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COMPREHENSIVE PHYSICOCHEMICAL CHARACTERIZATION OF ALGERIAN COAL POWDERS FOR THE ENGINEERING OF ADVANCED SUSTAINABLE MATERIALS

The object of the research is the intriguing and versatile material known as coal that attracted a lot of attention lately because of its potential use in a variety of fields, including cutting-edge building materials, environmental remediation methods, and creative energy storage solutions. This study presents an extensive characterization of Algerian natural coal powders, employing a multifaceted analytical approach that includes Scanning Electron Microscopy (SEM), X-ray Diffraction (XRD), Fourier Transform Infrared Spectroscopy (FTIR), and Raman Spectroscopy, to reveal their physicochemical properties including morphology, particle size distribution, crystalline structure, and functional groups.

The SEM analysis unveiled a heterogeneous morphology with a broad particle size distribution, indicative of the coal's complex structure. The XRD findings, refined using Rietveld analysis, distinguish Carbon (C) and Silicon Dioxide (SiO₂) as the primary phases, with crystallite sizes measuring 18.7539 nm for C and 16.6291 nm for SiO₂. These phases constitute 98.8 % and 12 % of the composition, respectively, while the presence of quartz underscores the coal's geological background and its thermal resilience.

Regarding the results of the FTIR spectroscopy, absorption peaks corresponding to various functional groups are highlighted, suggesting a rich organic and inorganic composition. Raman spectroscopy corroborates the presence of disordered and graphitic carbon structures, emphasizing the coal's potential for diverse applications. These findings underline the significance of Algerian coal powders for environmental remediation, energy storage, and advanced construction materials, contributing to the advancement of sustainable energy solutions.

Keywords: coal, SEM, XRD, FTIR, RAMAN, environmental remediation, energy storage, sustainable energy solutions.

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1. Introduction

Coal, a fascinating and multifunctional material, has garnered significant interest in recent years due to its potential utility across a wide range of applications [1], encompassing areas such as advanced construction materials [2], environmental remediation techniques [3], and innovative energy storage solutions [4]. This material's unique properties stem from its intricate structural and microstructural characteristics, which have been molded by an array of geological processes and environmental influences throughout its formation [5]. Delving into a detailed understanding of these features is indispensable for both optimizing the use of coal in its current applications and unlocking the potential for novel, groundbreaking applications in the future.

The appeal of coal lies in its remarkable physicochemical properties [6], which are attributed to its inherent

structural and microstructural makeup [7]. For instance, its inherent porosity and high specific surface area make it an excellent candidate for applications in adsorption and filtration [8], while its remarkable mechanical strength and durability [9] make it well suited for use in construction materials [2]. Furthermore, coal's electrochemical properties suggest promising potential for energy storage and conversion technologies [4].

These diverse applications are underpinned by the complex interplay of various geological processes, such as sedimentation, metamorphism, and diagenesis, as well as environmental factors like temperature, pressure, and the presence of various chemical species [6]. These factors collectively contribute to the formation of the intricate structural and microstructural features observed in coal, which include the arrangement of mineral phases, the presence of pores and voids, and the distribution of defects and impurities [10].

To fully harness the potential of this extraordinary material, it is imperative to develop a comprehensive understanding of the relationships between its structural and microstructural features and the resulting properties. This necessitates the use of cutting-edge analytical techniques that can provide detailed information at different length scales and resolution levels and the development of robust theoretical models that can accurately describe the material's behavior under various conditions. By acquiring a more profound understanding of the fundamental aspects of coal, researchers will be better equipped to optimize existing applications, as well as uncover new, transformative uses for this versatile material.

The aim of this research is detailed exploration of the properties of Algerian natural coal powders, a resource pivotal for its energy and material science applications. By integrating advanced analytical methodologies, including Scanning Electron Microscopy (SEM), X-ray Diffraction (XRD), Fourier Transform Infrared Spectroscopy (FTIR), and Raman Spectroscopy, we aim to unveil the nuanced morphological, structural, and chemical profiles of these powders. This comprehensive analysis is designed to reveal the unique characteristics of Algerian coal, assess its applicability across various industrial sectors, and evaluate the environmental implications of its use, thereby contributing to the advancement of coal technology and sustainable energy solutions. Enhanced by the inclusion of Raman Spectroscopy, our analytical arsenal provides a deeper insight into the molecular and crystallographic structure of these materials, enabling a more detailed understanding of their capabilities and limitations.

2. Materials and Methods

The characterization of coal powders was conducted using a multi-technique approach. Particle size and morphology were scrutinized using a Zeiss DSM 960A Scanning Electron Microscope (SEM) operating at 20 kilovolts. Phase composition analysis was performed through X-ray Diffraction (XRD) with an ADRX Benchtop diffractometer (manufactured in Canada) utilizing a Cu-K α radiation source ($\lambda_{Cu}=0.15418$ nm) and correlating with ICDD (PDF-2, 2014) and COD-2021 databases. Structural parameters were refined using HighScore Plus software (version 3.0.4), employing the Rietveld method. Concurrently, Fourier Transform Infrared Spectroscopy (FTIR), using an Agilent Cary 600 Series FTIR spectrometer from

Springvale, (made in Australia), covered a spectral range of 4000–600 cm^{-1} with baseline correction against a KBr spectrum for enhanced accuracy. Additionally, Raman spectroscopy was conducted using a Jasco NRS-500 spectrometer with a 532 nm laser (0.3 mW, 100X objective) to complement the morphological and compositional analysis of the coal powders.

3. Result and Discussion

3.1. Morphology analysis by Scanning Electron Microscopy (SEM). The comprehensive set of Scanning Electron Microscopy (SEM) images serves as the basis for a detailed morphological and particle size analysis [11] of natural coal samples extracted from Algerian deposits. The SEM micrographs, with their varied magnifications, as illustrated in Fig. 1, shows the heterogeneity inherent in the coal's particle size distribution, a key parameter influencing its application and performance in industrial processes.

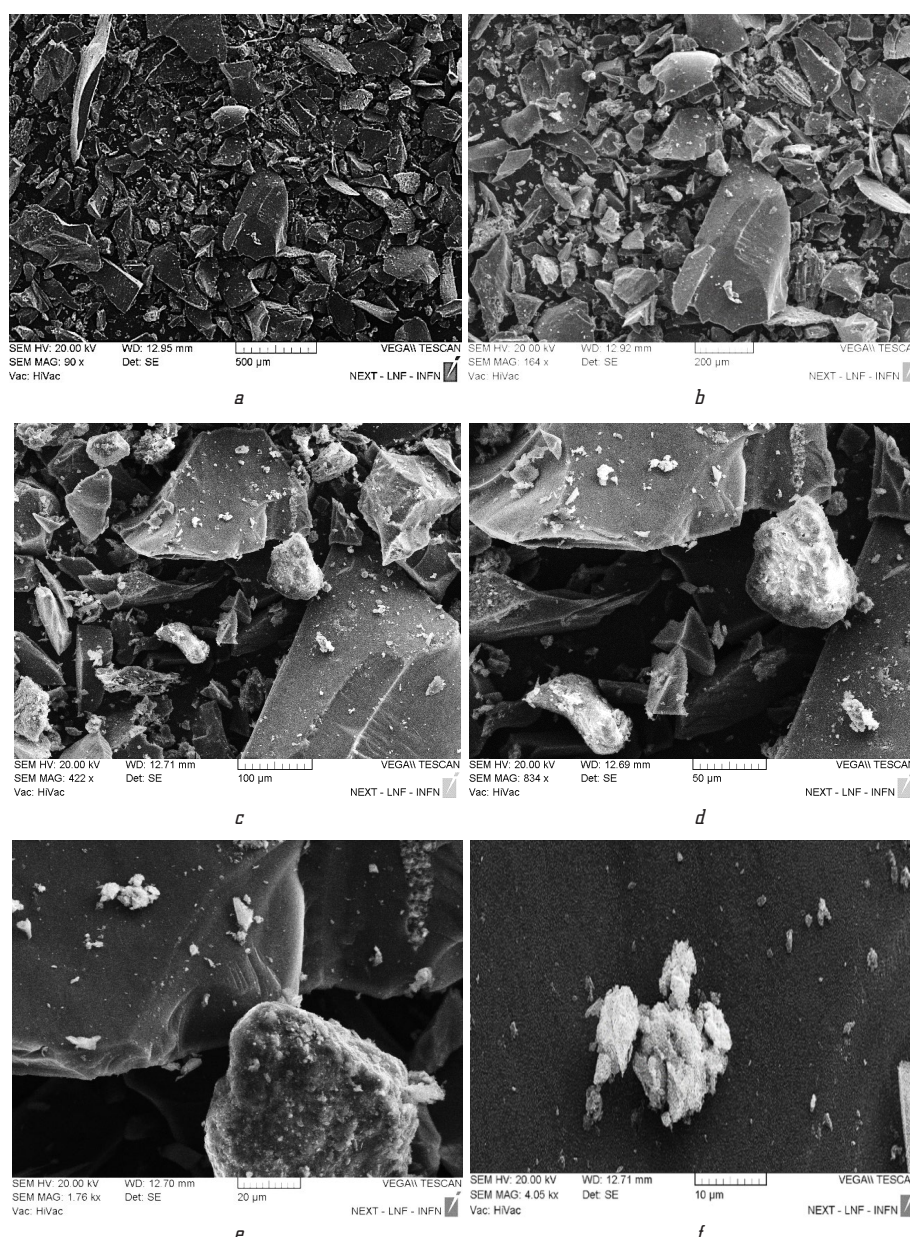


Fig. 1. SEM micrographs illustrating the heterogeneous morphology and particle size distribution of Algerian natural coal with different magnifications: *a* – 500 μm ; *b* – 200 μm ; *c* – 100 μm ; *d* – 50 μm ; *e* – 20 μm ; *f* – 10 μm

Higher magnification SEM images (Fig. 1, *d–f*), reveal the finer details of particle morphology, including edge angularity and surface texture, which are indicative of the mechanical fragmentation processes the coal has likely endured [12]. The observed angularity is typical of coal that has experienced significant maturation, suggesting a higher coal rank and brittleness, which are important factors in processes such as pulverization for combustion applications [13].

The particle size distribution, as derived from the SEM images, reveals a broad range from fine dust to larger aggregates. The presence of fine particles could be particularly relevant to the coal's combustion characteristics, as they tend to facilitate a faster burn rate due to their increased surface area to volume ratio [14]. On the other hand, the larger particles might affect the flow ability and handling characteristics of the coal, impacting operations such as storage and transportation [15].

Surface morphology, as evidenced by the roughness or smoothness of the particles, provides insight into the coal's maceral composition and the presence of mineral matter inclusions [16]. The diversity in texture observed across different particles could potentially affect the reactivity and coking properties of the coal, which are critical in applications like power generation and metallurgy [17].

Additionally, the high-magnification images illustrated in Fig. 1, *d–f*, indicate a level of porosity within the coal matrix, which is of significant interest for its implications on gas adsorption and storage capabilities [18]. This porosity is likely to affect the coal's behavior in gasification and liquefaction processes, as well as its efficiency in carbon capture and storage technologies [19].

The particle size analysis complements the qualitative observations from the SEM images with quantitative data, allowing for a more rigorous assessment of the coal's suitability for various applications. For instance, a narrow particle size distribution is often preferred for uniform combustion, whereas a broad distribution might be advantageous for certain metallurgical processes.

Comparative analysis with coal from other geographical locations suggests that the unique particle size distribution and morphology of Algerian coal could confer distinct advantages or present specific challenges, depending on the intended use [20]. The combination of SEM morphological analysis with particle size data provides a robust

foundation for predicting performance and guiding the optimization of coal utilization strategies.

3.2. Analysis by X-ray Diffraction (XRD) with Rietveld refinements. The comprehensive characterization of natural coal samples encompasses the elucidation of phase composition as well as structural and microstructural attributes. X-ray diffraction (XRD) peak broadening is instrumental in deducing the crystallite dimensions and the microstrain within the particulate matter. It is noteworthy that the broadening of the peak profile typically embodies a convolution of Gaussian and Lorentzian functions, representing microstrain-induced distortions and finite crystallite dimensions, respectively, as illustrated in Fig. 2 [21]. Furthermore, the accuracy of such measurements necessitates the consideration of instrumental contributions to peak broadening.

A suite of analytical models, including the Williamson-Hall, Halder-Wagner, Warren-Averbach, and Rietveld refinement [21], have been extensively employed for the quantitative determination of structural and microstructural parameters. Among these, the Rietveld refinement technique stands out for its robust full-pattern fitting capabilities, providing a detailed structural and microstructural analysis. This method has been lauded for its efficacy in both qualitative and quantitative phase assessment, especially in natural materials where overlapping diffraction peaks are prevalent [22]. The adoption of the Rietveld method has been validated through its successful application across a spectrum of material systems, as documented in the existing body of literature [22].

Fig. 2 presents a detailed examination of the refined X-ray diffraction (XRD) patterns of natural coal samples, illustrating the precision achieved through the refinement process. This figure serves as a testament to the meticulous analytical approach employed, showcasing the complex structural nuances of the coal samples under investigation.

The utilization of HighScore Plus software in the analytical examination of natural coal samples facilitated the substantiation of the existence of various phases, culminating in the identification of two distinct phases. Table 1 provides a detailed enumeration of the structural characteristics of the primary phases, which played a crucial role in the accurate fitting of the experimental XRD patterns associated with our samples.

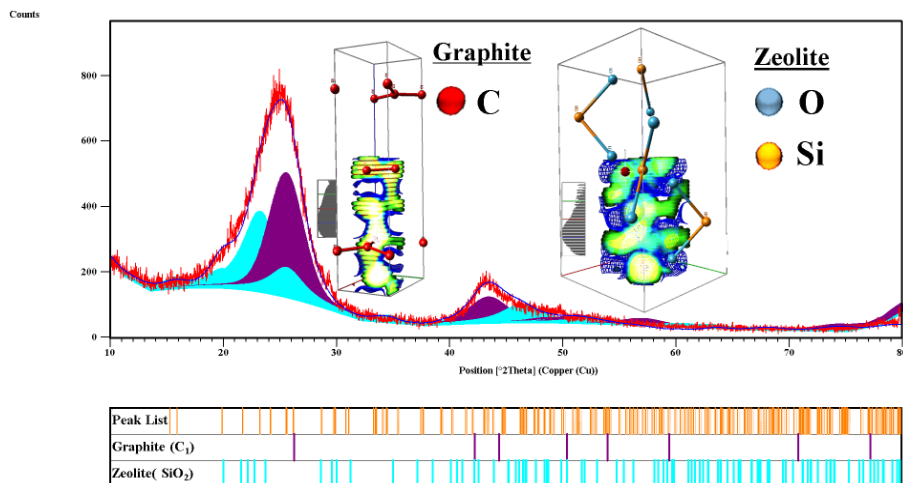


Fig. 2. Rietveld refinements of XRD patterns of Algerian natural coal

Table 1

Crystallographic parameters and proprieties of different phases used for the Rietveld refinements

Phases	C	SiO ₂
Chemical formula	C ₁	Si ₁ O ₂
Crystal system	Hexagonal	Monoclinic
Space group	P 63 m c	P 1 2/c 1
Cell parameters (<i>a</i> , <i>b</i> , <i>c</i>) in Å; (<i>α</i> , <i>β</i> , <i>γ</i>) in °	<i>a</i> =2.470; <i>b</i> =2.4700; <i>c</i> =6.7900	<i>a</i> =4.4003; <i>b</i> =4.4350; <i>c</i> =9.0400
	<i>α</i> =90; <i>β</i> =90; <i>γ</i> =120	<i>α</i> =90; <i>β</i> =114; <i>γ</i> =90
Volume (Å ³)	35.88	160.80
Density (g/cm ³)	2.16	2.48
Crystallite size (nm)	18.5739	16.6291
Microstrain (%)	5.0095	7.6772
Q Phase (%)	98.8	1.2
GOF	1.26	

Notes: C – Carbon; SiO₂ – Silicon dioxide

This incorporation of theoretical insights during the Rietveld Refinements is aimed at enhancing comparative analysis, especially given the widespread adoption of High-Score Plus software among researchers for the nuanced analysis of XRD patterns [23]. This approach underscores the software's utility in revealing complex phase compositions, thereby contributing to a deeper understanding of the material's structural intricacies.

The microstructural analysis of the natural coal samples was meticulously conducted through the determination of lattice parameters (*a*, *b*, *c*, *α*, *β*, *γ*, and unit cell volume in Å³), density (expressed in g/cm³), crystallite size (measured in nm), and microstrain (quantified as %). This analysis also included a quantitative phase analysis (expressed in %) and an assessment of the goodness of fit (GOF) parameter as part of the Rietveld refinement process. This method relies on the precise overlay of an experimental XRD pattern onto a simulated crystallographic model, aiming for optimal congruence, and is underpinned by sophisticated analytical functions. The extensive results from this analysis are systematically presented in Table 2, offering a comprehensive overview of the material's microstructural properties.

The Rietveld refinement of X-ray diffraction data for natural coal samples elucidates the microstructural composition, delineating the presence of two primary phases: Carbon (C) and Silicon Dioxide (SiO₂). This sophisticated analysis offers profound insights into the crystalline architecture and physicochemical properties of the coal, highlighting its significance for both industrial applications and environmental considerations.

For the carbon phase, the hexagonal lattice parameters, with identical *a* and *b* dimensions at 2.57508 Å and *c* at 7.25712 Å, along with angles *α* and *γ* at 90° and *β* at 120°, indicate a densely packed structure. This arrangement, reflected in a unit cell volume of 42.6751 Å³, is characteristic of the high strength and thermal conductivity typical of carbon materials [24]. The observed density of 1.91 g/cm³ is within the expected range for graphitic substances, suggesting a high degree of crystallinity [25].

The crystallite size, approximately 18.57 nm, and a microstrain of 5.0095 % suggest the presence of lattice imperfections, which are critical in influencing the material's catalytic and mechanical properties [9]. Dominating the phase composition at 98.8 %, the carbon phase underscores the sample's carbonaceous nature.

In contrast, the SiO₂ phase exhibits structural parameters suggestive of a less symmetrical framework, possibly indicating a triclinic or monoclinic system, as evidenced by a non-orthogonal *β* angle of 102° [26]. The larger unit cell volume of 289.5018 Å³ compared to carbon hints at a more open, possibly amorphous structure [27]. The density of 1.38 g/cm³ for SiO₂ and the crystallite size of 16.6291 nm, along with an elevated microstrain of 7.6772 %, indicate that this phase may contribute to internal stress, affecting the coal's thermal [28] and mechanical stability [29]. Despite constituting only 1.2 % of the phase composition, SiO₂'s role as a secondary phase could influence ash content and melting behavior during combustion [30].

The goodness of fit (GOF) value of 1.26 confirms the accuracy of the Rietveld refinement, ensuring a reliable match between observed and simulated XRD patterns [11]. This high level of accuracy in the analysis affirms the model's capability to accurately represent the coal's phase composition and microstructural details.

Table 2

Structural parameters of natural coal samples obtained from the Rietveld refinements

Samples	Natural coal	
Phases	C	SiO ₂
Cell parameters (<i>a</i> , <i>b</i> , <i>c</i>) in Å; (<i>α</i> , <i>β</i> , <i>γ</i>) in °	<i>a</i> =2.57508; <i>b</i> =2.57508; <i>c</i> =7.25712	<i>a</i> =6.34734; <i>b</i> =5.94149; <i>c</i> =7.84896
	<i>α</i> =90; <i>β</i> =90; <i>γ</i> =120	<i>α</i> =90; <i>β</i> =102; <i>γ</i> =90
Volume (Å ³)	42.6751	289.5018
Density (g/cm ³)	1.91	1.38
Crystallite Size (nm)	18.5739	16.6291
Microstrain (%)	5.0095	7.6772
Q Phase (%)	98.8	1.2
GOF	1.26	

3.3. Analysis by Fourier-Transform Infrared Spectroscopy (FTIR). Fourier Transform Infrared (FTIR) spectroscopy is revered as a cornerstone of analytical techniques, acclaimed for its remarkable sensitivity and the ability to discern the subtlest of compositional nuances within materials. Its finesse in detecting a wide array of bonding arrangements and functional moieties unfurls a vast spectrum of chemical intelligence [23]. Owing to its meticulous accuracy and comprehensive analytical scope, FTIR has become an essential tool in the domain of mineralogical investigations [23].

The robust nature of FTIR is demonstrated through its adeptness in distinguishing between crystalline and amorphous phases [31] as well as its capacity to unravel the complexities of organic molecular structures [32]. This versatile analytical approach facilitates a comprehensive elucidation of the materials under scrutiny.

Within the context of natural coal analysis, FTIR spectroscopy sheds light on the convoluted compositional attributes of such samples. Spectral data spanning the wavenumber interval from 600 to 4000 cm^{-1} reveal the molecular intricacies inherent to the sample. As illustrated in Fig. 3, the assortment of peaks and valleys mapped across the spectral landscape provides a detailed chemical and structural dossier of the coal specimen.

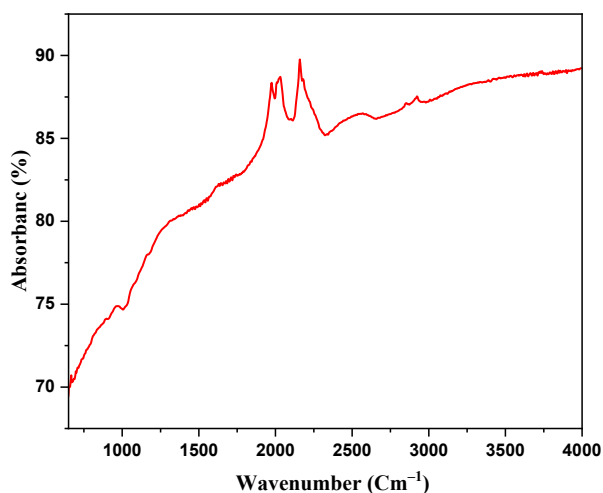


Fig. 3. FTIR spectral profile unveiling the molecular signature of Algerian natural coal

The Fourier Transform Infrared (FTIR) spectroscopy analysis of the Algerian natural coal stone samples revealed several characteristic absorption peaks, indicative of both organic and inorganic constituents. The spectra show prominent peaks at 667.19 cm^{-1} , 1975.49 cm^{-1} , 2031.40 cm^{-1} , 2158.13 cm^{-1} , 2568.14 cm^{-1} , and 2922.23 cm^{-1} , which can be attributed to various functional groups and bonding interactions within the coal matrix.

The absorption band at 667.19 cm^{-1} is typically related to the existence of CO_2 , which often accompanies coal deposits [5]. Alternatively, this band could denote specific carbon ring structures, reflecting the complex aromatic network within the coal [33]. At 1975.49 cm^{-1} and 2031.40 cm^{-1} , the peaks may be associated with metal-carbonyl complexes [34] or carbon-silicon (C-Si) stretching vibrations [35], respectively. Such bonds could result from trace minerals within the coal or from interactions with siliceous materials in the depositional environment [36].

The sharp peak observed at 2158.13 cm^{-1} corresponds to the stretching vibration of carbon monoxide (CO), which could be indicative of the presence of carbonyl groups bound to transition metals in the coal's inorganic fraction [37]. The presence of CO groups can be linked to low-temperature oxidation processes that the coal might have undergone [38]. The band at 2568.14 cm^{-1} may signify overtone or combination bands of fundamental vibrations, revealing the complex macromolecular structure of coal [39]. These overtones can provide indirect evidence of the coal's maturity and the types of chemical bonds present.

Finally, the peak at 2922.23 cm^{-1} aligns with the C-H stretching vibrations typical of aliphatic hydrocarbons [39]. This feature is a strong indicator of the coal's organic content, reflecting the aliphatic chains prevalent in the maceral components of the coal [39]. The presence of these specific peaks corresponds well with the expected composition of

coal and provides valuable insights into its structural and chemical properties. The combined organic and inorganic signatures corroborate the coal's complex formation history and subsequent alteration processes.

3.4. Analysis by Raman Spectroscopy. In Fig. 4, the results of employing Raman spectroscopy, renowned for its exceptional sensitivity and specificity, has been employed to probe the molecular intricacies of Algerian natural coal are showcased. Revealing distinctive vibrational modes that serve as indicators of its complex carbonaceous matrix. The analysis centered on pivotal frequencies 1350.16 cm^{-1} , 1586.01 cm^{-1} , 2936.75 cm^{-1} , and 2707.20 cm^{-1} each shedding light on different facets of the coal's structural and chemical constitution.

The presence of the D and G bands, at 1350.16 cm^{-1} and 1586.01 cm^{-1} respectively, unveils the dual nature of the carbon present within the coal [40]. The D band indicates a disordered or amorphous carbon structure, reflective of the coal's genesis and the myriad of physicochemical transformations it has undergone. This disordered carbon is a repository of the coal's historical narrative, encapsulating the diagenetic and catagenetic processes that have culminated in its present state. The G band, conversely, signals the presence of graphitic carbon, areas within the coal that exhibit a higher degree of crystalline order, and thus, thermal maturity [41].

Further spectral features at 2936.75 cm^{-1} and 2707.20 cm^{-1} enrich the characterization, hinting at the coal's organic makeup [42]. The former, associated with CH stretching vibrations, and the latter, possibly indicative of aromatic hydrocarbons, underscore the coal's organic complexity. This complexity is not merely a function of the coal's biological heritage but also of the transformative journey of organic matter from biomass to coal, underpinned by geological forces [42].

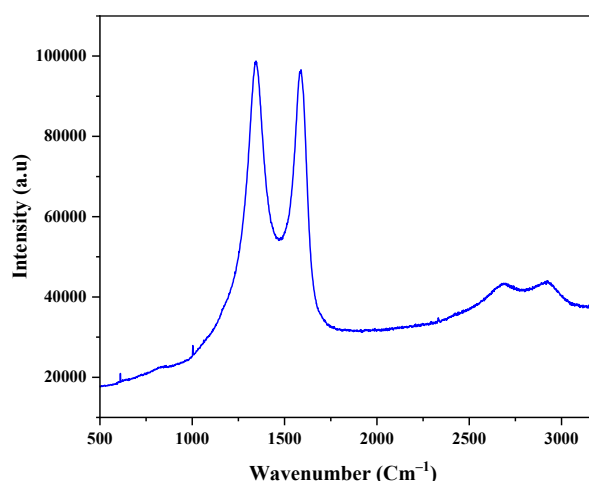


Fig. 4. Raman spectral analysis of Algerian natural coal highlighting key carbon structures

The implications of these findings extend beyond academic curiosity, influencing both energy production and materials science. The interplay between graphitic and amorphous carbon phases, as delineated by the spectral analysis, has a direct bearing on the coal's combustion efficiency and its potential as a precursor for advanced carbon materials. Moreover, the detailed insight into the coal's

hydrocarbon composition paves the way for novel applications, from enhancing energy storage solutions to innovating in the field of catalysis.

3.5. Limitations and perspectives on the results of the study. The findings from this study unveil the intricate internal microscopic properties of Algerian coal, marking a significant contribution to the field of nanotechnology. Notably, the discovery that the crystalline structures within the coal reach sizes of up to 16.6291 nm paves the way for numerous modern applications. This pioneering research serves as a cornerstone reference for Algerian coal, distinguished by its unprecedented focus on these microscopic details.

As a ground-breaking investigation into the microscopic properties of Algerian coal, this study emerges as a vital source of accurate information. It establishes a foundational reference for industries reliant on coal, such as the construction and energy sectors, highlighting its utility in enhancing industrial practices and material selection processes.

This research offers a robust framework for comparative studies and the integration of its novel findings with existing knowledge. The detailed insights into Algerian coal's properties afford an opportunity for international industries to reconsider and potentially augment their use of coal, based on the unique advantages identified. The potential for adopting Algerian coal in various countries is underscored by the study's applicability, encouraging a reevaluation of coal's role in global industrial applications.

The deployment of this research is guided by professional ethics, emphasizing the importance of crediting sources and maintaining integrity in information dissemination. The absence of specific restrictions further facilitates the broad application of these findings, provided standard ethical practices are adhered to.

Future inquiries will extend beyond the initial scope, exploring Algerian coal's diverse physical, chemical, and mechanical properties. This expansion is poised to uncover innovative applications, enhance existing technologies, and assess the material's sustainability and socio-economic impact, paving the way for transformative advancements in related fields.

4. Conclusions

In this study, we meticulously analyzed the physicochemical properties of Algerian natural coal powders, employing SEM, XRD, FTIR, and Raman Spectroscopy to elucidate their morphology, particle size distribution, crystalline structure, and functional groups. The SEM analysis revealed a heterogeneous morphology with a broad particle size distribution, highlighting the potential for diverse applications. The XRD results identified quartz as a significant mineral phase, indicative of the coal's geological history and its implications for thermal stability. Specifically, XRD results enhanced with Rietveld refinement identified Carbon (C) and Silicon Dioxide (SiO₂) as predominant phases with crystallite sizes of 18.5739 nm for C and 16.6291 nm for SiO₂, respectively, and phase quantities of 98.8 % for C and 12 % for SiO₂. FTIR spectroscopy highlighted the presence of functional groups typical of lignocellulosic materials, such as hydroxyl and carbonyl groups, suggesting avenues for chemical modification and utilization in polymer composites.

The absorption peaks at specific wavenumbers, such as 667.19 cm⁻¹, 1975.49 cm⁻¹, 2031.40 cm⁻¹, 2158.13 cm⁻¹,

2568.14 cm⁻¹, and 2922.23 cm⁻¹, highlighted a rich organic and inorganic composition. Raman spectroscopy further confirmed the amorphous nature of the carbon structures, aligning with the potential for high surface reactivity and adsorption capabilities. It revealed the presence of disordered and graphitic carbon structures, emphasizing the coal's potential for diverse applications. These multifaceted characteristics underscore the Algerian natural coal powders' applicability in environmental remediation, energy storage, and construction material innovations.

The study not only contributes to the expanding knowledge of natural coal resources but also opens new pathways for leveraging these materials in sustainable technologies. Future work will focus on enhancing the functionalization of these coal powders for specific industrial applications, aiming to optimize their performance and environmental benefits.

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Conflict of interest

The authors declare that they have no conflict of interest concerning this research, whether financial, personal, authorship, or otherwise, that could affect the study and its results presented in this paper.

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Data availability

The paper has no associated data.

Use of artificial intelligence

The authors confirm that they did not use artificial intelligence technologies when creating the current work.

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