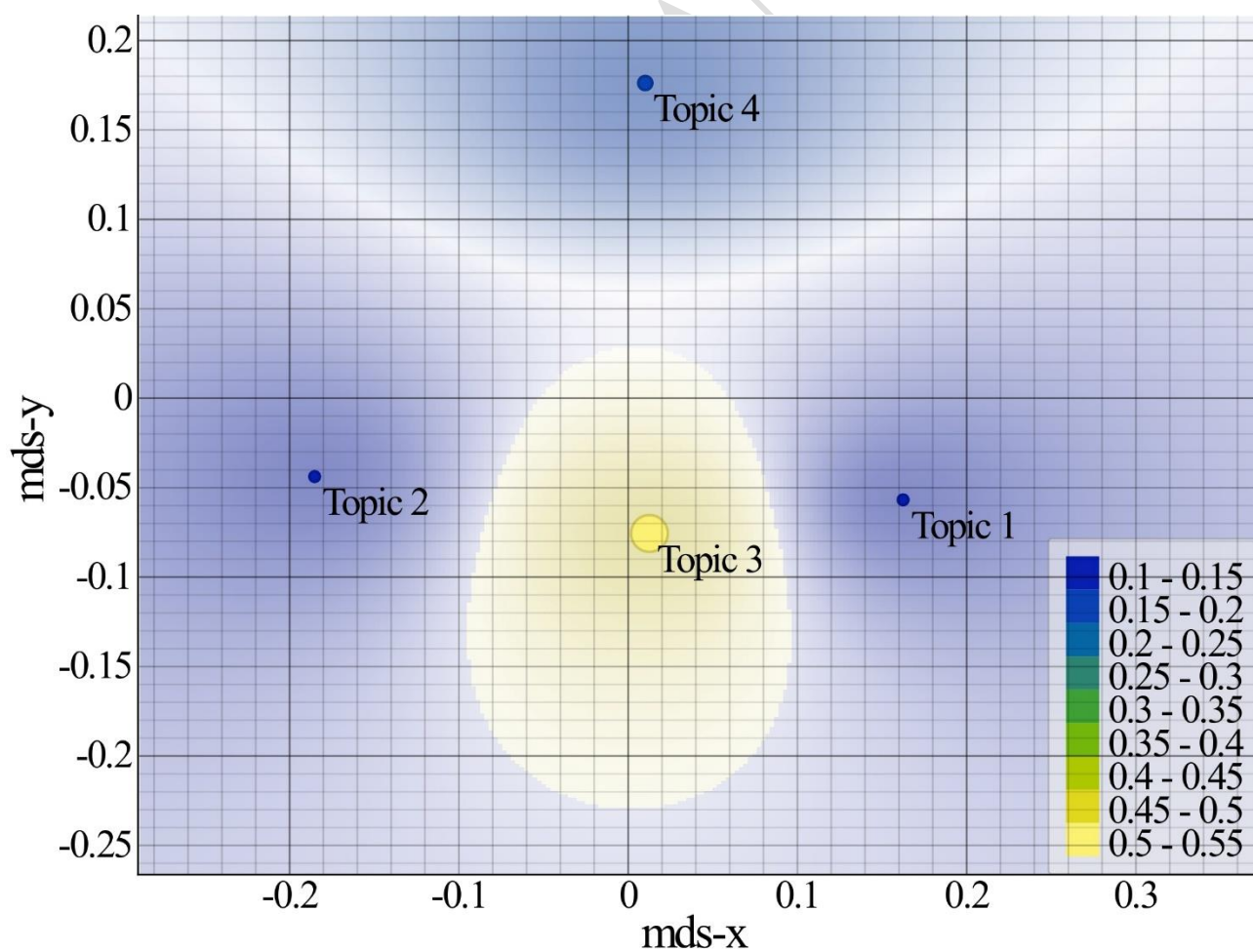
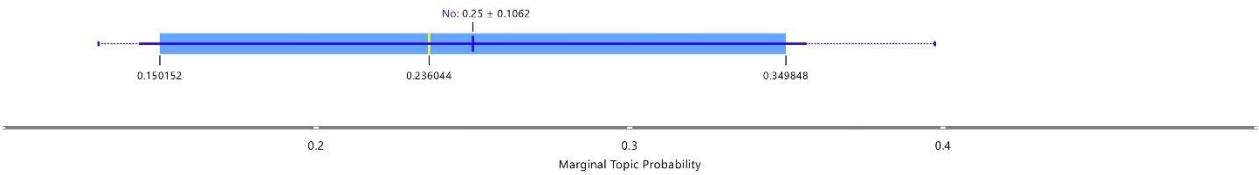


Figure 14 Scatter Plot for Topic Modeling Generated from Titles and Abstracts

377 Based on the bar plot and scatter plot for topic modeling generated from author keywords, there are
378 two main peaks at approximately 0.39 and 0.30, while the other topics are slightly less prominent.
379 These two topics reflect the use of RSM/DOE methods in optimizing wastewater treatment
380 solutions and processes, with emphasis on treatment and removal performance. The other, less
381 prominent peaks, with values of approximately 0.17 and 0.13, provide additional information
382 regarding design approaches and process types. Data collection relied on keywords to achieve a
383 balanced representation of “RSM applications” and “treatment performance application objectives.”
384 In relation to RQ2, the two thematic dimensions had an equally strong impact. Four thematic axes
385 form a rectangle, where Theme 1, located at the center, represents the methodological dimension of
386 RSM/DOE and connects Theme 3, which represents process applications; Theme 4, which
387 represents design architecture and process options such as CCD and electro-Fenton; and Theme 2,
388 which represents optimization. This wide distance emphasizes that these topics are orthogonal,
389 meaning that they are relatively independent within the discourse. By assessing the most influential
390 topics, future studies may identify representative literature for each branch based on Theme 1.

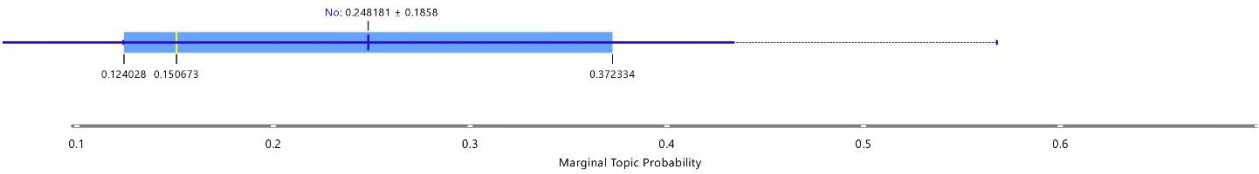
391 Meanwhile, based on the bar chart and scatter plot for topic modeling generated from titles and
392 abstracts, one theme accounts for more than half of the points analyzed, while the other themes
393 account for much smaller proportions. Many papers follow a thematic order, beginning with RSM
394 and DOE, followed by optimization of conditions, and ending with wastewater COD/TOC as the
395 final topic. Additional aspects included in some themes are process variants, such as electro- and
396 photo-based processes; important factors, such as pH, H₂O₂, and current; and catalyst or metal-
397 related aspects. Despite their varying appearances, the results indicate a consistent scientific
398 discourse on the issue. RQ2 assumes that the general topic under investigation is “performance
399 optimization,” which serves as the central core of the discussion, while other topics, such as process
400 variants and operational factors, form subthemes for evaluating the main papers on the subject.
401 According to the thematic map, Theme 3 is located at the center of the topic with maximum
402 intensity because the scientific consensus defines research on this issue as the “evaluation of

performance optimization conditions, such as COD/TOC removal, for wastewater.” The other three themes serve as less intensive variants of the main topic and are therefore secondary.



405

406 *Figure 15 Box Plot of Topic Modeling Analysis for Author Keywords*



407

408 *Figure 16 Box Plot of Topic Modeling Analysis for Titles and Abstracts*

The distribution of author keywords appears tighter, with a standard deviation of approximately 0.11, a narrow interquartile range, and no elongated tails. Therefore, the same article may divide its attention relatively evenly across different topics, which is consistent with the double peak observed earlier in the bar plot. This trend characterizes the “declarative vocabulary” mentioned in previous studies, in which authors place equal emphasis on RSM and DOE and their use for specific purposes. In contrast, titles and abstracts show much higher variability, with a standard deviation of approximately 0.19, long tails, and several outliers exceeding 0.6. This suggests that some papers focus primarily on a single topic, with optimization as the main theme, whereas others address multiple issues. To answer RQ2, titles and abstracts can be used to identify papers whose main messages are clearly expressed, thereby providing good examples of key ideas. However, author keywords may refer to a variety of approaches and methods used in the research rather than emphasizing a single topic.

Based on the methodological priorities outlined above, the results of topic modelling, bar plots, scatter plots, and box plots collectively indicate that the field is dominated by laboratory-scale RSM (CCD/BBD) studies applied to diverse wastewaters, including swine, paper mill, and textile

424 wastewater, with performance metrics such as COD/TOC/color. However, these visualizations also
425 reveal recurring weaknesses that limit external validity, such as incomplete regression diagnostics,
426 including discordance tests and residual analysis; narrow factor ranges with sparse sampling in
427 high-curvature regions; single-metric optimization without simultaneous assessment of reagent or
428 energy consumption; and limited external or pilot-scale validation (GilPavas et al., 2019; Guvenc et
429 al., 2017; Sabour & Amiri, 2017; Salmerón et al., 2019; Schenone et al., 2015; Toor et al., 2021; H.
430 Zhang et al., 2017; X. Zhou et al., 2020). These patterns, which are also evident when RSM results
431 are compared with ML approaches, suggest practical solutions, namely disclosing the design matrix
432 and response values, reporting complete regression diagnostics and residual plots, increasing
433 sampling density in curvilinear regions to reveal interactions and kinetic terms, applying cross-
434 validation or independent/pilot-scale testing to assess predictive performance, quantifying
435 uncertainty around predicted optima through confidence intervals, and adopting multi-objective or
436 hybrid RSM–ANN/kinetic strategies where nonlinear dynamics are expected (Baştürk & Alver,
437 2019; Hassanshahi & Karimi-Jashni, 2018). Implementing these steps, and explicitly documenting
438 them in future experimental reports and pilot demonstrations, will improve reproducibility,
439 strengthen the methodological interpretation of topic-model findings, and further support region-
440 specific multicenter scale-up and validation. This pattern is consistent with the core set, while
441 Guvenc et al. (2017) and Salmerón et al. (2019) confirmed convergence, X. Zhou et al. (2020) and
442 H. Zhang et al. (2017) reported divergent outcomes, highlighting reproducibility issues. DOE
443 designs such as CCD and BBD (Schenone et al., 2015; Toor et al., 2021) remain efficient but show
444 limits in non-linear domains (Baştürk & Alver, 2019), where RSM alone is inadequate. In these
445 cases, ML approaches like ANN (Baştürk & Alver, 2019; Sabour & Amiri, 2017) provide
446 complementary strength despite data demands.

447 Thematic mapping confirms the integration of narratives related to performance optimization,
448 represented by Theme 3, through three subthemes: process variation, factors/catalysts, and
449 experimental design framework. However, high term density does not necessarily imply scientific

significance or scholarly impact. To identify themes that have significantly influenced the
development of this field and examine how they are expressed in key publications, the current
literature review linked the LDA results with document-level citation networks using VOSviewer.

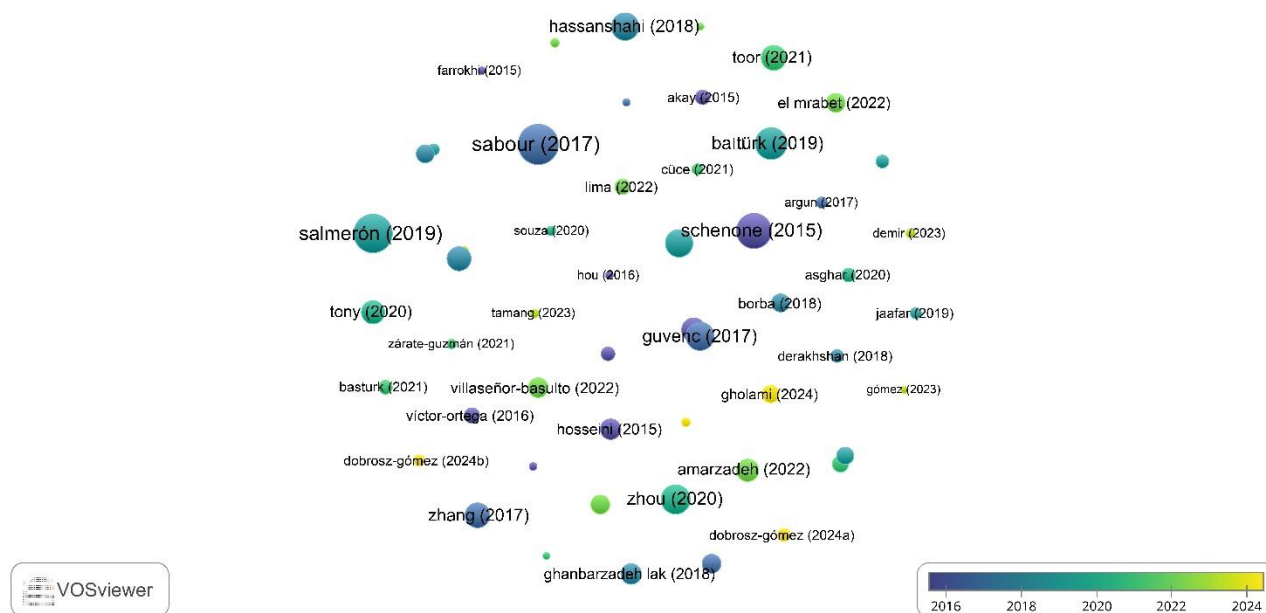


Figure 17 Overlay Visualization of Citation Networks

A key finding from the document-level overlay is that the citation structure displays several high-quality but sparsely linked papers, including several highly cited studies, namely Schenone et al. (2015), Sabour and Amiri (2017), Salmerón et al. (2019), Guvenc et al. (2017), X. Zhou et al. (2020), Toor et al. (2021), and Baştürk and Alver (2019). These studies support methodological standards related to RSM and DOE and are complemented by more recent studies on practical applications and process variations, including electro/photo-based processes, H₂O₂ pilot systems, and reactor design, located toward the network perimeter. Although relatively isolated, the ten most-cited papers all focus on the same thematic thread: optimization of AOP/Fenton processes and their efficiency based on specific metrics, such as COD/TOC, color, and biodegradability. Consequently, these papers are suitable candidates for an RQ2-based synthesis. From a methodological perspective, the findings demonstrate reliance on several key papers and open up possibilities for cross-citation, meta-analysis, and joint pilot implementation of the developed methods. These

467 efforts would strengthen generalizability, uncover latent methodological gaps related to reporting
468 and external validation, and provide a solid foundation for the RQ3 research analysis. The
469 document-level overlay reveals the presence of a small number of highly cited papers that shape
470 this field through overlay visualization, despite relatively limited citation links. The areas covered
471 by these highly cited documents include methodological fundamentals, such as RSM and DOE, as
472 well as applied research involving H₂O₂ and electro-Fenton electrocatalytic approaches.
473 Methodologically, this indicates reliance on paradigmatic studies in the process of scientific inquiry.
474 Addressing this issue will require cross-citation, meta-analytic synthesis, and pilot-scale studies
475 (Baştürk & Alver, 2019; Guvenc et al., 2017; Sabour & Amiri, 2017; Salmerón et al., 2019;
476 Schenone et al., 2015; Toor et al., 2021; X. Zhou et al., 2020). Notably, while Salmerón et al.
477 (2019) and Guvenc et al. (2017) reported consistent improvements at pilot scale, other core studies
478 such as X. Zhou et al. (2020) and H. Zhang et al. (2017) showed divergent outcomes under
479 comparable conditions, underscoring contradictions in reproducibility.

480 **3.3.DOE/RSM Configurations, Results, Gaps, and Future Developments**

481 The review establishes the methodological criteria under which DOE and RSM were applied in the
482 ten most-cited articles identified for RQ2. Using the information summarized in the tables of these
483 articles, this study identified several key elements, including the contextual environment, testing
484 matrix type and variant, research objectives and design, and DOE/RSM methodological criteria,
485 such as design type, independent variables, factor ranges and levels, matrix size, replication, and
486 blocking. Furthermore, modeling methodology, including model form, data transformation,
487 adequacy indices such as R², significance, and goodness of fit, as well as optimization techniques
488 and results, including optimization strategy, predicted optimal points, and accuracy based on
489 confirmatory results and error measures, were recorded. Performance indicators and resource
490 requirements were also documented. As a result, an evidence matrix was generated for all reviewed
491 articles, as shown in Table 3. The following summarizes the quality assessments for the ten core set
492 articles based on four domains: experimental design, model validation, statistical robustness, and

scale-up potential. The scoring scheme, with each domain scored from 0 to 6 and a total score ranging from 0 to 24, is briefly described in the Methodology section. Full scores and per-study comments are available in [Supplementary Table 6](#). This table is intended to provide methodological context for the content synthesis and to highlight strengths and limitations that influence the interpretation of the results.

Table 3 Ten Most-Cited Articles as the Core Set and Quality Assessment of Reviewed Studies

No	Authors, Journal, and Number of Citations	Experimental Design	Model Validation	Statistical Robustness	Scale-up Potential	Total Score
1	(Sabour & Amiri, 2017), <i>Waste Management</i> — 117 citations	5/6	4/6	4/6	1/6	14/24 (Moderate)
Commentary: Comparison of robust and structured methods; minimal external validation.						
2	(Salmerón et al., 2019), <i>Applied Catalysis B: Environmental</i> — 105 citations	5/6	4/6	4/6	6/6	19/24 (High)
Commentary: Pilot-scale validation and operational metrics are comprehensive; model reporting lacks some detail.						
3	(Schenone et al., 2015), <i>Journal of Environmental Management</i> — 89 citations	6/6	4/6	4/6	1/6	15/24 (Moderate)
Commentary: DOE/RSM is well implemented; however, there is no external testing or discussion of scale.						
4	(Baştürk & Alver, 2019), <i>Journal of Environmental Management</i> — 72 citations	5/6	3/6	3/6	1/6	12/24 (Moderate)
Commentary: The hybrid RSM–ANN approach is interesting; validation and statistical reporting need to be strengthened.						
5	(X. Zhou et al., 2020), <i>Chemosphere</i> — 63 citations	5/6	4/6	4/6	3/6	16/24 (Moderate)
Commentary: Optimization and kinetics analysis are sound; discussion of scale-up is present but not in-depth.						
6	(Guvenc et al., 2017), <i>Water Science and Technology</i> — 59 citations	5/6	3/6	4/6	2/6	14/24 (Moderate)
Commentary: DOE and statistics are adequate; external validation and scale-up analysis are lacking.						
7	(GilPavas et al., 2019), <i>Environmental Pollution</i> — 55 citations	5/6	4/6	4/6	3/6	16/24 (Moderate)
Commentary: Application and toxicity focus are strong; broader discussion of scale-up and validation is needed.						
8	(Hassanshahi & Karimi-Jashni, 2018), <i>Ecotoxicology and Environmental Safety</i> — 53 citations	4/6	3/6	3/6	2/6	12/24 (Moderate)
Commentary: The comparison is useful, but validation and statistical reporting are limited.						
9	(H. Zhang et al., 2017), <i>Chemosphere</i> — 46 citations	4/6	3/6	3/6	1/6	11/24 (Moderate)
Commentary: The microscale proof of concept is clear; there is almost no discussion of scale-up.						
10	(Toor et al., 2021), <i>Journal of Environmental Management</i> — 45 citations	5/6	4/6	5/6	1/6	15/24 (Moderate)
Commentary: DOE/RSM and statistical reporting are strong; external validation and scale-up are lacking.						

499 The assessment results indicate that most studies fall into the Moderate category, with one pilot-
500 scale study falling into the High category. These findings were used to guide discussions on model
501 validation, the need for external testing, and scale-up recommendations. For full transparency,
502 criteria weightings, scores per domain, and brief commentary on each article are also included in
503 [Supplementary Table 6](#). Several core studies demonstrate that adapting Fenton processes with
504 alternative iron sources or microscale pretreatment can directly inform industrial wastewater
505 applications, highlighting pathways toward practical deployment (GilPavas et al., 2019; H. Zhang et
506 al., 2017). The table above shows the application of Fenton oxidation to various processes,
507 including landfill leachate treatment, animal waste treatment, domestic wastewater treatment, textile
508 wastewater treatment, paper mill wastewater treatment, coking plant wastewater treatment, and
509 solid waste treatment, along with a pilot study on solar-assisted hydrogen peroxide production via
510 electrocatalysis. Each article used RSM and DOE for factor organization, quadratic model
511 development, and the determination of operational conditions. The selected DOE and RSM
512 approaches are discussed in this section along with performance data and study limitations.

513 In the ten core studies, RSM/DOE remained the primary framework, with CCD and three-level
514 designs chosen for cases requiring curvature modeling and pilot optimization, while BBD and
515 parsimonious-run variants were used when trial economy and factor consistency were more
516 important (Baştürk & Alver, 2019; Salmerón et al., 2019; Schenone et al., 2015; X. Zhou et al.,
517 2020). Many studies applied the quadratic RSM model with ANOVA and a desirability function for
518 optimization. Pilot-scale investigations (Guvenc et al., 2017; Salmerón et al., 2019) underline that
519 scaling electro-Fenton and solar-assisted systems introduces variability in reproducibility,
520 emphasizing the need for robust validation before industrial adoption. However, reporting practices
521 were often inadequate, with some articles not including the complete design matrix, factor coding,
522 residual diagnostics, lack-of-fit tests, or confidence intervals for predicted optima. To address
523 statistical validation needs, studies should report Adjusted R^2 and Predicted R^2 , predictive metrics
524 such as PRESS and RMSE, k-fold cross-validation or hold-out results, residual plots, and

525 confidence intervals for optima. Without these metrics, claims of “optimality” risk overfitting and
526 are difficult to verify or compare across studies (Baştürk & Alver, 2019; Sabour & Amiri, 2017;
527 Schenone et al., 2015). Furthermore, sparse sampling in high-curvature regions magnifies the
528 instability of interaction estimates and hinders extrapolation to pilot studies; therefore, designs with
529 higher sampling densities around curvature should be considered when kinetics or nonlinearity are
530 expected. Developments in AI/ML and hybrid strategies offer practical solutions. ANNs, hybrid
531 RSM–ANNs, genetic algorithms, and metaheuristics can capture strong nonlinearities, model
532 complex interactions, and solve multi-response problems that quadratic RSM often fails to address.
533 Evidence from comparative studies suggests improved out-of-sample predictions and more robust
534 identification of operating conditions before pilot testing. Therefore, benchmarking RSM against
535 ML/hybrid models, with reported cross-validated errors, model selection criteria, and
536 interpretability, should become standard practice when data exhibit nonlinearities or multiple
537 concurrent responses (Baştürk & Alver, 2019; GilPavas et al., 2019; Sabour & Amiri, 2017).
538 Hybridization also facilitates multi-objective Pareto optimization, enabling the integration of
539 technical and economic objectives within a single framework. Environmental sustainability is
540 reinforced by several studies (GilPavas et al., 2019; Hassanshahi & Karimi-Jashni, 2018), which
541 showed improved biodegradability and reduced toxicity following optimized treatments, linking
542 process efficiency to ecological outcomes. Collectively, several papers from core set (Guvenc et al.,
543 2017; Sabour & Amiri, 2017; Salmerón et al., 2019) indicates that while laboratory optimization is
544 mature, technology readiness remains at pilot-scale, requiring multicenter replication to advance
545 toward industrial TRL benchmarks.

546 Economic and operational aspects should be an explicit part of the optimization objective.
547 Economic considerations are evident in several studies (Toor et al., 2021; X. Zhou et al., 2020),
548 where optimization reduced reagent demand yet highlighted electricity and sludge handling as
549 major contributors to operational cost. Traditional control parameters, including pH, Fe dosage,
550 H_2O_2 , H_2O_2 ratio, and time, and electrochemical parameters, such as current density, electrode

spacing, oxygen supply, and in situ H_2O_2 production, affect not only removal efficiency but also energy per unit removal (kWh/kg COD), reagent consumption and oxidant yield, sludge production, electrode/catalyst degradation rate, and OPEX. These metrics are often overlooked in laboratory optimization (Guvenç et al., 2017; X. Zhou et al., 2020). Pilot studies on electrocatalytic H_2O_2 and electro-Fenton showed that mass transfer, energy limitations, and material wear can shift laboratory optimal conditions. Therefore, stepwise validation from mini-pilot to pilot scale, including records of energy use, reagent consumption, and material degradation, is mandatory for optimization to be technically and economically relevant (GilPavas et al., 2019; Salmerón et al., 2019).

For future RSM–Fenton studies, researchers should implement clear and documented validation protocols, such as selecting an appropriate DOE design (CCD, BBD, or three-level design) based on curvature and resource requirements, publishing a complete design matrix with factor coding and experiment sequence, and testing the fit of the quadratic model and its alternatives, such as ANN and mechanistic models, using ANOVA, lack-of-fit tests, residual plots, and quantitative predictive metrics such as Adjusted R^2 , Predicted R^2 , PRESS, and RMSE. Researchers should also conduct k-fold cross-validation or hold-out testing to assess predictive ability and include confidence intervals for optimal conditions. Next, stepwise validation from model to practice should be conducted by verifying optimal conditions through sequential experiments from mini-pilot to pilot scale that record energy per unit removal (kWh/kg COD), reagent consumption and yield ($\text{H}_2\text{O}_2/\text{Fe}$), sludge production, and electrode/catalyst degradation rates. When data exhibit nonlinearity or multi-response behavior, RSM should be benchmarked against AI/ML methods, including ANN and hybrid RSM–ANN, and genetic algorithms or metaheuristics should be considered for multi-objective Pareto optimization. Cross-validated errors, model selection criteria, and interpretability should also be reported to justify the chosen method. Finally, operational-economic metrics, including energy, OPEX, reagent costs, and toxicity/biodegradability impacts, should be integrated as objectives or constraints in the experimental design so that the reported “optima” are statistically

576 verifiable, technically relevant, and economically feasible at pilot or industrial scale. Based on these
 577 suggestions, a list of example questions for future RSM–Fenton studies is summarized in Table 4.

578 *Table 4 Example Checklist Questions for Future RSM-Fenton Studies*

Aspect/Context	Checklist Questions
Statistical validation	Does the study report fit measures, predictive evaluation, and model validation procedures?
Model selection and validation	Are procedures for model selection and out-of-sample testing, such as cross-validation or hold-out testing, included?
Experimental design and reporting	Are the experimental designs and technical information, including matrices, coding, and sequences, published?
Sampling and coverage	Does the sampling scheme cover the range and nonlinear regions relevant to the problem being tested?
Modeling approaches and benchmarking	Are alternative methods, such as ML, hybrid, or mechanistic approaches, systematically compared and benchmarked?
Multi-objective optimization	Does the optimization consider trade-offs among technical, economic, and environmental objectives?
Economic and operational metrics	Are operational and economic metrics, such as energy, material consumption, costs, and waste, reported or considered?
Scale-up and pilot validation	Is there a plan or evidence of phased validation from laboratory to pilot scale to verify transferability?
Material durability and maintenance	Are the durability aspects of components, such as electrodes and catalysts, as well as fouling and maintenance requirements, evaluated?
Transparency and reproducibility	Are raw data, analysis code, and documentation available for replication and meta-analysis?

579

4. Conclusion

Between 2015 and 2025, RSM–Fenton research shifted from an exploratory approach toward an optimization-oriented approach, with collaborations occurring mainly in specific geographic locations. Three themes were identified in the most influential articles: DOE and RSM methods, process variations, and performance studies. Although pollutant removal remains the primary outcome, several gaps affect the implementation of these findings, including insufficient detailed reporting, limited sampling, and low external validation. To address these shortcomings, future studies should adopt several concrete requirements. First, DOE matrices, factor coding, and experimental sequences should be mandatorily reported to ensure transparency and enable meta-analysis. Second, robust statistical validation using Adjusted R^2 and Predicted R^2 , PRESS, RMSE, residual diagnostics, and cross-validation should be applied to verify predictive reliability. Third, multi-objective optimization should integrate pollutant removal with energy use, reagent consumption, sludge formation, and toxicity endpoints. Fourth, incremental pilot validation from mini-pilot to pilot scale, capturing energy and material durability metrics, should be conducted to confirm the transferability of laboratory optimization. Fifth, RSM should be compared with AI/ML and hybrid approaches, including ANNs or evolutionary algorithms, to capture nonlinearities and improve generalizability. By adopting these requirements, future RSM–Fenton studies can strengthen methodological rigor, improve reproducibility, and accelerate the transition from laboratory optimization to sustainable industrial implementation.

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6. CRediT Author Statement

NDH: Funding acquisition, supervision, project administration, conceptualization, writing—review and editing; **GSW:** Conceptualization, methodology, software, data curation, writing—original draft; **SDN, BDZ, YMP, BSR:** Supervision, resources, conceptualization, validation, writing—review and editing.

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