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Review Article

A review of recent trends, advancements, and future directions in near-infrared spectroscopy applications in biofuel production and analysis

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ABSTRACT

The growing demand for sustainable energy solutions has intensified research into biofuel production and analysis techniques. Near-infrared spectroscopy (NIRS) has emerged as a promising tool in this field, yet a comprehensive understanding of its applications and impact remains lacking. This study aims to systematically review and analyse the applications of NIRS in biofuel production and analysis, providing insights into research trends, key contributors, and future directions. A bibliometric analysis was conducted using the Scopus database, covering publications from 1996 to 2023. The methodology included quantitative analysis, thematic mapping, factorial analysis, and citation analysis using the Bibliometrix package in R. The findings reveal a significant growth in NIRS biofuel applications, with an 11.85% annual increase in publications. The USA, China, and Brazil emerged as leading contributors, with strong international collaborations. Key applications include real-time monitoring of biodiesel production, biomass characterisation, and biogas production analysis. The integration of machine learning with NIRS data analysis represents a notable trend, enhancing prediction accuracy and model robustness. Thematic analysis identifies emerging research clusters in process monitoring, quality control, and feedstock analysis. These findings have important implications for both research and industry. The versatility of NIRS across various biofuel types and production stages suggests its potential for improving process efficiency and product quality. The identified research trends provide direction for future studies, particularly in standardising methodologies and developing more sophisticated data analysis techniques. This review highlights NIRS as a key technology that is enabling the advancement of sustainable biofuel production.

1. Introduction

The urgent need to address climate change and achieve sustainable development goals is driving a significant transformation in the global energy sector [1–3]. Biofuels, which have emerged as a promising alternative to conventional fossil fuels, are at the forefront of this transition [4–6]. As the world's energy demand rises, biofuels offer a viable pathway to reduce greenhouse gas emissions [7,8] and strengthen energy security [9,10], which is essential in creating a more sustainable and diverse energy mix.

Biofuels are particularly impactful in decarbonising transport sectors that are challenging to electrify, such as trucking, shipping, and aviation [11,12]. By serving as a low-carbon alternative compatible with existing engines, biofuels present a practical replacement for fossil fuels [13,14]. Under the global energy transition and aligned with the Net Zero Scenario, biofuel use in transport is expected to increase significantly by

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2030, with a growing portion derived from waste, residues, and non-food crops. In particular, the aviation sector will need substantial bio-jet kerosene advancements to meet decarbonization targets [15].

Most biofuel production relies on conventional feedstocks, including sugarcane, corn, and soybeans [16]. Expanding biofuel production to advanced feedstocks is crucial to reduce impacts on land use, food and feed prices, and environmental concerns [5,17]. This transition is essential to ensure biofuel growth does not compete with food production [18]. According to the International Renewable Energy Agency's (IRENA) 1.5 °C Scenario, bioenergy is projected to contribute over 18 % of total final energy consumption by 2050, comprising both direct uses (16 %) and electricity generation (2.3 %) [19]. This highlights biofuels' pivotal role in supporting climate and sustainable development goals through renewable energy solutions.

The importance of biofuels in transitioning to sustainable energy systems is increasingly evident as fossil fuel reserves dwindle and climate change concerns intensify. Biofuels offer a renewable alternative that contributes to carbon neutrality and supports a sustainable energy transition [4]. Derived from renewable biomass, biofuels are vital to the circular economy, converting organic waste and agricultural residues into valuable energy resources [20–22]. Their production aligns with sustainability principles, reducing carbon footprints, addressing waste management issues, and creating economic opportunities in rural and agricultural sectors [4,23]..

Biofuels are categorised into different generations, each with unique benefits and limitations. For example, first-generation (1G) biofuels are derived directly from food crops like corn and sugarcane, while second-generation (2G) biofuels come from non-food biomass, such as agricultural residues and woody crops [16,24]. On the other hand, third-generation (3G) biofuels are produced from algae and other microorganisms [25], and fourth-generation (4G) biofuels incorporate advanced technologies, including genetic engineering and nanotechnology, to enhance energy yield and reduce environmental impact [4,26]. These advancements are essential for promoting a green economy and achieving long-term sustainability.

In this context, developing and optimising biofuel production processes have become intense research and innovation areas [27,28]. Among the various analytical techniques employed in this field, near-infrared spectroscopy (NIRS) has emerged as a powerful and versatile tool, revolutionising how we approach biofuel production and analysis [29–31]. NIRS operates on the principle of measuring the absorption of near-infrared light by organic molecules, providing a rapid, non-destructive method for analysing the chemical composition of complex biological matrices [32–34]. This technique is particularly well-suited for analysing biomass and biofuels, providing significant advantages over traditional analytical methods [35–37]. The non-destructive nature of NIRS enables real-time monitoring of production processes without compromising sample integrity [29,38]. At the same time, its ability to analyse multiple components simultaneously provides a comprehensive view of sample composition with minimal sample preparation [33].

The application of NIRS in biofuel production covers the entire value chain, from feedstock characterisation [39,40] to process monitoring [41] and quality control [42,43] of the final product. NIRS has proven invaluable in biomass characterisation for rapidly assessing key properties such as moisture content [44,45], lignin, cellulose, and hemicellulose composition [46–48]. This capability is crucial for optimising feedstock selection and pretreatment strategies, ultimately enhancing the efficiency of biofuel production processes. During production, NIRS enables real-time monitoring of critical parameters in processes such as fermentation and transesterification, allowing for rapid adjustments to maintain optimal conditions and ensure consistent product quality [36]. The versatility of NIRS extends to the analysis of various types of biofuels, including bioethanol, biodiesel, and advanced biofuels, providing a unified analytical approach across different production platforms.

The growing body of literature on NIRS applications in biofuel research indicates the technique's versatility and potential to address key challenges in the sector. However, the sheer volume and diversity of published works can make it challenging for researchers to navigate the field and identify emerging trends, knowledge gaps, and promising avenues for future investigation. This is where bibliometric analysis comes into play, providing a systematic and quantitative approach to mapping the intellectual area of NIRS in biofuel research.

Bibliometric studies can reveal patterns in publication trends, research collaborations, and citation networks that may not be immediately apparent through traditional literature reviews by leveraging advanced data analytics and visualisation techniques [49–53]. Several existing review papers have touched upon the applications of NIRS in biofuel production and analysis, highlighting its importance in various stages of the biofuel value chain. For instance, Zhang [54] reviewed the developments in biodiesel quality analysis using the infrared technique. Sharma et al. [55] comprehensively reviewed the application of modern machine learning technologies in regulating and monitoring biofuel production from waste biomass. Horf et al. [56] evaluated the application of optical spectrometry, particularly NIRS, as a fast, cost-effective alternative to traditional laboratory analyses for determining nutrient concentrations in liquid organic fertilisers such as livestock manures and biogas digestates.

Other studies include Karimi and Taherzadeh [57], who critically reviewed the analytical methods used to evaluate the efficiency of lignocelluloses' physical, thermal, chemical, and biological pretreatments. Lupoi et al. [58] provided an overview of high-throughput and traditional spectroscopic techniques used for the compositional and structural characterisation of lignocellulosic biomass. Singh et al. [59] evaluated the microstructural and compositional analysis methods for lignocellulosic biomass using various imaging and spectroscopic techniques. Skvaril et al. [36] evaluated the application of NIRS in various biomass conversion processes, including physical, thermochemical, biochemical, and physiochemical processes. Challagulla et al. [60] reviewed the application of various analytical techniques, including fluorescent lipid-soluble dyes, nuclear magnetic resonance, Raman, Fourier transform infrared, and near-infrared spectroscopy, to assess lipids in microalgae. Yuan et al. [61] explore the application of nanomaterials in microalgal biotechnology to enhance the production of algal biomass and valuable metabolites, such as lipids, proteins, and exopolysaccharides.

Even though these reviews supra reveal pertinent insights into specific areas of NIRS application in biofuels, they do not provide a holistic, quantitative assessment of the field's evolution over time. This is where the current bibliometric analysis aims to fill a critical gap in the literature. This study seeks to uncover the underlying structure of the research domain, identify key players and influential works, and map the intellectual connections that have shaped the field by employing advanced bibliometric tools and techniques. The importance of conducting a bibliometric analysis on NIRS applications in biofuel production and analysis cannot be overstated. Firstly, it provides a bird's-eye view of the research areas, allowing researchers, policymakers, and industry stakeholders to quickly grasp the state of the art and identify emerging trends. This macro-level perspective is particularly valuable in a rapidly evolving field like biofuel research, where staying abreast of the latest developments is crucial for driving innovation and informing strategic decision-making. Secondly, bibliometric analysis can reveal hidden patterns and relationships within the literature that might not be apparent through traditional review methods. We can identify clusters of closely related research topics and trace the flow of ideas between different sub-domains of NIRS application in biofuels. This information can be invaluable for researchers looking to position their work within the broader field context or identify potential collaborators with complementary expertise. Moreover, bibliometric analysis can highlight knowledge gaps and underexplored areas, pointing to promising directions for future research. We can identify emerging topics gaining traction in the scientific community and areas that may be ripe for further investigation by examining the temporal evolution of research themes and methodologies.

2. Materials and methods

This review followed the Preferred Reporting Items for Systematic Reviews and Meta-Analyses (PRISMA) approach to conduct a comprehensive bibliometric analysis of NIRS applications in biofuel production and analysis. The Scopus database was selected as the primary source for data extraction, covering publications from 1996 to 2023. Scopus was chosen over other databases like Web of Science, PubMed, and Google Scholar due to its broader coverage of peer-reviewed literature, particularly in science, technology, and engineering [62,63]. Additionally, Scopus provides more consistent and accurate metadata, which is vital for conducting reliable bibliometric analyses. The database also provides comprehensive citation tracking and author profiles, enabling a more thorough examination of research impact and collaboration networks.

The search strategy employed a combination of keywords to retrieve relevant literature comprehensively. The primary search query was structured as follows: ("near infrared spectroscopy" OR "NIRS") AND ("biofuel" OR "biofuels" OR "biodiesel" OR "bioethanol" OR "biogas" OR "biomass fuel"). This combination of terms was designed to capture the full spectrum of near infrared spectroscopy applications across various biofuel types and production processes. The initial search in the Scopus database yielded 365 documents, as shown in Fig. 1.

After that, a set of inclusion and exclusion criteria was applied to refine the search results and ensure the inclusion of only the most relevant and high-quality publications. The inclusion criteria include peer-reviewed papers, conference papers, and book chapters, as these publication types typically undergo rigorous academic scrutiny and represent the most current and significant contributions to the field. On the other hand, the exclusion criteria filtered out non-English publications, short surveys, editorials, and books. Non-English publications were excluded to maintain consistency in the analysis and avoid potential misinterpretations due to language barriers. Short surveys and editorials were omitted as they often lacked the depth and original research content required for a comprehensive review. Books were excluded as they typically consolidate existing knowledge rather than presenting new research findings. Applying these criteria resulted in a final dataset of 345 documents for the bibliometric analysis.

The bibliometric analysis was conducted using the Bibliometrix package in R software, complemented by the Biblioshiny library, which provides a user-friendly web interface for advanced bibliometric analysis [64–66]. This powerful combination of tools allows for a multiple examination of the literature, enabling the extraction of valuable insights into research trends, collaborations, and impact. The following analyses were conducted: (i) quantitative analysis (examining publication trends over time, identifying the most productive authors, institutions, journals, and countries), (ii) conceptual structure (co-network analysis, thematic mapping and evolution, and factorial analysis) and (iii) keyword and trend topics analysis.

The use of Bibliometrix and Biblioshiny facilitated the generation of various visualisations, including thematic maps and evolutions, word cloud, trend topics, and country collaboration networks. These visual representations aid in interpreting complex bibliometric data, making patterns and trends more accessible to readers. Furthermore, the analysis included a temporal dimension, examining how research focus, methodologies, and collaborations have evolved over the study period (1996–2023). This longitudinal perspective is crucial for identifying emerging trends, shifts in research priorities, and the maturation of certain research areas within the field.

The methodology employed in this review provides a comprehensive overview of the current state of NIRS applications in biofuel production and analysis. It lays the groundwork for identifying research gaps and future directions. This study provides several perspectives on the field's development, key contributors, and emerging trends by systematically analysing the literature through multiple lenses – quantitative metrics, thematic evolution, collaboration networks, and citation impacts. The rigorous data collection and analysis approach and advanced bibliometric tools ensure that this review's findings are comprehensive and

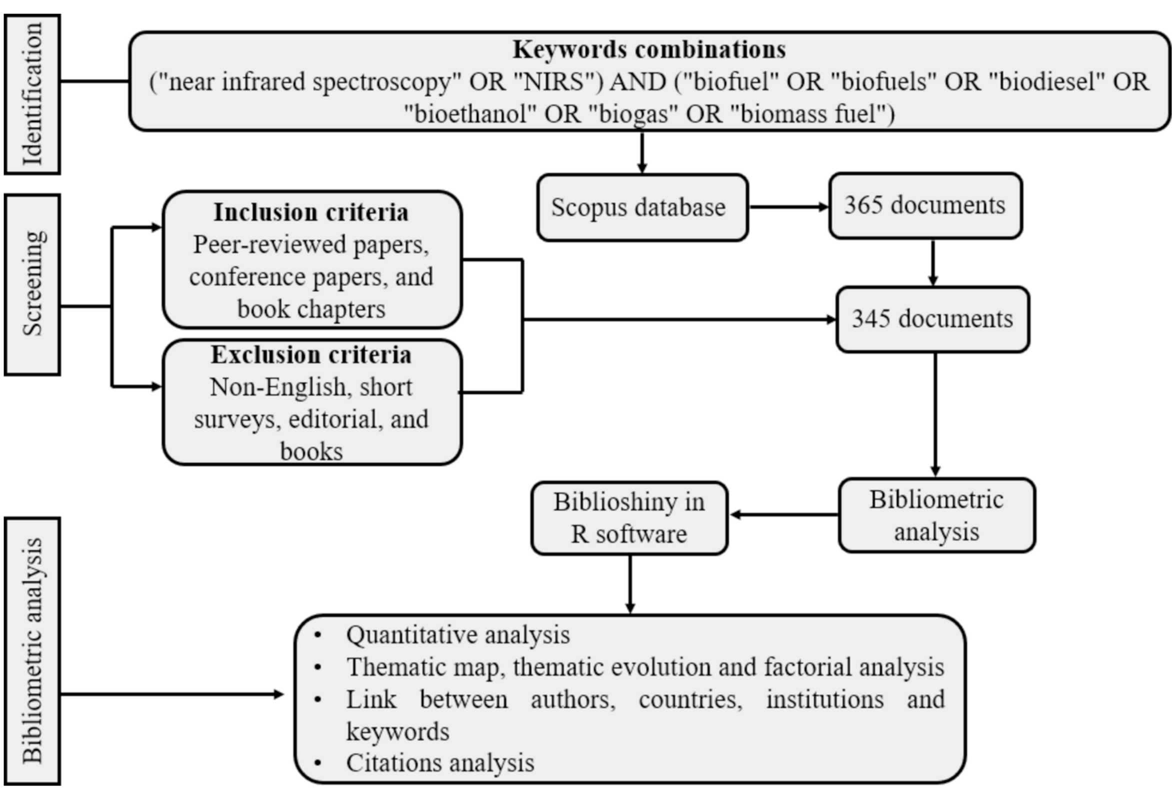

Fig. 1. Study approach adopted for the bibliometric analysis.

robust. The insights derived from this methodology can inform researchers, policymakers, and industry practitioners about the current field of NIRS in biofuel research, highlighting areas of rapid growth, the potential for collaboration, and promising avenues for future investigation.

3. Results and discussion

The results obtained from the bibliometric analysis are presented and discussed comprehensively in the section.

3.1. Overview of bibliometric data

Fig. 2 presents an overview of the bibliometric data extracted from the Scopus database from 1996 to 2023. It can be observed that the research areas have shown significant growth and collaboration, as evidenced by the substantial number of documents (1348) produced by a large pool of authors (3366) from 403 different sources. The annual growth rate of 11.85 % indicates a steady increase in research output over the two decades, suggesting the growing importance and interest in NIRS biofuel applications. The number of references means the research is well-grounded in existing literature, building upon previous work in the field. The average of 4.63 co-authors per document and the 19.44 % rate of international co-authorship demonstrate a strong collaborative nature in this research area, likely contributing to knowledge exchange and potentially more comprehensive studies. However, the presence of 36 single-author publications indicates that some researchers are also working independently, perhaps on more specialised topics. The average document age of 7.78 years suggests that while ongoing research exists, a body of established work remains relevant. The impressive average of 28.42 citations per document indicates that the research in this field has a significant impact and is widely referenced by other scientists, emphasising its importance in the broader context of biofuel research and development. The 2967 author's keywords highlight several specific topics and approaches within the field.

3.2. Publications trend and distribution

The annual article production in Fig. 3 shows a clear trend of growth and fluctuation in research output over nearly three decades. The research area started with minimal activity in the late 1990 s, with only sporadic publications (1 in 1996, 1 in 1999). The early 2000 s saw a slight increase in interest, but publication numbers remained in the low single digits annually. A significant turning point occurred in 2007, marking the beginning of a rapid growth phase. From 2007 to 2011, there was a substantial increase in publications, jumping from 7 articles in 2007 to a peak of 31 in 2011. This surge indicates growing interest in biofuels as alternative energy sources, and advancements in NIRS technology make them more applicable to biofuel research. The period from 2011 to 2018 represents the most productive year for the field, with annual publications consistently above 20 and reaching as high as 32 in 2017. This sustained high output suggests a development of the research area, with established methodologies and a growing community of researchers. However, from 2019 onwards, there was a noticeable decline in publication numbers, dropping to 15 in 2019 and further to 10 in 2020. This decrease could be attributed to various factors, including shifts in research priorities, funding changes, or the impact of the global COVID-19 pandemic on research activities.

However, there was a slight rebound in 2021 and 2022, with a more significant increase to 26 publications in 2023, possibly indicating renewed interest or the fruition of delayed research projects. The overall trend implies that NIRS in biofuel production and analysis has evolved from a niche topic to a well-established research area, experiencing phases of rapid growth, stability, and recent fluctuations. These patterns may indicate broader trends in renewable energy research, technological advancements in spectroscopy, and changing global priorities in sustainable fuel production. The recent uptick in publications could signal a new phase of innovation or application in the field, potentially driven by increasing urgency in developing sustainable energy solutions.

3.3. Countries' distribution and collaborations

Fig. 4 illustrates the global distribution of research output across 45 countries. The results show significant disparities in research activity and potential areas of expertise. Brazil has the highest publications, closely followed by China and the USA, forming a clear top tier of countries driving research in this field. This leadership likely stems from these nations' substantial investments in biofuel technology, vast agricultural resources, and strong research infrastructures. The dominance of Brazil is particularly noteworthy, possibly demonstrating its long-standing commitment to biofuels, especially ethanol production from sugarcane [67–69]. The second tier comprises European countries, with Germany, Spain, France, and Denmark showing significant contributions. This European cluster suggests a strong regional focus on NIRS applications in biofuels, possibly driven by EU renewable energy policies and research funding initiatives. The presence of Denmark in this group is especially interesting, indicating a specialisation in this area despite its smaller size compared to its neighbours. A third tier of countries, including Italy, Portugal, Japan, and Sweden, with publications ranging from 39 to 56 articles, represents nations with moderate

Fig. 2. Overview of bibliometric data.

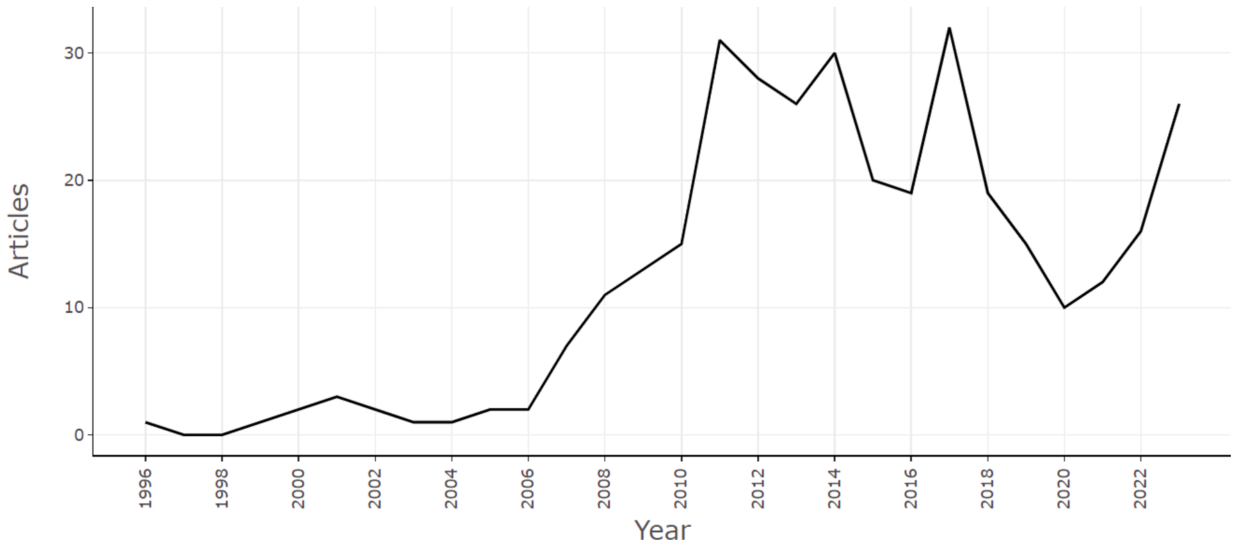

Fig. 3. Publication trend and distribution.

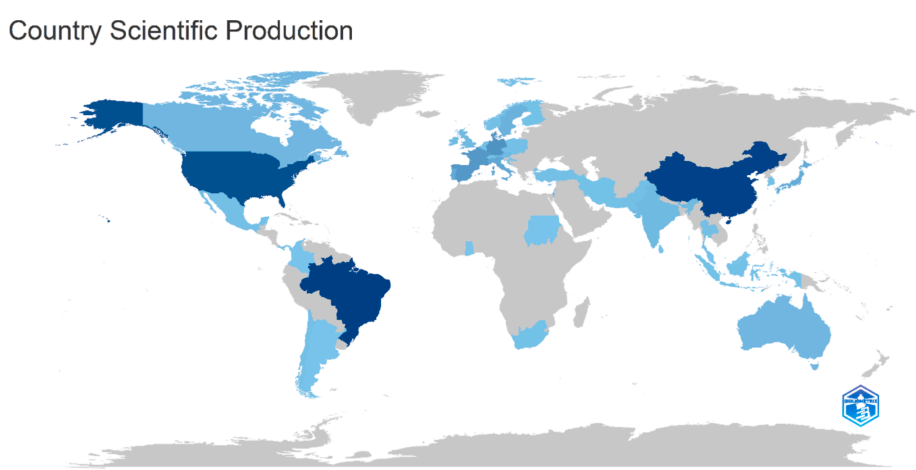

Fig. 4. Country specific production.

but consistent research output. The involvement of Japan in this tier highlights Asian interest beyond China, while Portugal's position suggests a strong focus relative to its size. The map reveals a long tail of countries with fewer than 30 publications, including developed nations like Canada, Australia, and the UK, as well as emerging economies such as India and Thailand. This distribution implies that while research on NIRS in biofuels is globally distributed, it remains concentrated in a relatively small number of countries. The presence of countries like Israel, Pakistan, and South Africa, although with fewer publications, indicates a widespread interest in this technology, possibly driven by local energy needs or agricultural capabilities. These publications' distributions have several implications. First, it suggests that knowledge and expertise in NIRS for biofuel applications are not evenly distributed globally, which could impact the adoption and development of these technologies in different regions. Second, the strong showing of countries like Brazil and China alongside traditional research countries like the USA and Germany indicates a shift in the global research sector, with emerging economies playing a crucial role in renewable energy technologies. Lastly, the wide geographical spread, comprising countries from all continents, highlights the global relevance of NIRS in biofuel research, reflecting worldwide interest in sustainable energy solutions and the potential for international collaboration in advancing this technology.

The country collaboration map in Fig. 5 shows a complex network of international research partnerships with significant implications for the global advancement of this technology. The map shows varying levels of collaboration intensity, represented by different shades of blue, with darker shades indicating higher levels of research output or collaboration. The United States, China, and Brazil emerge as key nodes in this global research network, as evidenced by their darker blue colouration. This is consistent with these countries' significant investments in biofuel technology and their large agricultural sectors. Europe also appears as an important collaborative hub, with several countries shown in medium to dark blue, suggesting strong intra-European cooperation and connections with other global partners. The map illustrates several prominent collaboration lines, notably between the US and Europe, the US, and South America (likely Brazil), as well as Europe and China. These lines represent the most significant international research partnerships in the field. Australia and parts of Southeast Asia are also highlighted, indicating their involvement in the global research effort, although potentially to a lesser degree than the major players. The participation of countries across different continents accentuates the global nature of NIRS research in biofuels, suggesting the widespread interest in sustainable energy solutions. However, the map also reveals potential gaps in collaboration, particularly in Africa, Central Asia, and parts of South America, which appear in lighter shades of grey. This

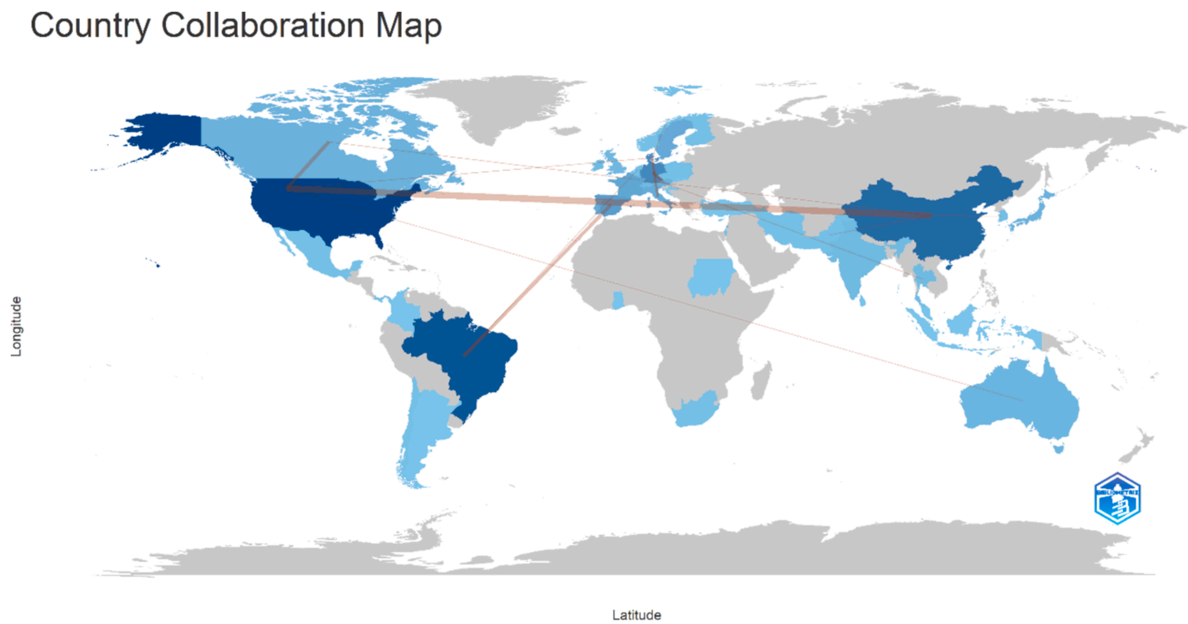

Fig. 5. Country collaboration map.

disparity could indicate limited research capacity, fewer resources, or different energy priorities in these regions.

The results suggest that knowledge transfer and technological advancements in NIRS for biofuels are likely concentrated among a select group of countries, potentially leading to faster progress and risking excluding other nations from cutting-edge developments. Also, the strong international links, especially between continents, imply that research in this field benefits from diverse perspectives and resources, potentially leading to more robust and globally applicable solutions. However, the apparent lack of involvement from certain regions could mean missed opportunities for developing localised solutions or tapping into unique biomass resources. Furthermore, this collaboration pattern may influence global policies and standards related to biofuel production and analysis, with the most active countries potentially shaping the direction of the field.

Fig. 6 depicts the corresponding authors' countries. It can be seen that Brazil emerges as the leading contributor with 41 single country publications (SCP) and 8 multiple country publications (MCP). This suggests Brazil's significant investment and interest in NIRS biofuel applications, likely due to its well-established biofuel industry, particularly in ethanol production from sugarcane. China and the USA follow closely, each with 31 SCPs, but China edges ahead with 9 MCPs compared to the USA's 5, indicating China's stronger international collaboration in this field. European countries, notably Denmark (14 SCP, 7 MCP), Germany (16 SCP, 5 MCP), and Spain (11 SCP, 3 MCP), also demonstrate substantial contributions, reflecting the European Union's push for renewable energy sources.

France's 12 SCPs without MCP suggest a more internally focused research approach. The presence of developed and developing nations, including Thailand, Poland, and Israel, emphasises the global interest in

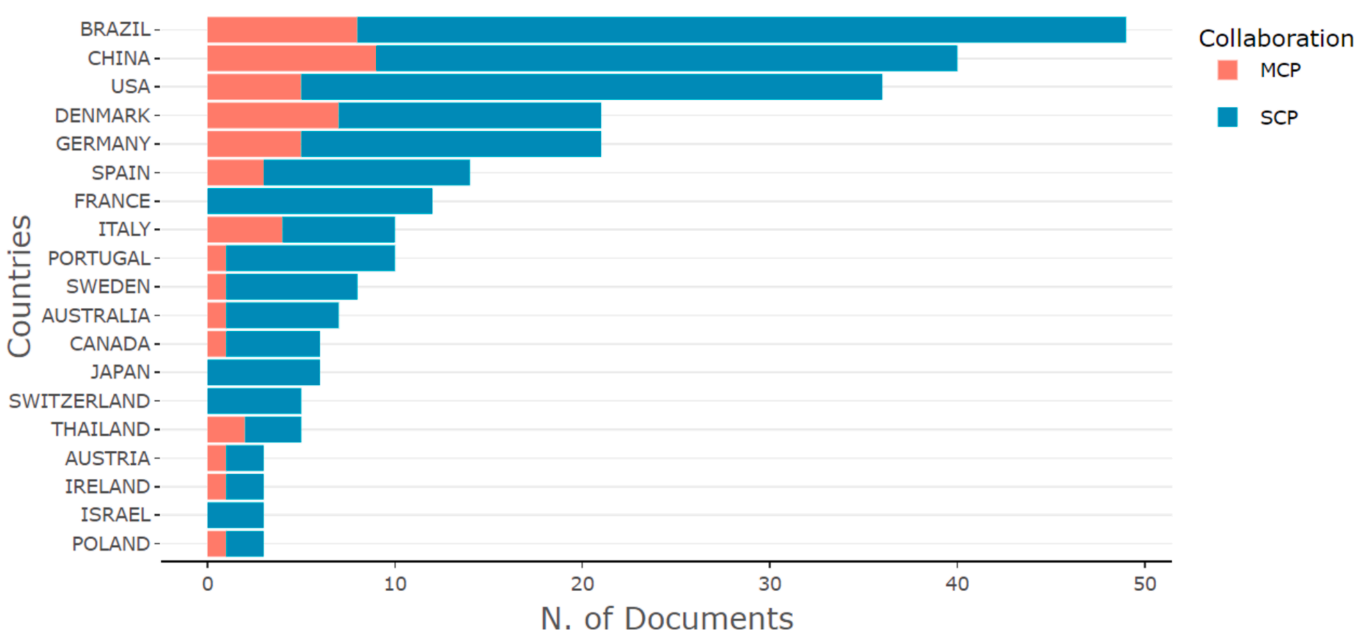

Fig. 6. Depicts the corresponding authors' countries.

NIRS for biofuel applications. The varying ratios of SCP to MCP across countries indicate differing levels of international collaboration, with some nations like Denmark and Italy showing a higher proportion of collaborative work. This global distribution of research efforts highlights the widespread recognition of NIRS as a crucial tool in biofuel production and analysis. The results also imply potential opportunities for increased international collaboration, especially for countries with low MCP counts. Furthermore, the concentration of publications among a few top countries suggests these nations may be at the forefront of technological advancements in this field, potentially influencing global biofuel policies and markets. The relatively lower publication counts from some technologically advanced countries like Japan and Switzerland may indicate either a different research focus or untapped potential in NIRS applications for biofuels in these regions.

4. Keyword analysis and identification of trends topics

The word cloud in Fig. 7 presents a comprehensive overview of the key concepts and applications. It can be seen that the most prominent terms are “near-infrared spectroscopy,” “anaerobic digestion,” and “biodiesel,” indicating their central importance in this field. The prominence of these terms suggests that NIRS is widely used for analysing and monitoring biodiesel production processes, particularly those involving anaerobic digestion. Other significant terms include “biogas,” “biomass,” “multivariate calibration,” and “chemometrics,” highlighting the diverse range of biomass feedstocks and analytical techniques employed in biofuel research and production. The presence of terms like “partial least squares regression” and “multivariate analysis” accentuates the importance of advanced statistical and mathematical methods in interpreting NIRS data for biofuel applications. Including specific biofuels such as “bioethanol” and “biodiesel” indicates that NIRS is utilised across various biofuel types. Terms like “process monitoring,” “online monitoring,” and “quality control” suggest that NIRS is valued for its real-time monitoring capabilities in biofuel production processes. The appearance of “lignin” and “lignocellulosic biomass” points to the relevance of NIRS in analysing complex biomass compositions, which is crucial for optimising biofuel production from plant-based materials. The occurrence of “calibration models” and “NIR spectroscopy” emphasises the need for accurate and reliable spectroscopic methods in biofuel analysis.

It is worth mentioning that this word cloud reveals the multidisciplinary nature of NIRS applications in biofuel research, comprising aspects of analytical chemistry, process engineering, and data science. It also highlights the potential of NIRS as a versatile and powerful tool for characterising biomass feedstocks, monitoring biofuel production processes, and ensuring the quality of biofuel products. The implications of these results suggest that NIRS plays a crucial role in advancing the field of biofuels by providing rapid, non-destructive, and cost-effective analytical solutions throughout the biofuel production chain, from feedstock assessment to final product quality control.

The trend topics within the study period are shown in Fig. 8. The most recent trends, appearing towards the top of the figure, show a growing emphasis on advanced analytical techniques such as machine learning and partial least squares regression. This suggests a shift towards more sophisticated data analysis methods to interpret the complex spectral data obtained from NIRS. The emergence of “biochemical methane potential” as a recent topic indicates an increased interest in biogas production and its optimisation using NIRS. Process monitoring and ash content analysis have also gained prominence recently, highlighting the importance of real-time quality control in biofuel production processes.

Moving down the figure, core NIRS applications in biofuel analysis, including spectroscopy, near-infrared spectroscopy, and chemometrics, have maintained consistent relevance throughout the period. This highlights the enduring importance of NIRS as a fundamental analytical tool in the biofuel industry. The middle section of the figure shows a cluster of topics that gained significant attention around 2014–2016, including PLS (partial least squares), multivariate data analysis, and various biofuel-related terms such as biomass, anaerobic digestion, and biodiesel. This period appears to mark a surge in research activity applying NIRS to diverse aspects of biofuel production.

Earlier topics on the timeline, such as transesterification, glycerol, and calibration models, indicate the initial focus on developing and refining NIRS methods for specific biofuel production processes, particularly biodiesel. The presence of “enzymatic hydrolysis” in the earlier years suggests early applications of NIRS in bioethanol production from lignocellulosic biomass.

These trends have several implications. Firstly, they demonstrate the dynamic nature of NIRS applications in biofuel research, with continuous advancements in the scope of applications and the sophistication of data analysis techniques. The shift towards machine learning and advanced regression methods implies a growing capability to extract

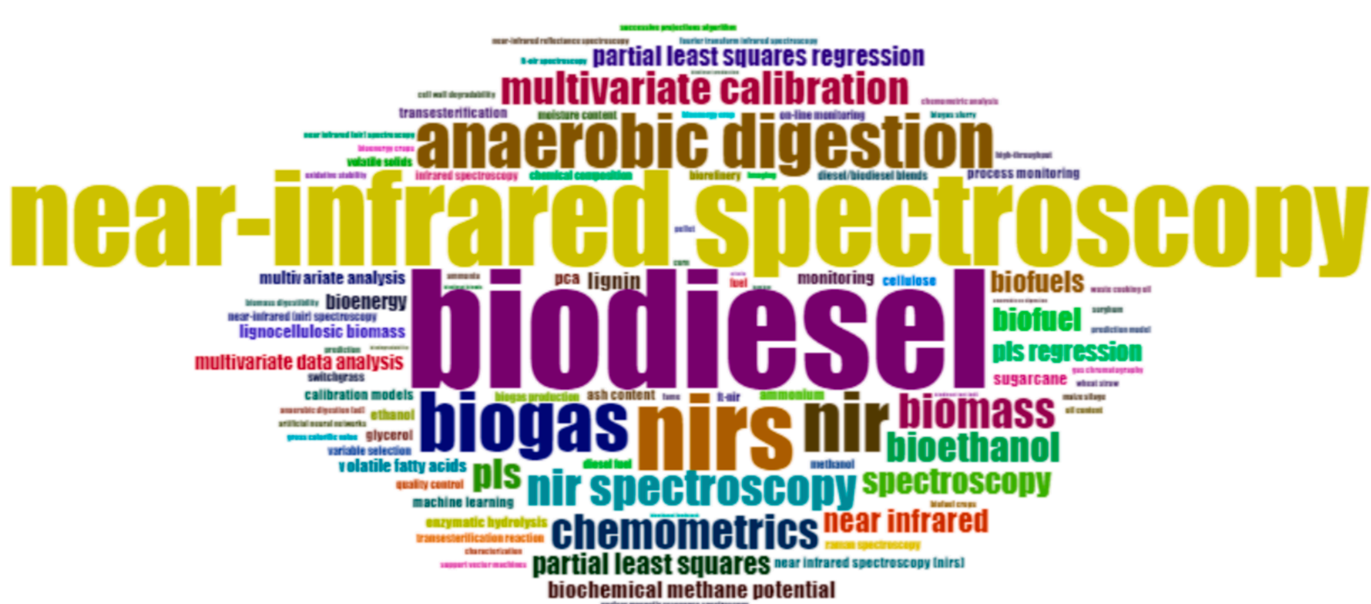

Fig. 7. Word cloud of keywords (100 words).

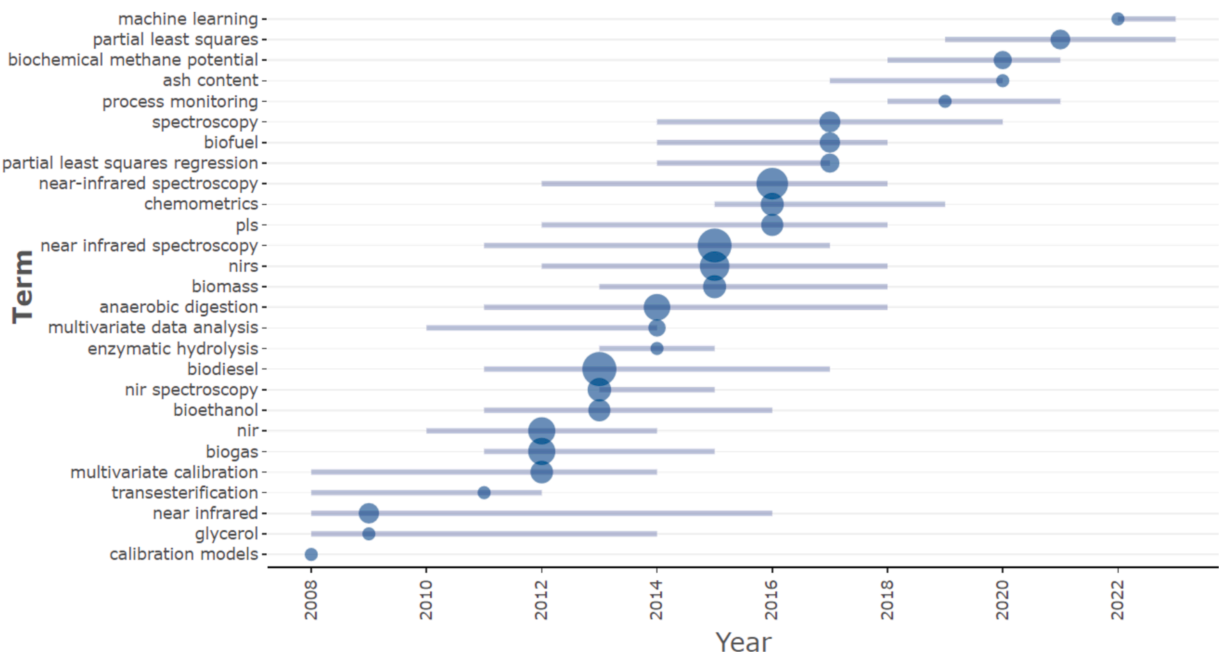

Fig. 8. Trend topics within the study period.

more accurate and nuanced information from NIRS data. This could improve process control, feedstock characterisation, and product quality assessment in biofuel production. The consistent presence of various biofuel types (biodiesel, bioethanol, biogas) throughout the timeline emphasises the versatility of NIRS across different biofuel sectors. Furthermore, the recent focus on process monitoring and specific parameters like biochemical methane potential suggests a trend towards more targeted and specialised applications of NIRS in biofuel production, potentially leading to optimised processes and increased efficiency in the industry.

4.1. Conceptual structure

The conceptual structure involves examining the thematic map, thematic evolution, factorial analysis, and co-occurrence network analysis. Each method provides insights into the development and interrelationships within a field. The co-occurrence network analysis in Fig. 9 maps the relationships between frequently co-occurring keywords or themes. It can be observed that "near-infrared spectroscopy" and "biodiesel," are at the centre of the network, indicating their central importance and frequent co-occurrence in the literature. This centrality accentuates NIRS as an essential analytical technique in biodiesel research and production. From these central nodes, several distinct clusters can be observed that represent different aspects of biofuel research. It can also be seen that one prominent cluster revolves around "biogas," linking to terms like "biochemical methane potential," "volatile fatty acids," and "anaerobic digestion." This cluster highlights the significant application of NIRS in biogas production and process monitoring. Another notable cluster centres on "biomass," connecting to terms such as "lignocellulosic biomass," "cellulose," and "enzymatic hydrolysis," which points to the use of NIRS in analysing and characterising biomass feedstocks for various biofuel applications. The

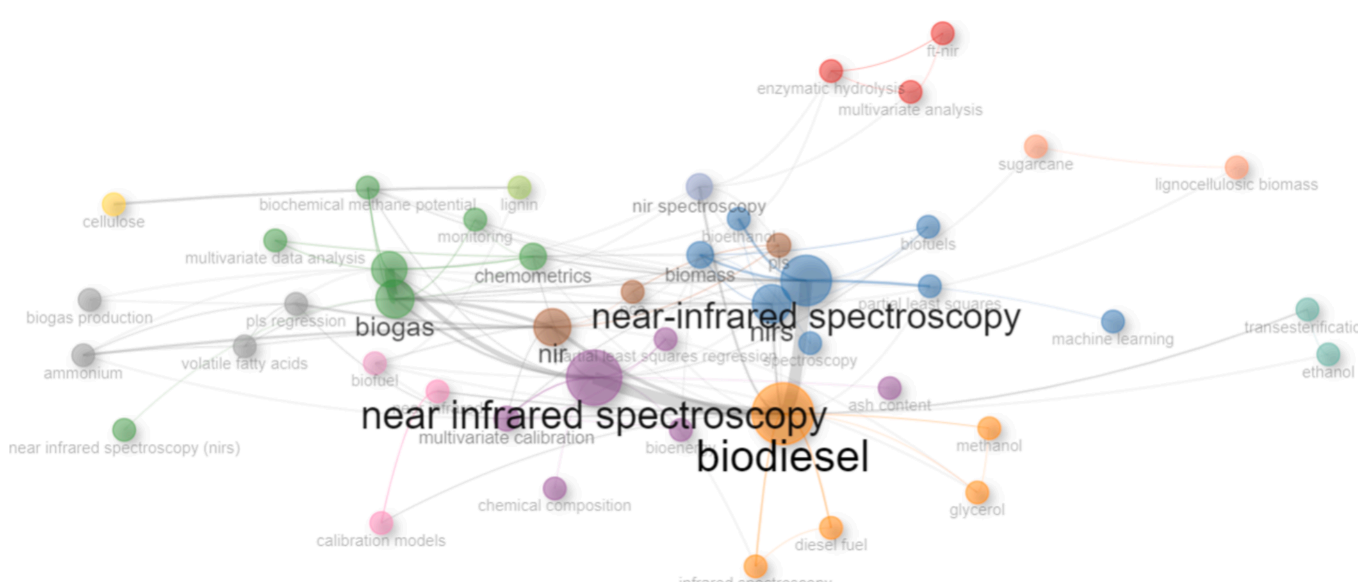

Fig. 9. Keywords co-occurrence network analysis.

network also reveals the importance of data analysis techniques in NIRS applications, with nodes for "chemometrics," "multivariate analysis," "pls regression," and "machine learning." These connections demonstrate the crucial role of advanced statistical and computational methods in interpreting the complex spectral data obtained from NIRS.

Also, links between different biofuel types, such as connections between biodiesel, biogas, and bioethanol nodes, can be seen. This suggests that NIRS techniques and methodologies are often transferable or relevant across various biofuel production processes. The presence of terms like "process monitoring," "quality control," and "online monitoring" indicates the value of NIRS for real-time analysis and quality assurance in biofuel production. The network also highlights specific components and properties relevant to biofuel analysis, such as "glycerol," "fatty acids," "ash content," and "chemical composition." This demonstrates the versatility of NIRS in measuring a wide range of parameters critical to biofuel quality and production efficiency.

The implications of this co-occurrence network are significant for biofuel research and production. Firstly, it emphasises the versatility and widespread adoption of NIRS across various aspects of biofuel science, from feedstock characterisation to process monitoring and final product analysis. The strong interconnections between NIRS and different biofuel types suggest that advancements in NIRS techniques could benefit the entire biofuel industry. The prominence of data analysis and chemometric nodes implies a trend towards more sophisticated interpretation of NIRS data, potentially leading to more accurate and informative results. This could drive improvements in process optimisation, quality control, and efficiency in biofuel production. Furthermore, the network reveals potential areas for interdisciplinary research, where techniques developed for one type of biofuel could be adapted or applied to others.

Fig. 10 displays the thematic mapping visually representing the distribution and relationship of key themes within the research domain. The thematic mapping categorises themes based on their degrees of development (density) and relevance (centrality). The basic themes contain words such as "biogas," "NIR," and "anaerobic digestion," which are fundamental concepts with high relevance but lower development. This suggests that these topics form the core foundation of NIRS applications in biofuel research but may not be the current focus of intense development. The motor themes quadrant, representing highly developed and central topics, includes "near-infrared spectroscopy," "bioenergy crops," "biomethane," and advanced analytical techniques like "artificial neural networks" and "support vector machines." This indicates that NIRS is a driving force in biofuel research, particularly in analysing bioenergy crops and optimising biomethane production, with a strong emphasis on sophisticated data analysis methods. The presence of "lignocellulosic biomass" and "multivariate analysis" in this quadrant highlights the importance of NIRS in characterising complex biomass feedstocks and the necessity of advanced statistical techniques in interpreting spectral data. The niche themes quadrant, representing highly developed but less central topics, includes specific applications and techniques such as "anaerobic digestion," "process analytical technologies," and "biodiesel production." This suggests that though these areas are well-developed, they may be more specialised or focused within the broader field of NIRS in biofuel research. The presence of "handheld spectrometers" and "virtual standards" in this quadrant indicates ongoing innovation in NIRS instrumentation and calibration methods. The emerging or declining themes quadrant, showing less developed and less central topics, includes "oxidative stability" and various biofuel-related terms. This could indicate either nascent research areas that are beginning to gain attention or topics that are becoming less relevant in current research trends.

The implications of this thematic map are significant for the field of NIRS in biofuel production and analysis. It highlights the complex nature of NIRS applications, from fundamental processes like anaerobic digestion to advanced data analysis techniques. The prominence of motor themes suggests that current research is heavily focused on

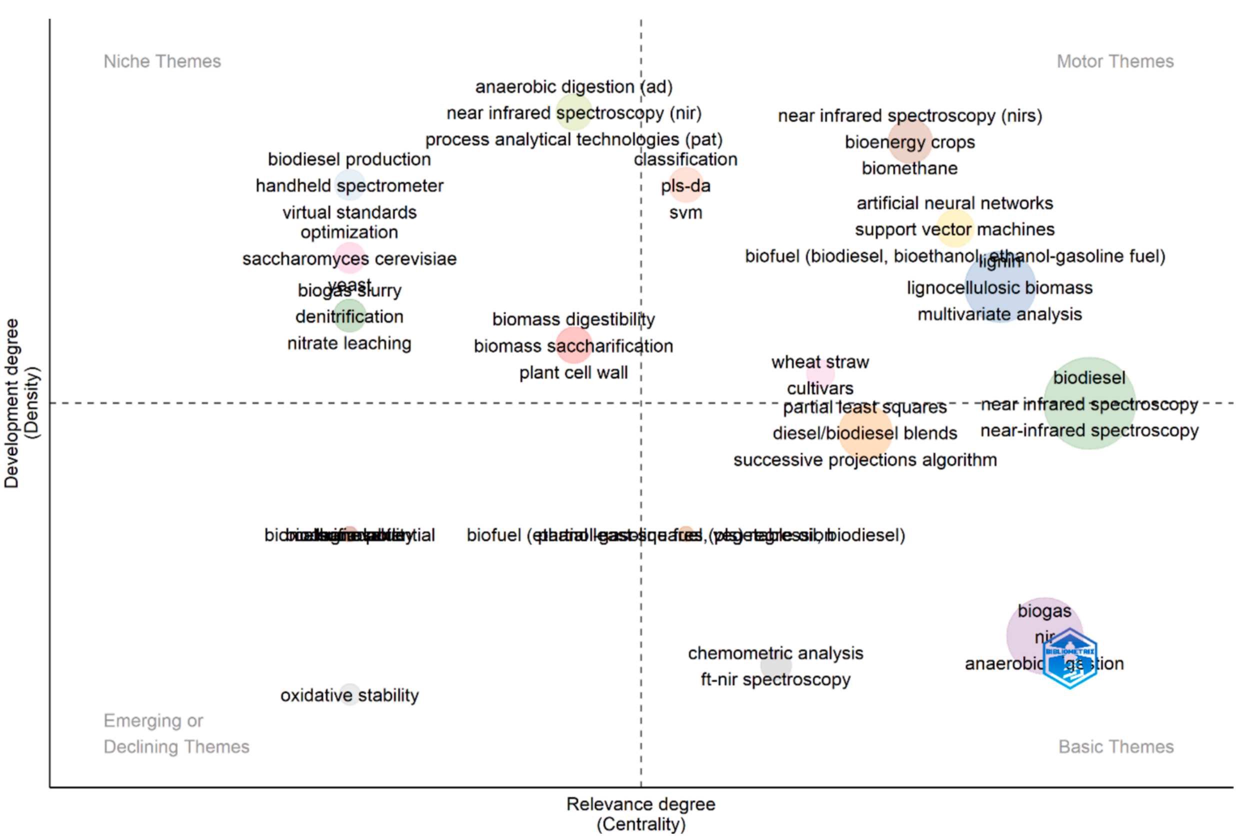

Fig. 10. Thematic map of keywords.

optimising NIRS for bioenergy crop analysis and biomethane production, with a strong emphasis on machine learning and multivariate analysis techniques. This implies a trend towards more sophisticated and accurate NIRS-based analytical methods in biofuel production. The presence of niche themes indicates specialised research areas that could become more central with further development or application. The map also reveals potential gaps or opportunities for future research, particularly in connecting niche themes with motor themes or in further developing emerging topics. This thematic analysis demonstrates the dynamic and evolving nature of NIRS applications in biofuel research, suggesting that the field is progressing towards more advanced, specialised, and integrated approaches to biofuel production and analysis using near-infrared spectroscopy.

The thematic evolution in Fig. 11 tracks how themes within the research area have developed over time. The earliest period (1996–2011) displays a foundation of core concepts, including NIRS, bioethanol, multivariate data analysis, bioenergy, and biogas. This indicates that the initial research was broadly focused on establishing NIRS as a viable analytical technique across various biofuel types. Moving into the 2012–2014 period, there is a noticeable shift toward more specific applications, with biodiesel emerging as a prominent theme alongside continued focus on NIRS. The appearance of cellulose and transesterification suggests a growing interest in feedstock analysis and specific production processes. The introduction of FT-NIR spectroscopy indicates advancements in instrumentation. The 2015–2018 period shows a significant expansion and diversification of research themes. Although near-infrared spectroscopy remains central, we see new topics such as lignin, Raman spectroscopy, and partial least squares (PLS) analysis emerge. This suggests a trend towards more sophisticated analytical techniques and a deeper focus on biomass composition. The reappearance of biogas and the introduction of biofuel as a distinct keyword indicates a continued broad interest across different biofuel types. The inclusion of sugarcane points to an increased focus on specific feedstocks. In the most recent period (2019–2023), we observe a consolidation of previous themes alongside the emergence of cutting-edge topics. NIRS remains prominent, suggesting their enduring importance. The appearance of machine learning as a key theme indicates the growing integration of advanced data analysis techniques with NIRS. Biomass emerges as a central keyword, indicating a shift towards a more comprehensive feedstock analysis. The continued presence of chemometrics and spectroscopy underscores the importance of advanced analytical methods in interpreting NIRS data.

The results demonstrate the persistent relevance of NIRS in biofuel research and production over nearly three decades, adapting to new challenges and opportunities in the field. Over time, the increasing diversity of keywords suggests a trend towards more specialised and sophisticated applications of NIRS, moving from general biofuel analysis to specific processes, feedstocks, and data analysis techniques. The emergence of machine learning in the latest period implies a future where NIRS data interpretation becomes more accurate and insightful, potentially leading to optimised biofuel production processes. The consistent presence of various biofuel types (bioethanol, biodiesel, biogas) throughout the timeline highlights the versatility of NIRS across different sectors of the biofuel industry. Furthermore, the evolution reveals a shift from focusing solely on end products to a more holistic approach that includes detailed feedstock analysis and process monitoring. This suggests that NIRS is increasingly critical in the entire biofuel production chain, from raw material characterisation to final product quality control. It is worth mentioning that the thematic evolution map highlights the dynamic nature of NIRS applications in biofuel research and suggests that future developments will likely involve even more advanced analytical techniques, possibly integrating real-time monitoring and artificial intelligence to optimise biofuel production processes further.

The factorial analysis displayed in Fig. 12 examines the underlying factors that contribute to the structure of the research area. The triangular distribution of terms indicates distinct clusters and relationships between various aspects of this field. The top left comprises terms like "enzymatic hydrolysis" and "ft nir" (Fourier transform near-infrared), suggesting a focus on fundamental spectroscopic techniques and biomass breakdown processes. The presence of "multivariate analysis" in this region implies using advanced statistical methods to interpret complex spectral data. Moving towards the center-left comprises a cluster of terms related to biomass feedstocks and processing, including "sugarcane," "biomass," "bioethanol," and "biorefinery." This grouping highlights the interconnectedness of raw materials, conversion processes, and end products in the biofuel industry. The lower left corner contains terms associated with analytical techniques and calibration, such as "infrared spectroscopy," "Raman spectroscopy," "chemometrics," and "calibration models." This suggests a strong emphasis on developing and refining measurement methodologies for biofuel analysis. The central region of the map features terms like "monitoring," "online monitoring," and "process monitoring," indicating a focus on real-time analysis and quality control in biofuel production. Moving toward the right side of the map, show terms related to specific biofuel processes and products, such as "biogas production," "volatile fatty acids," and "volatile solids." The presence of "pls regression" (partial least squares regression) in this area suggests the application of this statistical technique for quantitative analysis of these compounds.

The distribution of keywords demonstrates the multidisciplinary nature of NIRS applications in biofuels, from raw material characterisation to process monitoring and final product analysis. The map also reveals the importance of advanced analytical and statistical methods in extracting meaningful information from spectroscopic data. This comprehensive overview of the field suggests that NIRS, coupled with multivariate analysis techniques, is pivotal in optimising biofuel

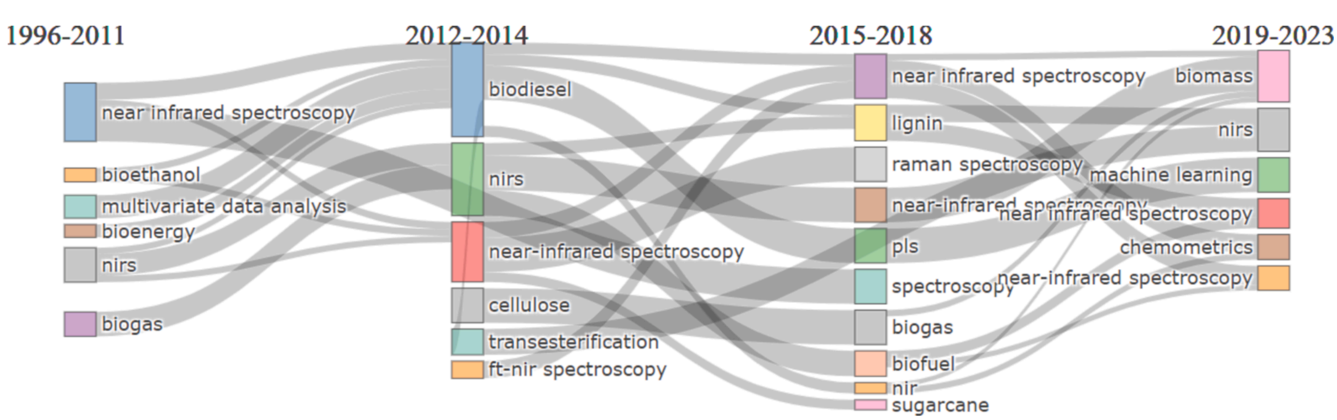

Fig. 11. Thematic evolution of keywords.

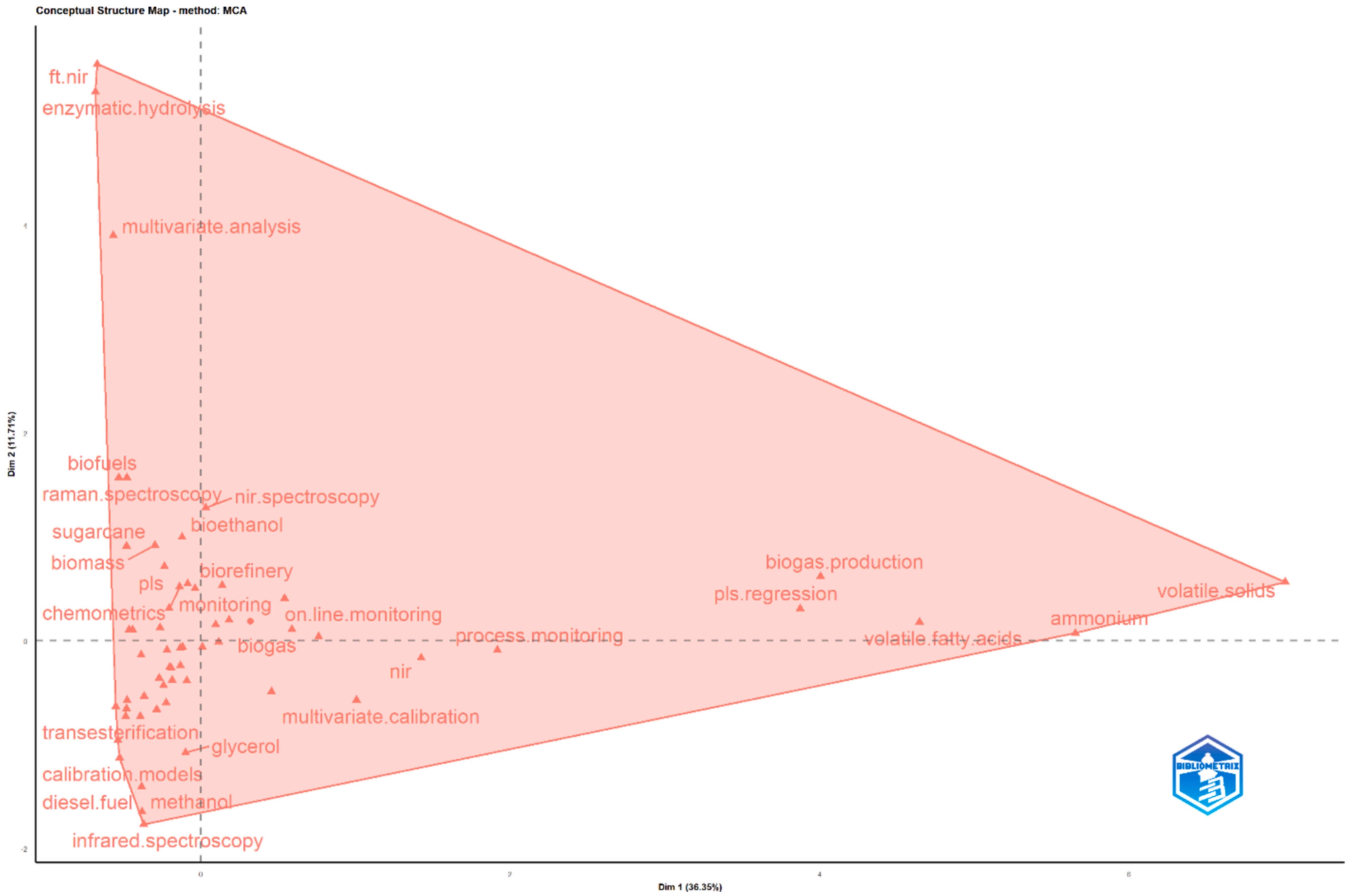

Fig. 12. Factorial analysis.

production processes, ensuring product quality, and advancing the overall efficiency of the bioenergy sector. The interconnectedness of terms across the map highlights the integrated approach required in biofuel research and development, where spectroscopic analysis serves as a linking technology between various stages of production and quality control.

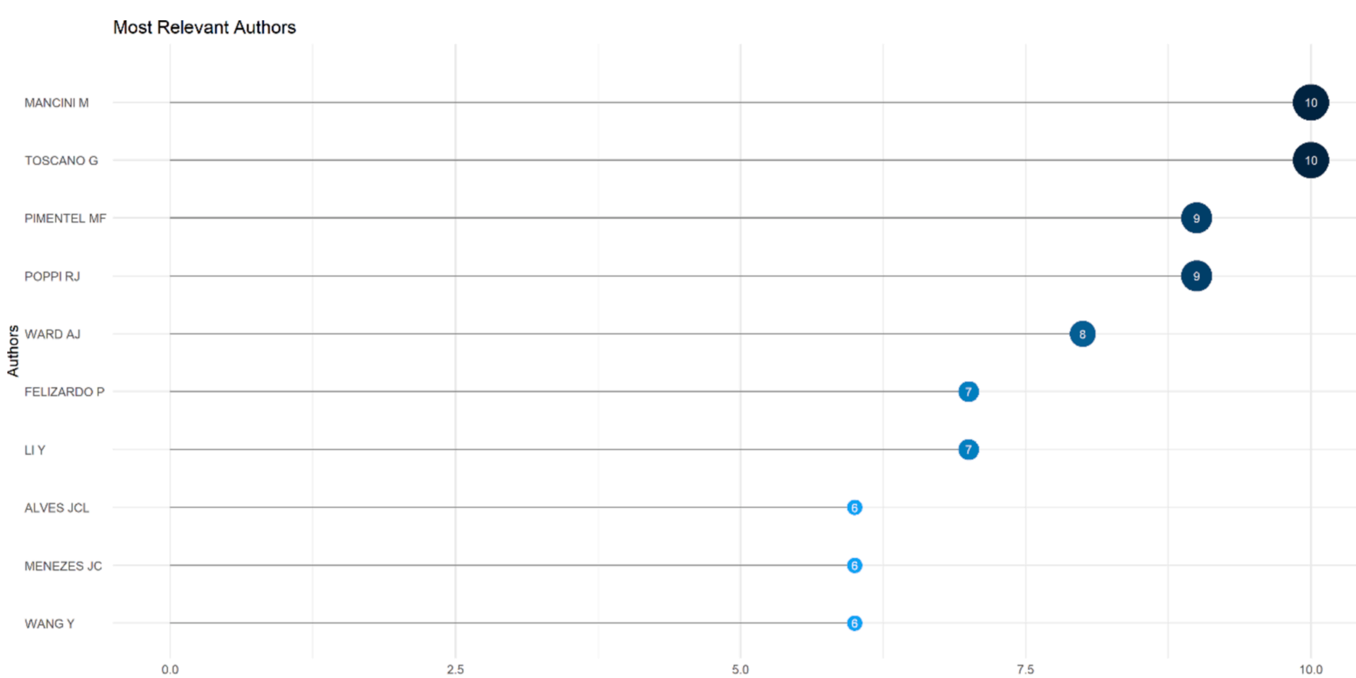

Fig. 13. Most relevant authors.

4.2. Most relevant authors, journals, institutions and citations analyses

Fig. 13 presents a ranking of the most relevant authors based on the number of published documents. It can be seen that MANCINI M and TOSCANO G are the top authors, with 10 published papers each indicating their significant contributions and expertise in this area. They are followed closely by PIMENTEL MF and POPPI RJ, each with 9 publications, suggesting a high level of involvement and research output in NIRS and biofuels. WARD AJ comes next with 8 documents, while FELIZARDO P and LI Y have each produced 7 publications on the subject. The list rounds out with ALVES JCL, MENEZES JC, and WANG Y, each contributing 6 documents to the field. This distribution of publications among top researchers implies a relatively concentrated field of expertise, with a small group of authors leading the way in research output. The proximity in publication numbers among these authors suggests a competitive and collaborative research environment where multiple experts actively contribute to advancing NIRS applications in biofuel research. The presence of international names (based on the diversity of surnames) indicates that this is a global research area, not limited to a single country or institution. This global interest highlights the importance of NIRS in biofuel analysis and production across different regions. The high productivity of these authors, with multiple publications each, demonstrates the ongoing relevance and rapid development of NIRS techniques in the biofuel sector. It also suggests that these researchers are likely leading research groups or collaborating extensively, given the volume of their output. The results highlight the key figures whose work should be followed for the latest advancements in the field, indicate potential collaboration opportunities for other researchers or industry partners, and provide a starting point for literature reviews on NIRS in biofuel applications. Furthermore, the concentration of expertise among these authors suggests that they may be influential in setting research directions and methodologies in this specialised field, potentially shaping future developments in biofuel analysis and production techniques using NIRS.

The authors' productivity based on Lotka's law is shown in Fig. 14. Lotka's law, a fundamental bibliometric principle, posits that the number of authors making n contributions is approximately $1/n^2$ of those making one contribution, with the frequency decreasing as the number of contributions increases [70]. The figure depicts this relationship, showing the percentage of authors on the y-axis against the number of documents written on the x-axis. The steep decline in the curve from left to right demonstrates that many authors (around 60–80 %) have contributed only one document to the field. This initial sharp drop is followed by a rapid decrease in the percentage of authors as the number of documents increases, with the curve flattening towards the right side of the graph. This pattern aligns closely with Lotka's law, indicating that a few highly productive authors are responsible for a disproportionate amount of the literature in this field.

The presence of two lines (solid and dashed) suggests comparing the observed data and the theoretical distribution predicted by Lotka's Law. The close alignment of these lines implies that the authorship pattern in NIRS for biofuel research closely follows the expected theoretical distribution. This has several implications for the field. For example, it suggests a concentration of expertise and research output among a select group of authors who may be considered leaders or key influencers. The large number of single-publication authors could indicate a broad but perhaps shallow interest in the topic, with many researchers contributing occasionally but not specialising in this area. The field may benefit from increased collaboration and mentorship to help more researchers move from single publications to sustained contributions. For newcomers to the field, identifying and following the work of the most productive authors could provide a pathway to the core knowledge and latest developments. Funding agencies and research institutions might use this information to target resources toward the most productive researchers or to develop strategies to increase sustained engagement from a broader base of contributors. The pattern may reflect the interdisciplinary nature of NIRS in biofuel research, attracting contributions from various related fields but with fewer researchers dedicating their entire focus to this specific intersection of topics.

Fig. 15 shows the most relevant sources for NIRS in biofuel production and analysis research dissemination. Bioresource Technology is the leading source with 29 documents, indicating its significant contribution to the field. This is followed by the Journal of Near Infrared Spectroscopy and Fuel, with 19 and 18 papers, respectively, highlighting their importance in disseminating research on NIRS applications in biofuels. Analytica Chimica Acta also shows substantial relevance with 13 papers. The prominence of these journals suggests a strong focus on both technological aspects and analytical chemistry in biofuel research using NIRS. Bioenergy-specific journals such as Bioenergy Research and Biomass and Bioenergy, both with 11 documents, emphasise the direct application of NIRS in bioenergy production processes. The presence of Biotechnology for Biofuels and Talanta, each with 10 papers, further emphasises the interdisciplinary nature of this research area, bridging biotechnology and analytical sciences. The distribution of documents

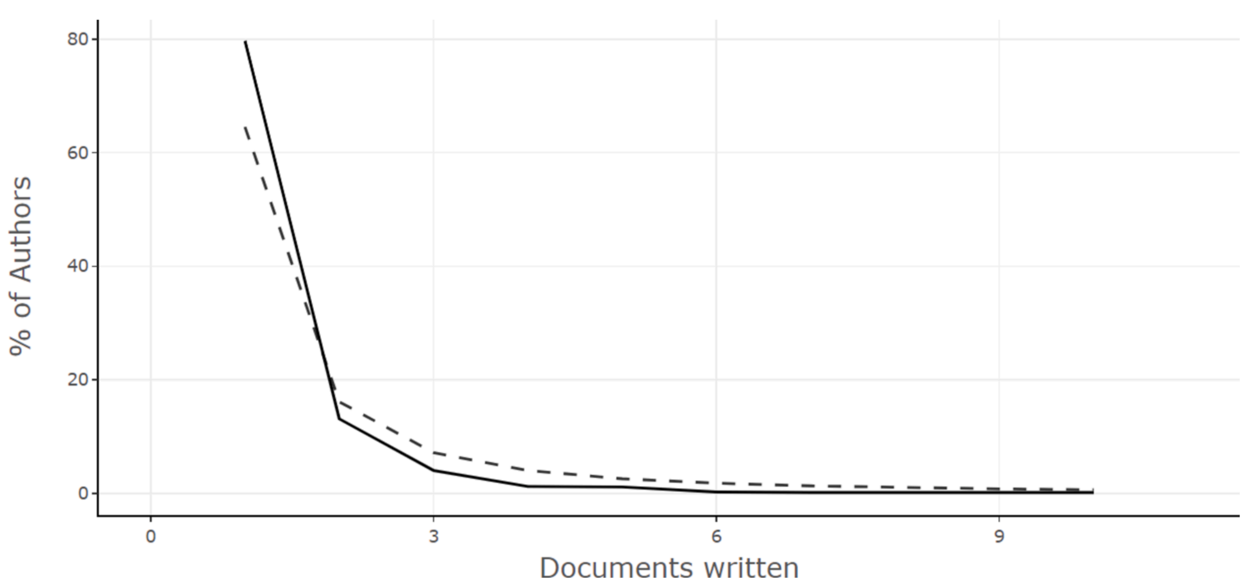

Fig. 14. Author Productivity through Lotka's Law.

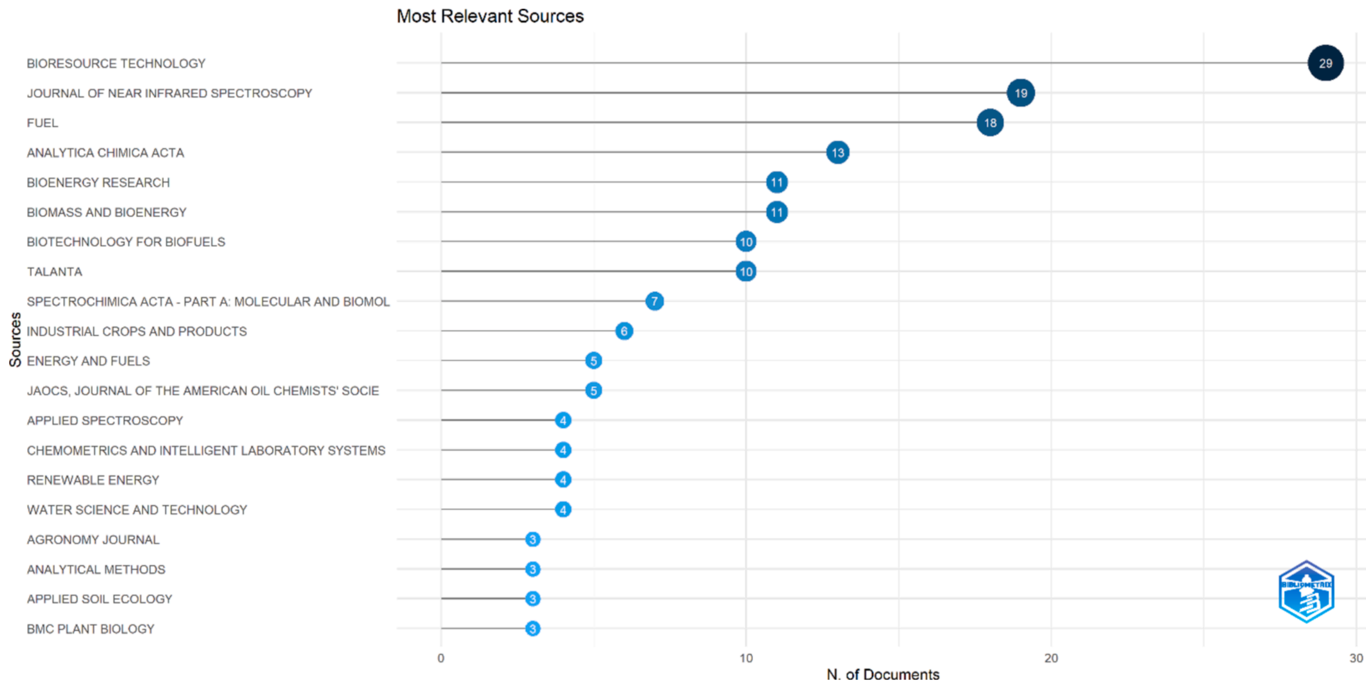

Fig. 15. Most relevant sources.

across various journals, including those focused on spectroscopy, chemistry, and agricultural sciences, demonstrates the multiple approaches required in biofuel research and development. This diverse spread of sources implies that NIRS is being utilised across different stages of biofuel production, from crop analysis (as evidenced by the inclusion of Agronomy Journal) to final product characterisation. The inclusion of journals like Water Science and Technology and BMC Plant Biology, although with fewer documents, suggests that NIRS applications extend to related areas such as environmental monitoring and plant science in the context of biofuel production.

Fig. 16 presents the most relevant affiliations researching NIRS in biofuel production and analysis. The figure reveals a global distribution of research efforts, with institutions from various countries contributing significantly to this field. Huazhong Agricultural University in China is the leading institution with 30 published articles, followed by Università Politecnica delle Marche in Italy with 26 articles. This suggests a strong focus on NIRS applications in biofuel research from Asian and European academic centres. The University of Campinas (UNICAMP) in Brazil ranks third with 21 articles, indicating substantial South American involvement in this research area. The presence of multiple Brazilian institutions (UNICAMP, Technical University of Lisbon, Universidade Federal de Pernambuco, and Universidade Estadual da Paraiba)

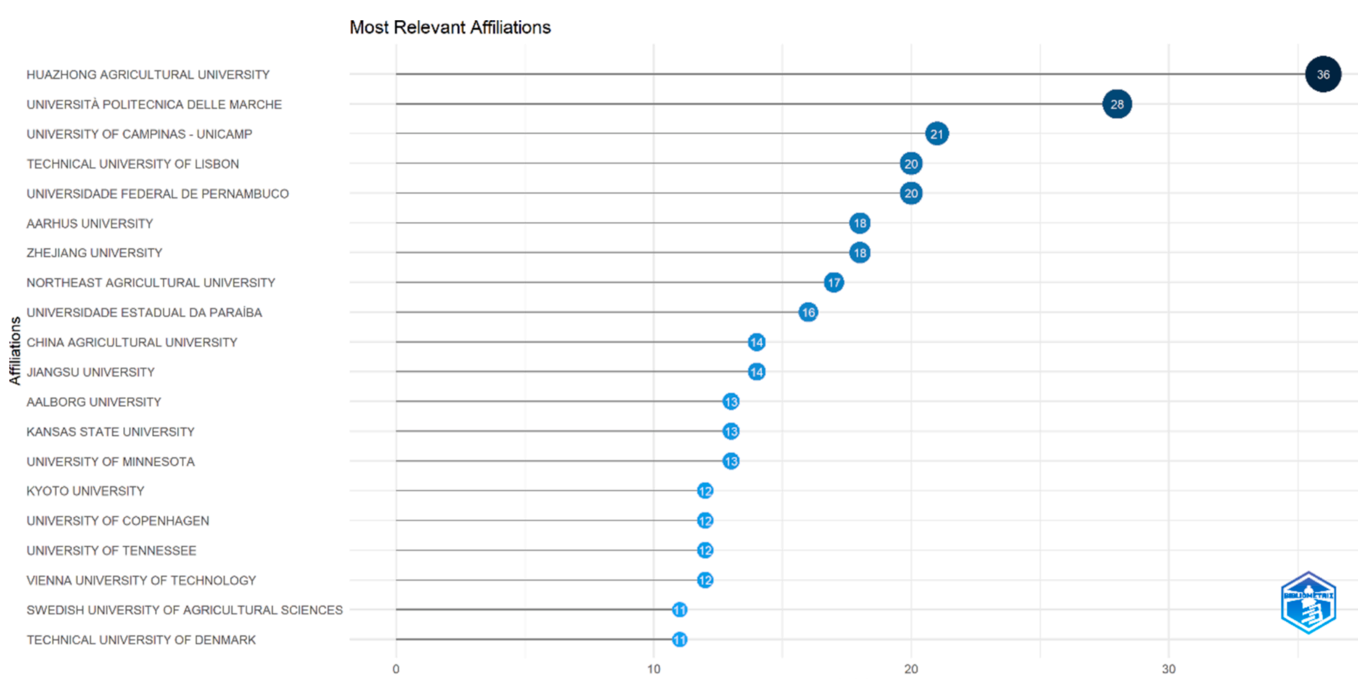

Fig. 16. Most relevant affiliations.

highlights Brazil's commitment to biofuel research, likely due to its significant bioethanol industry. European universities are well-represented, with institutions from Denmark (Aarhus University, Aalborg University, University of Copenhagen), Italy, Austria, and Sweden featuring prominently. This demonstrates Europe's strong interest in renewable energy technologies. Including several agricultural universities (Huazhong Agricultural, Northeast Agricultural, China Agricultural, and Swedish University of Agricultural Sciences) highlights the critical connection between agricultural research and biofuel production. United States institutions like Kansas State University and the University of Minnesota also appear with fewer publications than the top-ranked institutions. The diverse geographical spread of these affiliations implies a global recognition of the importance of NIRS in biofuel research, with contributions from North and South America, Europe, and Asia. This international collaboration and competition in the field suggest rapid advancements in NIRS biofuel analysis and production techniques. The prominence of agricultural and technical universities indicates a focus on the fundamental science and practical applications of NIRS in the biofuel sector. The relatively high number of publications from the top institutions (ranging from 30 to 13 articles) suggests that these are established research programmes with sustained interest and funding in this area. This concentration of research efforts will likely drive innovation in biofuel technologies, potentially leading to more efficient and sustainable biofuel production methods by applying NIRS techniques.

The most cited countries in Fig. 17 show significant disparities in both the total and average citations per article, suggesting varying levels of influence and research quality across different nations. The USA leads with 1,960 total citations and an average of 54.4 citations per article, indicating a strong and influential research output in this field. Brazil follows closely with 1,658 total citations but has a lower average citation per article (33.8). This suggests that while Brazilian research is prolific, it may not be as impactful as the USA on a per-article basis. Despite having only 1,165 total citations, Switzerland stands out with an exceptionally high average citation per article of 233. This suggests that while less frequent, Swiss research is of very high quality and impact, possibly indicating a focus on groundbreaking or highly innovative studies. Sweden and Denmark also show notable average citations per article, with 100.4 and 33.6, respectively, reflecting significant contributions to the field, although with lower total citations than the top countries. With 786 total citations and an average of 19.6 citations per article, China indicates a growing but still developing influence in NIRS research within biofuels. European countries like France, Germany, and Portugal demonstrate moderate citation figures, suggesting steady contributions to the field. In contrast, despite lower total citations, countries like Ireland and the Netherlands have high average citations per article, pointing to impactful research efforts. It can be seen that nations such as India and Israel, though lower in total citations, show relatively high average citations per article (53.5 and 24.7, respectively), highlighting the importance of their contributions. Conversely, countries like Japan and Italy, with low average citations per article (12.0 and 14.8), might suggest that their research is either more niche or less recognised in the broader academic community. The implications of these results point to a concentration of influential NIRS research within a few key countries, while others are emerging as significant contributors, which could impact global biofuel research collaboration and funding opportunities. This analysis emphasises the importance of quantity and quality in scientific research, with certain countries excelling in producing high-impact studies that shape the future of NIRS applications in biofuel production.

4.3. Analysis of the top cited papers

Table 1 displays the top 50 most cited papers available in the Scopus database. It can be observed that the most cited papers predominantly focus on using NIRS for quality control, composition analysis, and process monitoring in biodiesel production. Notably, studies by Balabin and Smirnov and Knothe stand out with high citation counts, indicating their significant impact on the field. These works emphasise the importance of NIRS in variable selection, transesterification monitoring, and blend level determination. The studies comprise various aspects of biofuel production, including biodiesel classification, moisture content prediction, and detection of adulterants. There is also a notable interest in applying NIRS to biomass feedstock analysis, as seen in Templeton et al. and Sanderson et al. papers. The consistent presence of papers focusing on multivariate analysis and chemometric techniques suggests the complexity of data interpretation in this field. Recent highly cited papers, such as Jin et al., indicate an ongoing interest in applying NIRS to emerging bioenergy crops like Miscanthus. The breadth of topics covered, from online monitoring of anaerobic digestion to rapid analysis of biomass composition, demonstrates the versatility of NIRS in biofuel applications.

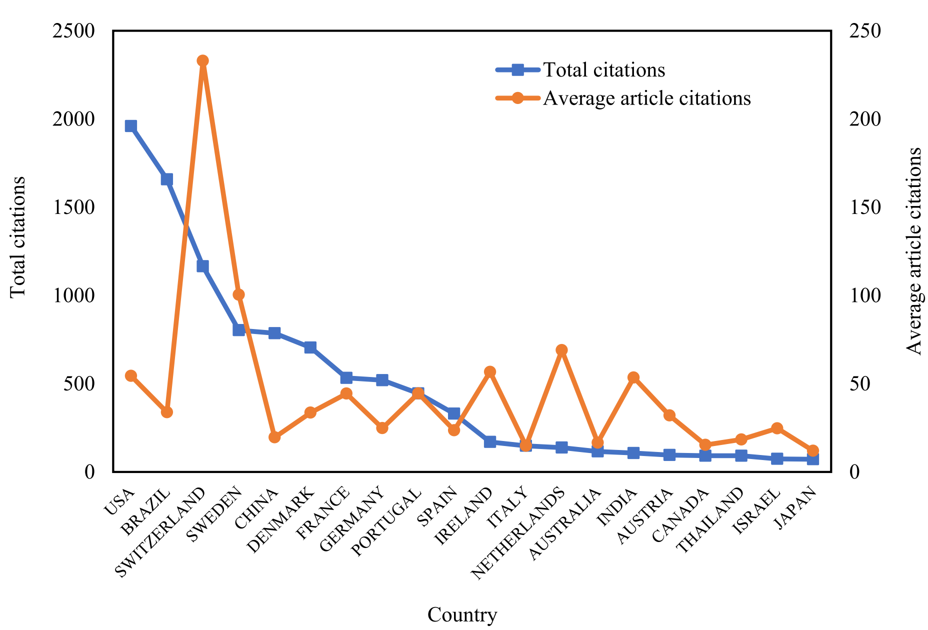

Fig. 17. Most cited countries.

Table 1
Top 50 most cited papers.

Author(s)	Title of paper	Total citations	Total citations per year
Balabin & Smirnov [71]	Variable selection in near-infrared spectroscopy: Benchmarking of feature selection methods on biodiesel data	366	26.14
Knothe [101]	Monitoring a progressing transesterification reaction by fiber-optic near infrared spectroscopy with correlation to 1H nuclear magnetic resonance spectroscopy	268	10.72
Balabin et al. [102]	Neural network (ANN) approach to biodiesel analysis: Analysis of biodiesel density, kinematic viscosity, methanol and water contents using near infrared (NIR) spectroscopy	222	15.86
Knothe [73]	Determining the blend level of mixtures of biodiesel with conventional diesel fuel by fiber-optic near-infrared spectroscopy and 1H nuclear magnetic resonance spectroscopy	174	7.25
Fernanda Pimentel et al. [75]	Determination of biodiesel content when blended with mineral diesel fuel using infrared spectroscopy and multivariate calibration	169	8.89
Knothe [72]	Rapid monitoring of transesterification and assessing biodiesel fuel quality by near-infrared spectroscopy using a fiber-optic probe	128	4.92
Templeton et al. [103]	Assessing corn stover composition and sources of variability via NIRS	127	7.94
Balabin & Safieva [104]	Biodiesel classification by base stock type (vegetable oil) using near infrared spectroscopy data	116	8.29
Menezes et al. [105]	Multivariate near infrared spectroscopy models for predicting methanol and water content in biodiesel	106	5.89
Sanderson et al. [86]	Compositional analysis of biomass feedstocks by near infrared reflectance spectroscopy	105	3.62
Lestander & Rhén [87]	Multivariate NIR spectroscopy models for moisture, ash and calorific content in biofuels using bi-orthogonal partial least squares regression	96	4.80
Fagan et al. [88]	Prediction of moisture, calorific value, ash and carbon content of two dedicated bioenergy crops using near-infrared spectroscopy	95	6.79
Lesteur et al. [81]	First step towards a fast analytical method for the determination of Biochemical Methane Potential of solid wastes by near infrared spectroscopy	91	6.50
Jin et al. [47]	Determination of hemicellulose, cellulose and lignin content using visible and near infrared spectroscopy in Miscanthus sinensis	90	11.25
Zhang [54]	Review on analysis of biodiesel with infrared spectroscopy	89	6.85
Holm-Nielsen et al. [97]	On-line near infrared monitoring of glycerol-boosted anaerobic digestion processes: Evaluation of process analytical technologies	86	5.06
Alves & Poppi [74]	Biodiesel content determination in diesel fuel blends using near infrared (NIR) spectroscopy and support vector machines (SVM)	84	7.00

Table 1 (*continued*)

Author(s)	Title of paper	Total citations	Total citations per year
Correia et al. [106]	Multivariate near infrared spectroscopy models for predicting the iodine value, CFPP, kinematic viscosity at 40 °C and density at 15 °C of biodiesel	82	4.82
Gaydou et al. [107]	Evaluation of multiblock NIR/MIR PLS predictive models to detect adulteration of diesel/biodiesel blends by vegetal oil	79	5.64
Ye et al. [108]	Fast classification and compositional analysis of cornstover fractions using Fourier transform near-infrared techniques	76	4.47
Correia et al. [76]	Multivariate near infrared spectroscopy models for predicting the methyl esters content in biodiesel	76	4.47
Ward et. al. [80]	Real time monitoring of a biogas digester with gas chromatography, near-infrared spectroscopy, and membrane-inlet mass spectrometry	74	5.29
Huang et al. [96]	A rapid and consistent near infrared spectroscopic assay for biomass enzymatic digestibility upon various physical and chemical pretreatments in Miscanthus	73	5.62
Reza et al. [90]	Hydrothermal carbonization (HTC): Near infrared spectroscopy and partial least-squares regression for determination of selective components in HTC solid and liquid products derived from maize silage	70	6.36
Lestander et al. [109]	NIR techniques create added values for the pellet and biofuel industry	66	4.13
Everard et al. [37]	Prediction of biomass gross calorific values using visible and near infrared spectroscopy	65	5.00
Payne & Wolfrum [89]	Rapid analysis of composition and reactivity in cellulosic biomass feedstocks with near-infrared spectroscopy	63	6.30
Liebmann et al. [98]	Determination of glucose and ethanol in bioethanol production by near infrared spectroscopy and chemometrics	63	3.94
de Oliveira et al. [110]	Application of correlation constrained multivariate curve resolution alternating least-squares methods for determination of compounds of interest in biodiesel blends using NIR and UV–visible spectroscopic data	62	5.64
Killner et al. [95]	A PLS regression model using NIR spectroscopy for on-line monitoring of the biodiesel production reaction	59	4.21
Correia et al. [77]	Portable near infrared spectroscopy applied to fuel quality control	58	8.29
Jacobi et al. [82]	Use of near infrared spectroscopy in monitoring of volatile fatty acids in anaerobic digestion	57	3.56
Vaknin et al. [111]	Predicting Jatropha curcas seed-oil content, oil composition and protein content using near-infrared spectroscopy—A quick and non-destructive method	55	3.93
Uva et al. [112]	Monitoring Biodiesel Fuel Quality by near Infrared Spectroscopy	55	3.06
Fernandes et al. [113]	Determination of biodiesel content in biodiesel/diesel blends using	54	3.86

(continued on next page)

Table 1 (*continued*)

Author(s)	Title of paper	Total citations	Total citations per year
	NIR and visible spectroscopy with variable selection		
Raju et al. [85]	Comparison of near infra-red spectroscopy, neutral detergent fibre assay and in-vitro organic matter digestibility assay for rapid determination of the biochemical methane potential of meadow grasses	53	3.79
Lomborg et al. [83]	Near infrared and acoustic chemometrics monitoring of volatile fatty acids and dry matter during co-digestion of manure and maize silage	53	3.31
Lomborg et al. [83]	Near-Infrared Reflectance Spectroscopy Prediction of Leaf and Mineral Concentrations in Alfalfa	52	2.48
Triolo et al. [114]	Near Infrared Reflectance Spectroscopy (NIRS) for rapid determination of biochemical methane potential of plant biomass	51	4.64
Filgueiras et al. [99]	Quantification of animal fat biodiesel in soybean biodiesel and B20 diesel blends using near infrared spectroscopy and synergy interval support vector regression	49	4.45
Pontes et al. [78]	Screening analysis to detect adulteration in diesel/biodiesel blends using near infrared spectrometry and multivariate classification	49	3.50
Gillespie et al. [115]	Prediction of biomass pellet quality indices using near infrared spectroscopy	48	4.80
Richard et al. [94]	On-line monitoring of the transesterification reaction between triglycerides and ethanol using near infrared spectroscopy combined with gas chromatography	48	3.43
Stockl & Lichti [84]	Near-infrared spectroscopy (NIRS) for a real time monitoring of the biogas process	47	6.71
Bruun et al. [91]	Prediction of the degradability and ash content of wheat straw from different cultivars using near infrared spectroscopy	46	3.07
Skvaril et al. [36]	Applications of near-infrared spectroscopy (NIRS) in biomass energy conversion processes: A review	45	5.63
Simeone et al. [92]	Near infrared spectroscopy determination of sucrose, glucose and fructose in sweet sorghum juice	45	5.63
de Lima et al. [100]	In-line monitoring of the transesterification reactions for biodiesel production using NIR spectroscopy	44	4.00
Xiao et al. [93]	NIR and Py-mbms coupled with multivariate data analysis as a high-throughput biomass characterization technique: a review	43	3.91
Pilar Dorado et al. [116]	Visible and NIR Spectroscopy to assess biodiesel quality: Determination of alcohol and glycerol traces	43	3.07
de Oliveira et al. [117]	Application of near infrared spectroscopy and multivariate control charts for monitoring biodiesel blends	43	2.69
a Silva Medeiros et al. [79]	Assessment oil composition and species discrimination of Brassicas seeds based on hyperspectral imaging and portable near infrared (NIR) spectroscopy tools and chemometrics	42	14.00

The papers in Table 1 show a comprehensive analysis of NIR spectroscopy and related spectroscopic techniques across multiple studies, revealing its versatility and effectiveness in biodiesel production, classification, and quality control. In view of this, Balabin and Smirnov [71] demonstrated that selecting the right feature selection methods could significantly improve the accuracy of biodiesel property predictions, enhancing broader spectroscopic applications. Knothe's studies [72,73] emphasised the rapidity and reliability of NIR, fibre-optic probes, and ^{1}H Nuclear Magnetic Resonance (NMR) spectroscopy for monitoring transesterification reactions and blend levels in biodiesel-diesel mixtures. Alves and Poppi [74] further advanced this by showing the superiority of support vector machine (SVM) models over partial least squares (PLS) for biodiesel content predictions, highlighting their potential for industry implementation.

Multiple studies, including those by Pimentel et al. [75] and Menezes & Correia [76], validated the application of multivariate calibration techniques like PLS and principal component analysis (PCA) for determining chemical components in biodiesel and detecting contaminants. Additionally, studies such as Correia et al. [77] and Pontes et al. [78] extended the utility of NIR in fuel quality control and adulteration detection, employing advanced chemometric models and pattern recognition techniques. The portability and accuracy of NIR techniques were further evidenced by da Silva Medeiros et al. [79], where NIR-Hyperspectral Imaging (NIR-HSI) excelled in classifying Brassica species and quantifying oil content and fatty acids. These collective findings emphasise the pivotal role of NIR and related spectroscopic methods in optimising biodiesel production, ensuring fuel quality, and enhancing industrial efficiency.

Studies on applying NIRS in anaerobic digestion and biochemical methane potential (BMP) assessment demonstrate its effectiveness as a rapid, non-invasive monitoring tool. Ward et al. [80] compared four monitoring methods for anaerobic digestion. The authors found NIRS effective for estimating volatile fatty acids but less precise in liquid phase quantifications. Lesteur et al. [81] showed that NIRS could accurately predict BMP in municipal solid waste, providing a faster alternative to traditional methods. Similarly, Jacobi et al. [82] validated NIRS for tracking key anaerobic digestion parameters like volatile fatty acids, propionic, and acetic acid, showing strong correlations for most parameters over long-term operation. Lomborg et al. [83] demonstrated the capability of NIRS for online monitoring of biogas processes, particularly for total and volatile solids, with good predictive accuracy. Stockl and Lichti [84] further supported NIRS' utility by successfully monitoring volatile fatty acids and total inorganic carbon with high precision. Raju et al. [85] extended these findings by using NIRS to predict BMP in meadow grasses and plant biomass, respectively, revealing moderate to high predictive success. These studies highlight NIRS' potential to streamline monitoring and prediction tasks in anaerobic digestion, making it a valuable tool in biogas production.

The use of NIRS in biomass feedstock and bioenergy research has shown promising results across various studies. Sanderson et al. [86] evaluated NIRS for predicting biomass chemical composition. The authors found that it was effective for constituents like extractives and lignin but less for others like mannose and galactose. Lestander and Rhén [87] demonstrated NIRS's high accuracy in predicting moisture, ash content, and calorific value in biofuels, supporting its utility for real-time biofuel characterisation. Fagan et al. [88] similarly found NIRS effective for predicting moisture and calorific value in Miscanthus and willow crops, though carbon and ash content predictions were less reliable. Jin et al. [47] explored VIS/NIR spectroscopy to quantify lignocellulosic components in Miscanthus sinensis, using advanced statistical techniques highlighting its applicability in rapid feedstock analysis. Payne and Wolfrum [89] applied NIRS to predict biomass composition and carbohydrate release, offering rapid screening for bioenergy applications, though with some uncertainties. Reza et al. [90] successfully used NIRS for fibre component prediction in hydrochar from maize silage, showing its capability in hydrothermal processes.

Studies like those by Everard et al. [37] and Bruun et al. [91] reinforced the value of NIRS for predicting gross calorific value and degradability in bioenergy crops and residues. Skvaril et al. [36] discussed the broader applications of NIRS in biomass conversion processes, emphasising its potential for process optimisation. Additionally, Simeone et al. [92] and Xiao et al. [93] highlighted NIRS's use in characterising sugar content in sweet sorghum and complementing other methods like pyrolysis-mass spectrometry for biomass analysis. NIRS offers a rapid, cost-effective method for monitoring biomass feedstock characterisation and biofuel process. However, its predictive accuracy can vary based on the component analyzed and the calibration models used.

The application of NIRS for monitoring biofuel production processes has been explored in multiple studies. For instance, Richard et al. [94] developed an NIRS-based method to monitor the transesterification reaction between high oleic sunflower oil and ethanol, achieving real-time reaction analysis without sample preparation. Killner et al. [95] extended this approach using PLS regression and proton nuclear magnetic resonance (^{1}H NMR) spectroscopy to monitor soybean oil transesterification with high predictive accuracy across different temperatures. Huang et al. [96] demonstrated NIRS's ability to predict enzymatic digestibility in Miscanthus, which is critical for biofuel processing, by developing robust calibration models with strong predictive performance. Holm-Nielsen et al. [97] integrated NIRS with Transflexive Embedded Near-Infrared Sensors (TENIRS) to monitor thermophilic anaerobic digestion systems, particularly focusing on biogas production with glycerol addition, and found NIRS highly effective for real-time process control. Liebmann et al. [98] applied NIRS to bioethanol production, developing models using genetic algorithms and PLS regression to accurately predict glucose and ethanol concentrations. Filgueiras et al. [99] compared different multivariate methods for determining animal fat content in biodiesel blends. The study found that the support vector regression model with variable selection outperformed traditional methods, demonstrating the benefits of advanced NIRS techniques in biofuel monitoring. De Lima et al. [100] focused on in-line monitoring of soybean oil transesterification, developing NIR-based calibration models for different glyceride components, further supporting the applicability of NIRS for process control in biodiesel production. It is noteworthy that studies collectively highlight the versatility of NIRS in biofuel applications, providing fast, accurate, and real-time monitoring capabilities across various stages of biofuel production.

5. Conclusion and direction for future research

Based on the comprehensive bibliometric analysis presented in this study, it is evident that NIRS has emerged as a pivotal analytical technique in biofuel production and analysis, demonstrating significant growth and diversification over the past three decades. From 1996 to 2023, the research area reveals a dynamic field characterised by increasing sophistication in methodologies, expanding applications across various biofuel types, and a growing emphasis on advanced data analysis techniques. The global distribution of research efforts, with notable contributions from Brazil, China, and the United States, highlights the international recognition of NIRS's potential in advancing sustainable energy solutions. The evolution of research themes, from basic spectroscopic applications to the integration of machine learning and real-time monitoring systems, indicates the field's maturation and adaptation to emerging biofuel production challenges. Key areas of focus have included biodiesel quality control, biomass characterisation, anaerobic digestion monitoring, and process optimisation across various stages of biofuel production. The consistent presence of chemometrics and multivariate analysis throughout the study period highlights the crucial role of advanced statistical methods in interpreting complex spectral data. The analysis of highly cited papers reveals the broad impact of NIRS applications, ranging from rapid feedstock analysis to real-time process monitoring and final product quality assurance. The emergence of portable and online NIRS systems, coupled with sophisticated data analysis techniques, points towards a future where real-time, in-situ monitoring becomes standard in biofuel production facilities. However, the concentration of expertise among a relatively small group of highly productive authors suggests a need for broader engagement and collaboration within the scientific community to further accelerate innovation in this field. The interdisciplinary nature of NIRS applications in biofuels, evident from the diverse range of journals and research institutions involved, indicates the potential for cross-pollination of ideas and methodologies across related fields such as analytical chemistry, process engineering, and agricultural sciences. As the global focus on renewable energy intensifies, the role of NIRS in optimising biofuel production processes, ensuring product quality, and improving overall efficiency is likely to become increasingly critical. Despite these advancements, there are several promising directions for future research. In view of this, the following directions are proposed for researchers to advance NIRS applications for biofuel production and analysis:

- Future research should focus on developing advanced neural network architectures and deep learning models tailored explicitly for NIRS data in biofuel applications; although machine learning techniques have been applied in NIRS data analysis, there is room for more sophisticated AI integration. This could lead to more accurate predictions and better handling of complex, non-linear relationships in spectral data.
- Even though some studies have explored online monitoring, there is a need for more robust, real-time, in-situ NIRS systems that can withstand the harsh conditions of industrial biofuel production. Further research should aim to develop miniaturised, durable NIRS sensors that can be directly integrated into production equipment, providing continuous, real-time data for process control.
- While traditional NIRS has been widely used, the potential of multispectral and hyperspectral NIR imaging in biofuel applications remains relatively unexplored. Future studies should investigate how these advanced imaging techniques can provide spatial information along with spectral data, potentially offering new insights into feedstock heterogeneity and reaction dynamics.
- Standardisation is needed given the variety of NIRS instruments and calibration methods used across different studies. Researchers could focus on developing robust calibration transfer methods that allow models developed on one instrument to be applied across different devices and even between laboratories, enhancing the reproducibility and comparability of results in further studies.
- Despite the fact that NIRS has shown great promise, combining it with other spectroscopic or analytical methods could provide even more comprehensive insights. Researchers could explore synergistic combinations of NIRS with techniques like Raman spectroscopy, mass spectrometry, or electrochemical sensors to create more powerful hybrid analytical systems in future studies.
- As new biofuels and production methods emerge, such as algal biofuels or synthetic biology approaches, future studies should investigate how NIRS can be adapted and optimised for these novel systems.

- Researchers could explore how NIRS data can be integrated into broader environmental impact and biofuel production life cycle assessments, potentially providing real-time sustainability metrics in further studies.

The scientific community can further enhance the capabilities of NIRS in biofuel applications, ultimately contributing to more efficient, sustainable, and economically viable biofuel production processes by pursuing these research directions.

CRediT authorship contribution statement

Flavio Odoi-Yorke: Writing – review & editing, Writing – original draft, Visualization, Validation, Software, Resources, Investigation, Data curation, Conceptualization, Methodology, Supervision. **Sandra Ama Kabiru:** Writing – review & editing, Writing – original draft, Visualization, Validation, Software, Methodology, Investigation, Conceptualization. **Rita Elsie Sanful:** Writing – review & editing, Writing – original draft, Visualization, Validation, Software, Methodology, Investigation, Data curation. **Gifty Serwaa Otoo:** Writing – review & editing, Writing – original draft, Visualization, Validation, Supervision. **Francis Padi Lamptey:** Writing – review & editing, Writing – original draft, Visualization, Supervision. **Agnes Abeley Abbey:** Writing – review & editing, Writing – original draft, Visualization. **Ephraim Bonah Agyekum:** Writing – review & editing, Writing – original draft, Supervision. **Ransford Opoku Darko:** Writing – review & editing, Writing – original draft, Supervision, Formal analysis, Project administration.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Data availability

Data will be made available on request.

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