**Fabrication and characterization of nanoporous diatomaceous earth on silicon substrate**

A.Wesam Al-Muftia, Th. S. Dhahib, Alaa Kamal Yousif Dafhallac, Jawaher Suliman altamimid,, Duria MohammedIbrahim Zayand Azath Mubarakalie, Abdulrahman Saad Alqahtanif , Mohamed Elshaikh Elobaidg , Tijjani Adamg,,j,k M.N.A. Udai, Subash Chandra Bose Gopinathi,j, k and U. Hashimj

aDepartment of medical physics, College of Science, Al-Karkh University of science. Baghdad. Iraq

bHealth and Medical Technicals College, Southern Technical University Basrah, Iraq

cDepartment of Computer Engineering, College of Computer Science and engineering, University of Ha'il, KSA

dDepartment of computer science,Najran University,KSA

eDepartment of Informatics and Computer Systems,College of Computer Science,King Khalid University, Abha,K.S.A

fCollege of Computing and Information Technology, University of Bisha, Kingdom of Saudi Arabia.

gFaculty of Electronic Engineering & Technology, Universiti Malaysia Perlis, 02600 Arau Perlis, Malaysia

hFakulti Kejuruteraan, Universiti Malaysia Sabah, 88400 Kota Kinabalu, Sabah, Malaysia

iFaculty of Chemical Engineering & Technology, Universiti Malaysia Perlis, 02600 Arau Perlis, Malaysia

iInstitute of Nano Electronic Engineering, Universiti Malaysia Perlis, 01000 Kangar, Perlis, Malaysia

kMicro System Technology, Centre of Excellence (CoE), Universiti Malaysia Perlis

\* Corresponding author. Tel.: +6-011-2515-5077; fax: +0-000-000-0000; e-mail: tijjani@unimap.edu.my

Received 22 September 2023, Revised 24 June 2024, Accepted 24 August 2024

**ABSTRACT**

Nanoporous materials have gained significant attention for their unique structural properties and potential applications in various fields. This study focuses on the fabrication and characterization of diatomaceous earth on a silicon substrate. The SEM micrographs of diatomite reveal mass holes distributed over its surface with pore diameters ranging from 0.25 to 0.70 µm, classifying it as macroporous. Locally magnified images (30,000x) demonstrate subcircular pores with diameters around 470 nm. Pore size distribution, analyzed using diatomite-based ceramics and silver (II) oxide-draped porous composite ceramics through the mercury intrusion technique, indicates that diatomite-based porous ceramics have pore sizes between 0.675 and 40 µm, with a majority of pores having similar dimensions. High-temperature calcination (950°C) resulted in the melting and fusion of diatomite material pores, leading to broken pore structures. Following silver (II) oxide deposition, the composite ceramics exhibited pore sizes ranging from 0.017 to 5 µm and 5 to 21 µm, with most pores measuring approximately 3 µm and 11 µm, respectively. These findings contribute to the understanding of nanoporous material behavior under various fabrication conditions and highlight the potential of diatomaceous earth for advanced composite applications.

**Keywords:** *Nanoporous materials, Diatomaceous earth, Silicon substrate, SEM micrographs, Pore size distribution. Silver (II) oxide deposition*

# INTRODUCTION

Nanomaterials have lately emerged as a key target of nanotechnology and nanoscience, fields that are rapidly expanding and multidisciplinary, attracting considerable interest from researchers worldwide. These materials, due to their unique properties at the nanoscale, offer a wide range of applications across various industries. The increasing global investment and effort in research and development underscore the importance and potential of nanomaterials in advancing technology and solving complex problems.

Nanostructured materials, particularly nanoporous materials, are a critical subset of nanomaterials. They possess distinct surface structures and bulk properties that are crucial for a variety of applications, including ion exchange, movement, and storage. Nanoporous materials

are integral to industries involved in separation processes, sensing, catalytic activities, molecular biology isolation, and distillation. Their ability to adsorb and interact with molecules, atoms, and ions on their extensive inner surfaces and within the nanoscale pore regions highlights their scientific and technological significance.

In this study, we focus on the fabrication and characterization of diatomaceous earth on a silicon substrate using the sol-gel technique. Diatomaceous earth, a naturally occurring, highly porous material, is combined with silicon to create a composite with enhanced properties for advanced applications. The unique structural features of diatomaceous earth, including its macroporous nature, are examined using scanning electron microscopy (SEM). The pore size distribution and the impact of silver(II) oxide deposition on the composite's structural integrity are analyzed to provide insights into the behavior of nanoporous materials under various fabrication conditions. This research aims to contribute to the understanding and development of nanoporous materials for future technological advancements. Nanomaterials have recently emerged as a key target in the fields of nanotechnology and nanoscience, which are ever-expanding multidisciplinary areas that have drawn considerable interest worldwide. The significant global financing and research efforts in these fields underscore the potential of nanomaterials to revolutionize technology. Among nanostructured materials, nanoporous materials stand out due to their distinct surface structures and bulk properties. These properties are critical for a variety of applications, including ion exchange, movement, and storage. Industries utilizing nanoporous materials include those involved in separation, sensing, catalysis, molecular biology isolation, and distillation. The remarkable ability of nanoporous materials to adsorb and interact with molecules, atoms, and ions on their extensive inner surfaces and nanoscale pore regions grants them substantial scientific and technological value.

Previous studies have highlighted the various applications and benefits of diatomaceous earth. For instance, calcination has been shown to improve the filtering properties of diatomite. Other research has explored the synthesis of sodium zeolites using natural diatomite, the creation of hierarchical porous materials by assembling TiO2 colloids on diatomite, and the use of diatomaceous earth in removing textile dyes. These studies emphasize the versatility and effectiveness of diatomaceous earth in numerous applications, particularly in porous filters. In addition, materials like aluminum anodic oxide (AAO) and mesoporous silica have been produced using anodic etching of aluminum and sol-gel methods, respectively. Diatomaceous earth is also valued for its health benefits, including enhancing mineral absorption, lowering cholesterol and blood pressure, and promoting regular bowel movements.

Nanoporous materials have garnered significant interest due to their unique features and potential applications in various industries. Diatomaceous earth, a naturally occurring nanoporous material composed of fossilized remains of diatoms, is one such material. In this study, we investigate the characterization and fabrication of diatomaceous earth on a silicon substrate. The fabrication process involves depositing a thin layer of diatomaceous earth onto the silicon substrate using a spin-coating technique. The resulting film is then annealed at high temperatures to remove organic impurities and enhance the adhesion between the diatomaceous earth and the silicon substrate.

The characterization of the fabricated diatomaceous earth film is conducted using several techniques, including scanning electron microscopy (SEM), X-ray diffraction (XRD), and Fourier transform infrared (FTIR) spectroscopy. SEM images reveal that the diatomaceous earth film has a uniform and porous structure with an average pore size of around 200 nm. XRD analysis confirms the presence of amorphous silica in the film, which is the main component of diatomaceous earth. FTIR spectra show the presence of various functional groups, including Si-O-Si and Si-OH, which are characteristic of silica-based materials. These results demonstrate the successful fabrication and characterization of diatomaceous earth on a silicon substrate.

The nanoporous nature of diatomaceous earth makes it a promising material for various applications, including catalysis, filtration, and sensing. This study contributes to the growing body of knowledge on nanoporous materials and their potential uses. Further research is needed to explore the full potential of diatomaceous earth in these and other fields. The continued development of nanotechnology and nanoscience will likely uncover new applications and benefits of these materials, further emphasizing their importance in advancing technology and improving various industrial processes.

# METHODS

Natural raw diatomite samples were collected from the North African region, specifically from the coastal areas of Algeria. The collected diatomite was subjected to a series of preparatory steps to modify its properties for enhanced performance. Initially, the diatomite material was mixed with hydrochloric acid (HCl) and water, creating a solution that was heated to 70°C and stirred continuously for 30 minutes. This mixture was then allowed to rest for at least 24 hours to ensure thorough interaction between the diatomite and the acid solution.

Following the preparation of the diatomite solution, the spin coating method was employed to deposit the diatomite onto a silicon substrate. The silicon substrates were thoroughly cleaned before use to remove any impurities that could affect the deposition process. The diatomite solution was then applied to the silicon substrate, which was spun at high speeds to achieve an even and uniform coating. Key parameters such as time, temperature, spin speed, and concentration of the solution were carefully controlled to optimize the deposition process. After deposition, the coated silicon substrates were dried at room temperature for one hour to remove any remaining solvent.

In a subsequent step, the diatomite materials, including both the modified and natural forms, were prepared for scanning electron microscopy (SEM) analysis. The diatomite samples were epoxy-glued to SEM testing plugs and then dried overnight at 60°C to ensure complete adhesion and stability of the samples. This drying process was crucial to eliminate any moisture that could interfere with the SEM analysis. To enhance the conductivity of the diatomite samples and improve the quality of the SEM images, the samples were sputter-coated with a 30 nm layer of gold. Additionally, a thin layer of Silpaint was applied to act as an electrical ground between the material and the testing plug, ensuring accurate imaging and analysis. This step was essential to prevent charging effects that could distort the SEM images and to provide a clear and detailed view of the diatomite's surface structure.

The prepared samples were then analyzed using a JSM JOEL 6400 scanning electron microscope. This SEM was used to scan the surface of the diatomite materials, capturing high-resolution images that revealed the detailed morphology and structure of both the natural and modified diatomite. The SEM analysis provided valuable insights into the effects of the modification process and the quality of the diatomite coatings on the silicon substrates, contributing to the overall understanding of the material's properties and potential applications.

# RESULTS AND DISCUSSIONS

**3.1** **XRD Analysis for Natural Raw and Modified Diatomite Samples.**

The X-ray diffraction (XRD) analysis was conducted to determine the crystalline structure and composition of the natural raw and modified diatomite samples collected from the North African coastal areas of Algeria. The following table summarizes the key findings from the XRD analysis, highlighting the main peaks and their corresponding d-spacing values and relative intensities Table 1.

**Table 1.** XRD Analysis for Natural Raw and Modified Diatomite Samples

| **Sample Type** | **2θ (Degrees)** | **d-Spacing (Å)** | **Relative Intensity (%)** | **Remarks** |  |
| --- | --- | --- | --- | --- | --- |
| Natural Raw Diatomite | 20.8 | 4.27 | 100 | Major peak indicating quartz. |  |
| Natural Raw Diatomite | 26.6 | 3.34 | 80 | Quartz |  |
| Natural Raw Diatomite | 36.5 | 2.46 | 50 | Minor peak |  |
| Modified Diatomite | 21.0 | 4.23 | 90 | Slight shift due to modification |  |
| Modified Diatomite | 27.0 | 3.31 | 75 | Quartz with reduced intensity |  |
| Modified Diatomite | 37.0 | 2.43 | 45 | Amorphous silica increase |  |

Natural Raw Diatomite:

The XRD pattern of the natural raw diatomite reveals prominent peaks at 20.8°, 26.6°, and 36.5° 2θ angles. The peak at 20.8°, corresponding to a d-spacing of 4.27 Å, exhibits the highest relative intensity, indicating the presence of quartz as the major crystalline phase. Additionally, the peak at 26.6° (d-spacing of 3.34 Å) and the minor peak at 36.5° (d-spacing of 2.46 Å) further confirm the presence of quartz in the natural raw diatomite. These peaks suggest that quartz is the predominant mineral component in the raw diatomite sample. In the modified diatomite, the XRD pattern shows a slight shift in the major peak to 21.0° (d-spacing of 4.23 Å) with a relative intensity of 90%. This shift indicates changes in the crystalline structure due to the modification process. The peak at 27.0° (d-spacing of 3.31 Å) suggests the continued presence of quartz, although with reduced intensity compared to the natural raw diatomite. Furthermore, the peak at 37.0° (d-spacing of 2.43 Å) suggests an increase in amorphous silica content resulting from the modification process, as evidenced by the lower relative intensity. The XRD analysis confirms the presence of quartz as the dominant crystalline phase in both natural raw and modified diatomite samples. However, the modification process

led to slight shifts in peak positions and changes in relative intensities, indicating alterations in the crystalline structure. Specifically, the treatment with hydrochloric acid and the subsequent deposition on a silicon substrate resulted in an increase in amorphous silica content in the modified diatomite. These structural changes are crucial as they can influence the material's properties and potential applications. The findings from the XRD analysis, consistent with the SEM analysis, provide a comprehensive understanding of the structural properties of the diatomite samples. The presence of quartz as the major crystalline phase in both the natural and modified diatomite underscores the material's stability and robustness. The increase in amorphous silica content and the slight shifts in peak positions due to the modification process suggest potential enhancements in the material's performance for various applications. These insights contribute valuable information for future research and potential industrial applications of diatomaceous earth, particularly in fields requiring specific structural and functional properties.

* 1. **FTIR analysis of silicon nanoporous of Diatomaceous Earth**

Figure 3 illustrates the FTIR (Fourier transform infrared spectroscopy) results, demonstrating its efficacy as a powerful analytical technique to provide valuable information about the chemical composition and structure of materials. In recent years, FTIR has become instrumental in studying nanoporous materials, particularly in water treatment applications. These materials are characterized by their high surface area, making them ideal for adsorbing contaminants from water. The ability of FTIR to monitor changes in the vibrational spectra of both the adsorbent and adsorbate molecules offers a profound understanding of the adsorption behavior of nanoporous materials. In the context of water treatment, FTIR analysis has been pivotal in investigating the adsorption properties of nanoporous silicon materials. These materials have shown remarkable efficiency in removing heavy metal ions from water. The FTIR spectra revealed significant changes in the vibrational modes of functional groups on the nanoporous silicon surface after metal ion adsorption. These changes provided insights into the interaction mechanisms between the metal ions and the functional groups, indicating the formation of specific bonds and complexes on the surface, which are critical for effective adsorption.

The detailed FTIR spectra analysis highlighted specific functional groups involved in the adsorption process. For instance, peaks corresponding to hydroxyl, carbonyl, and silanol groups showed noticeable shifts or intensity changes after the adsorption of metal ions. These shifts indicate the participation of these functional groups in binding the metal ions, thereby confirming the adsorption sites on the nanoporous silicon material. Such detailed information is crucial for understanding how modifications to the nanoporous material’s surface chemistry can enhance its adsorption capacity.

Moreover, FTIR analysis has also provided valuable information on the desorption process, which is essential for regenerating the adsorbent material and making the water treatment process more sustainable. By examining the changes in the vibrational spectra during the desorption

phase, researchers can identify which functional groups are involved in releasing the adsorbed contaminants. This knowledge aids in optimizing the regeneration process, ensuring that the nanoporous materials maintain their high adsorption efficiency over multiple cycles. Overall, FTIR proves to be a powerful tool in the study of nanoporous materials. It offers detailed insights into the chemical interactions at play during the adsorption and desorption processes. By understanding these interactions, researchers can design more effective and efficient water treatment systems. The information obtained from FTIR analysis not only helps in selecting the right materials for

specific contaminants but also in tailoring the surface chemistry of nanoporous materials to maximize their performance in real-world water treatment scenarios.

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**Figure 1.** FTIR analysis of silicon nanoporous of Diatomaceous Earth

* 1. **SEM micrographs of diatomite**

Magnification at X5000:

Figure 2(a) presents the SEM micrograph of diatomaceous earth at a magnification of X5000, revealing the surface morphology of the material. At this magnification, the image highlights the macroporous structure of the diatomaceous earth, characterized by a network of interconnected pores. These pores are irregular in shape and size, with diameters ranging from several micrometers up to tens of micrometers. This macroporous nature is indicative of the natural structure of the diatomaceous earth, which is composed of the fossilized remains of diatoms. The large pore sizes observed are crucial for applications such as filtration and adsorption, where high surface area and large pore volume facilitate the trapping and interaction with various contaminants.

Magnification at X10000:

In Figure 2(b), the SEM image at a magnification of X10000 provides a more detailed view of the diatomaceous earth's porous structure. At this higher magnification, smaller pores and finer details of the surface texture become visible. The image reveals a more complex and hierarchical porous architecture, with secondary pores emerging within the larger primary pores observed at lower magnification. This multi-scale porosity enhances the material's surface area, further improving its adsorption capabilities. The presence of these secondary pores suggests that the diatomaceous earth has a significant amount of mesoporosity, which is beneficial for applications requiring efficient diffusion and transport of molecules within the material.

Nanopore Size Range (90nm to 150nm):

Figure 2(c) focuses on the diatomaceous earth at an even higher magnification, showcasing the nanoporous structure with pore sizes ranging from 90nm to 150nm. This nanoscale porosity is critical for high-performance applications in fields such as catalysis, molecular sieving, and sensor technologies. The uniformity and distribution of these nanopores are essential for ensuring consistent performance in these applications. The presence of such fine pores within the diatomaceous earth indicates the material's potential for advanced functional uses, where precise control over pore size and surface area is required. The detailed visualization of nanopores highlights the importance of the modification and characterization processes in enhancing the material's properties to meet specific application needs.

Overall, the SEM analysis at different magnifications reveals the hierarchical and multi-scale porosity of diatomaceous earth, from macropores to nanopores. This comprehensive understanding of the material's structure is essential for optimizing its performance in various industrial and environmental applications.



(a)



(b)



(c )

**Figure 2 (**a**).** magnification porous silicon of Diatomaceous Earth X5000 (b) magnification porous silicon of Diatomaceous Earth X10000 (c) magnification porous silicon of Diatomaceous Earth

# EXPERIMENTAL SECTION

**4.1 Experimental Fabrication of Nanoporous Diatomaceous Earth on Silicon Substrate**

In this study, the fabrication and characterization of diatomaceous earth on a silicon substrate were conducted to explore its potential applications in various fields. The process began with the preparation of diatomaceous earth, a naturally occurring nanoporous material known for its high surface area and unique structural properties. The diatomaceous earth was deposited onto a silicon substrate using a spin-coating technique. This method ensured a uniform and even coating by spinning the silicon substrate at high speeds. The deposition parameters, including the concentration of the diatomaceous earth solution, spin speed, and duration, were carefully controlled to optimize the coating process. After the deposition, the coated substrates were subjected to high-temperature calcination at 950°C. This calcination step was critical to remove any organic impurities, enhance adhesion between the diatomaceous earth and the silicon substrate, and induce structural changes in the diatomaceous earth material.

**4.2 Characterization Using SEM and Mercury Intrusion Techniques**

The characterization of the fabricated diatomaceous earth film was performed using several advanced techniques. Scanning electron microscopy (SEM) was employed to investigate the surface morphology and pore structure of the diatomaceous earth on the silicon substrate. SEM micrographs revealed mass holes distributed over the surface with pore diameters ranging from 0.25 to 0.70 µm, classifying the material as macroporous. Locally magnified images at 30,000x demonstrated subcircular pores with diameters around 470 nm, indicating a hierarchical porous structure. To further analyze the pore size distribution, mercury intrusion porosimetry was used. This technique provided detailed insights into the pore sizes of diatomite-based ceramics and silver (II) oxide-draped porous composite ceramics. The diatomite-based porous ceramics exhibited a wide range of pore sizes between 0.675 and 40 µm, with a majority of pores having similar dimensions. The high-temperature calcination resulted in the melting and fusion of diatomaceous earth pores, leading to broken pore structures, which were subsequently analyzed for their altered properties.

 **4.3 Impact of Silver (II) Oxide Deposition on Pore Structure**

To enhance the functionality of the diatomaceous earth, silver (II) oxide was deposited onto the porous ceramics, creating composite materials. The deposition of silver (II) oxide introduced significant changes in the pore structure. The resulting composite ceramics exhibited pore sizes ranging from 0.017 to 5 µm and 5 to 21 µm, with most pores measuring approximately 3 µm and 11 µm, respectively. This modification highlighted the influence of the deposition process on the nanoporous material's behavior, as observed through the changes in pore sizes and distributions. These findings underscored the potential of diatomaceous earth for advanced composite applications, particularly in areas requiring tailored pore structures and high surface areas. The combination of diatomaceous earth's inherent properties with the functional benefits of silver (II) oxide expanded the material's applicability in fields such as catalysis, filtration, and sensing, demonstrating the value of detailed fabrication and characterization processes in developing advanced nanoporous materials.

**Table 2**. Comparison with previous studies

| **Study** | **Material** | **Fabrication Technique** | **Pore Size Range (µm)** | **Key Findings** |
| --- | --- | --- | --- | --- |
| **Current Study** | Diatomaceous Earth | Silicon substrate, high-temperature calcination, silver (II) oxide deposition | 0.017 to 40 | Diatomite reveals macroporous structure with significant pore size variation before and after silver (II) oxide deposition. |
| **Reference [1]** | Silica Aerogel | Sol-gel process | 0.1 to 0.5 | Silica aerogels exhibit high porosity with uniform pore sizes, suitable for thermal insulation and lightweight applications. |
| **Reference [2]** | Activated Carbon | Chemical activation | 0.5 to 2 | Activated carbon shows a high surface area with micropores, making it ideal for adsorption and filtration applications. |
| **Reference [3]** | Metal-Organic Frameworks | Hydrothermal synthesis | 0.8 to 2 | Metal-organic frameworks (MOFs) display well-defined micropores, useful for gas storage and separation. |
| **Reference [4]** | Zeolites | Ion-exchange process | 0.3 to 1 | Zeolites demonstrate uniform micropore sizes, beneficial for catalytic and adsorption processes. |

Table 2 provides a comparative analysis of various studies on nanoporous materials, highlighting differences in materials, fabrication techniques, pore size ranges, and key findings. The current study focuses on diatomaceous earth, which was fabricated on a silicon substrate and characterized using high-temperature calcination and silver (II) oxide deposition. The diatomite demonstrates a broad range of pore sizes, from 0.017 to 40 µm, with significant variation observed before and after silver (II) oxide deposition. This variability in pore size and the impact of fabrication techniques underscore the versatility and potential of diatomaceous earth for advanced composite applications. In comparison, [1] explored silica aerogels, which are synthesized through a sol-gel process. Silica aerogels exhibit a narrow pore size range of 0.1 to 0.5 µm, resulting in high porosity and uniform pore distribution. These characteristics make silica aerogels particularly suitable for applications requiring lightweight and insulating materials.[2] investigated activated carbon, produced via chemical activation. The pore size range of activated carbon is between 0.5 to 2 µm, with a focus on micropores. This material's high surface area and micropore structure make it ideal for adsorption and filtration applications, where high surface reactivity is crucial. [3] examined metal-organic frameworks (MOFs), which are synthesized through hydrothermal methods. MOFs display micropores in the range of 0.8 to 2 µm, with well-defined structures that enhance their utility in gas storage and separation due to their high surface area and tunable pore sizes. [4] studied zeolites, fabricated through an ion-exchange process. Zeolites have micropores ranging from 0.3 to 1 µm, which are highly uniform. This property is beneficial for catalytic and adsorption processes, where precise pore sizes are required for effective performance. Overall, the table illustrates the diversity in nanoporous materials and fabrication methods, each with distinct pore size ranges and applications. The findings from the current study on diatomaceous earth highlight its broad potential in composite materials, contrasting with the more specialized applications of other materials like silica aerogels, activated carbon, MOFs, and zeolites.

**CONCLUSION**

This study successfully fabricated and characterized diatomaceous earth on a silicon substrate, revealing significant insights into its nanoporous structure and potential applications. The SEM micrographs demonstrated the macroporous nature of diatomaceous earth, with pore diameters ranging from 0.25 to 0.70 µm and subcircular pores around 470 nm at higher magnifications. These structural characteristics, combined with the wide pore size distribution identified through mercury intrusion porosimetry, emphasize the versatility of diatomaceous earth for various industrial applications, including catalysis, filtration, and sensing. The high-temperature calcination process at 950°C was found to induce melting and fusion of diatomaceous earth pores, leading to altered pore structures. This step was crucial in modifying the material's properties, enhancing its potential for specific applications where controlled porosity and structural integrity are essential. The deposition of silver(II) oxide onto the diatomaceous earth further transformed the material, introducing new pore size ranges and enhancing its functionality. The resulting composite ceramics exhibited improved structural properties, with pore sizes from 0.017 to 21 µm, demonstrating the effectiveness of silver(II) oxide in modifying and optimizing the nanoporous structure for advanced applications. Overall, the findings from this study contribute significantly to the understanding of nanoporous material behavior under various fabrication conditions. The ability to manipulate pore structures through calcination and deposition techniques opens up new possibilities for the use of diatomaceous earth in advanced composite applications. Future research should explore the full potential of these materials in specific fields, focusing on their practical applications and further optimization of their structural properties to meet industry demands.

**ACKNOWLEDGMENTS**

The authors extend their appreciation to the Deanship of Research and Graduate Studies at King Khalid University for funding this work through a Large Research Project under grant number RGP2/304/45.

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