

Sonochemical Synthesis of SiO₂ Nanoparticles for Heavy Metal Remediation from Wastewater

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Abstract - Heavy metal contamination in industrial wastewater is a major environmental and public health concern due to the toxic, non-biodegradable, and bioaccumulative nature of metal ions such as trivalent chromium (Cr³⁺). This contaminant can cause serious health issues and long-term ecological damage if not properly treated. Conventional wastewater treatment methods often face limitations such as low removal efficiency, high operational costs, and the generation of secondary sludge, which creates additional disposal challenges. Therefore, there is a growing need for more efficient, cost-effective, and environmentally friendly treatment technologies.

Nanotechnology has emerged as a promising solution, particularly through the use of silica (SiO₂) nanoparticles. These nanoparticles are highly suitable for water treatment due to their large surface area, adjustable pore structure, good chemical stability, and the presence of active surface groups that enhance adsorption capacity. In this study, SiO₂ nanoparticles are synthesized using a sonochemical method, which involves the use of ultrasonic waves. This technique generates acoustic cavitation, where microscopic bubbles form and collapse rapidly, creating localized high temperature and pressure conditions. This leads to faster reaction rates, uniform particle size distribution, and improved surface properties compared to conventional synthesis methods.

Evaluating the adsorption performance of the synthesized nanoparticles for removing heavy metals from wastewater. Batch adsorption experiments are carried out to study the effect of important parameters such as adsorbent dosage. These studies help in determining the efficiency, capacity, and feasibility of the synthesized SiO₂ nanoparticles for practical wastewater treatment applications. The study aims to develop an effective and eco-friendly adsorption system using sonochemically synthesized SiO₂ nanoparticles, which can significantly improve the removal of heavy metals from industrial wastewater and support the advancement of sustainable water treatment technologies.

KEYWORDS

SiO₂ Nanoparticle, Ultrasonication, adsorption, heavy metal remediation

INTRODUCTION

The rapid increase in industrialization and urbanization has created serious challenges for water resources across the world. Many industries discharge untreated or partially treated wastewater into natural water bodies such as rivers, lakes, and groundwater systems. This wastewater contains various pollutants, among which heavy metals are considered highly dangerous. Industrial sectors such as electroplating, mining, battery production, textile dyeing, and leather processing are major contributors to heavy metal contamination. These metals enter the environment in dissolved or particulate form and remain there for a long time. Unlike organic pollutants, heavy metals do not degrade naturally and therefore persist in water systems. Their accumulation over time increases toxicity levels in the environment. The water pollution caused by heavy metals has become a major global concern. Proper treatment of industrial wastewater is necessary to prevent long-term environmental damage.

Heavy metals such as trivalent chromium (Cr³⁺) are commonly found in contaminated wastewater. This metal is a toxic even at very low concentrations and can cause severe damage to living organisms. When released into water bodies, heavy metals either remain dissolved or settle in sediments. Aquatic organisms such as plankton absorb these metals first, and then they move up the food chain when larger organisms consume smaller ones. This process is known as bioaccumulation and biomagnification. As the concentration increases at higher levels of the food chain, humans become exposed through drinking water or consumption of contaminated fish. This exposure can lead to serious health issues. Therefore, controlling heavy metal pollution is essential for protecting both human health and the environment.

The toxic effects of heavy metals are well documented in scientific studies. Lead (Pb) exposure is known to cause brain damage, especially in children, and can affect their development. Cadmium (Cd) can damage the kidneys and weaken bones, while chromium (Cr) has been linked to cancer and genetic mutations. These harmful effects occur because heavy metals interfere with normal biological processes in the body. Even small amounts consumed over a long period can lead to serious health conditions. Due to these risks, safe limits for heavy metals in drinking water have been established by organizations such as the World Health Organization. However, maintaining these limits remains a challenge in many developing and industrial regions.

To reduce heavy metal contamination, various traditional wastewater treatment methods have been developed and used for many years. Chemical precipitation is one of the most common methods, where chemicals are added to convert dissolved metals into solid particles that can be removed. This method is effective when metal concentrations are high, but it produces a large amount of sludge. The sludge contains toxic materials and requires proper disposal, which increases treatment costs. Additionally, this method is not very effective at low metal concentrations. Therefore, it is not always suitable for modern wastewater treatment needs.

Ion exchange which involves replacing harmful metal ions in water with safer ions using special resins. Although this technique can achieve high removal efficiency, it is expensive and requires regular regeneration of the resins. The regeneration process uses chemicals that may create secondary pollution. Membrane filtration methods such as reverse osmosis and nanofiltration are also used for heavy metal removal. These methods can remove very small particles and provide high-quality treated water. However, they require high energy input and are affected by membrane fouling, which reduces efficiency over time.

Coagulation and flocculation methods are also used in wastewater treatment to remove suspended particles and some dissolved metals. In this process, chemicals are added to form larger particles that can be easily separated from water. While effective, this method produces chemical sludge and requires careful control of chemical dosage. Electrochemical treatment is another option that uses electricity to remove metal ions. Although it is efficient, it consumes a large amount of energy and requires skilled operation. Overall, traditional methods have several limitations such as high cost, complexity, and production of secondary waste.

Due to these challenges, researchers have focused on developing alternative treatment methods that are efficient, cost-effective, and environmentally friendly. Among these methods, adsorption has gained significant attention. Adsorption is a simple process in which pollutants attach to the surface of a solid material called an adsorbent. This method is easy to operate and does not require complex equipment. It can be used for removing a wide range of pollutants, including heavy metals, dyes, and organic compounds. Because of these advantages, adsorption is widely used in modern wastewater treatment systems.

Different types of materials can be used as adsorbents, such as activated carbon, natural clays, agricultural waste, and bio-based materials. These materials are generally low-cost and easily available. However, they often have limited adsorption capacity and may not effectively remove metals at low concentrations. In some cases, their structure may degrade after repeated use, reducing their performance. Therefore, there is a need to develop advanced adsorbent materials with better efficiency and stability. This has led to increasing interest in nanomaterials.

Nanomaterials are extremely small materials with particle sizes in the nanometer range. Due to their small size, they have a very high surface area compared to their volume. This means more active sites are available for adsorption. Nanomaterials also have unique physical and chemical properties that enhance their interaction with pollutants. These properties make them highly effective for water treatment applications. In addition, their surface can be modified to improve selectivity for specific contaminants. This makes nanomaterials more efficient than traditional adsorbents.

Among different nanomaterials, silica nanoparticles are considered very promising for heavy metal removal. These particles are chemically stable, non-toxic, and environmentally safe. They have a large surface area and a porous structure, which increases their adsorption capacity. The presence of silanol groups on their surface provides active sites for binding metal ions. These groups can interact with metal ions through different mechanisms such as electrostatic attraction, ion exchange, and surface complexation. As a result, silica nanoparticles show high efficiency in removing heavy metals from water.

However, the performance of silica nanoparticles depends on their synthesis method. Traditional methods such as sol-gel and precipitation techniques are commonly used, but they have some drawbacks. These methods often require long reaction times and careful control of conditions such as pH and temperature. They may also involve the use of toxic chemicals, which can affect environmental safety. In addition, achieving uniform particle size and good dispersion is difficult in these methods. Therefore, researchers are exploring new techniques for nanoparticle synthesis.

Sonochemical synthesis has emerged as a modern and efficient method for preparing silica nanoparticles. This technique uses ultrasonic waves to enhance chemical reactions in liquids. The main mechanism involved is acoustic cavitation, where tiny bubbles form and collapse, producing high temperature and pressure for a short time. These extreme conditions help in forming uniform and fine nanoparticles quickly. Sonochemical synthesis reduces reaction time, improves particle quality, and minimizes the use of harmful chemicals. It also enhances dispersion and prevents particle aggregation. Due to these advantages, this method is considered environmentally friendly and suitable for large-scale production.

The sonochemical method offers better control over the size and shape of silica nanoparticles compared to conventional methods. By adjusting parameters such as ultrasonic frequency, power, and reaction time, researchers can control the properties of the nanoparticles. This flexibility allows the production of uniform and stable particles. Uniform particles are important because they provide consistent performance during adsorption. In addition, sonication improves mixing in the solution, ensuring that all reactants are evenly distributed. This leads to better reaction efficiency and higher product quality. Therefore, sonochemical synthesis is considered a reliable technique for producing high-quality silica nanoparticles.

One of the key advantages of sonochemical synthesis is the rapid formation of nanoparticles. In traditional methods, the formation process may take several hours or even days. However, in sonochemical synthesis, the reaction is completed within a much shorter time due to the high energy generated by ultrasonic waves. This reduces overall processing time and improves efficiency. Faster synthesis also means lower energy consumption and reduced operational costs. These benefits make the sonochemical method suitable for large-scale production of nanoparticles. As industries move toward faster and more efficient processes, such techniques become highly valuable.

Another important benefit of sonochemical synthesis is improved dispersion of nanoparticles in the solution. Nanoparticles tend to stick together due to their high surface energy, forming aggregates. Aggregation reduces the effective surface area available for adsorption. Ultrasonic waves help to break these aggregates and keep the particles evenly dispersed. This increases the contact between nanoparticles and metal ions in the solution. Better contact leads to higher adsorption efficiency. Therefore, sonication not only helps in synthesis but also improves the performance of nanoparticles in water treatment applications.

The adsorption performance of silica nanoparticles is influenced by several factors. One of the most important factors is pH of the solution. The surface charge of silica nanoparticles changes with pH, which affects their interaction with metal ions. At certain pH levels, the surface becomes negatively charged, attracting positively charged metal ions. Another factor is adsorbent dosage, which determines the number of active sites available for adsorption. Contact time also plays a role, as sufficient time is required for the metal ions to attach to the surface. Temperature and initial metal concentration are additional factors that influence the adsorption process.

The mechanism of adsorption on silica nanoparticles involves multiple interactions. One of the main mechanisms is electrostatic attraction between negatively charged silica surfaces and positively charged metal ions. Another mechanism is ion exchange, where metal ions replace hydrogen ions present on the surface. Surface complexation is also important, where strong chemical bonds form between metal ions and functional groups on the nanoparticle surface. These combined mechanisms enhance the efficiency of heavy metal removal. Understanding these mechanisms helps in improving the design and application of nanoparticles for wastewater treatment.

Surface modification of silica nanoparticles further improves their adsorption capacity and selectivity. Functional groups such as amines ($-NH_2$) and thiols ($-SH$) can be introduced onto the surface. These groups have a strong affinity for certain metal ions, allowing selective removal. For example, amine groups are effective for binding chromium ions, while thiol groups show strong interaction with mercury ions. Surface modification also improves the stability of nanoparticles in different environmental conditions. This makes them suitable for treating complex wastewater containing multiple contaminants.

The use of nanotechnology in wastewater treatment has gained significant attention due to its efficiency and versatility. Nanoparticles can remove pollutants at lower concentrations compared to traditional materials. They also require smaller amounts to achieve effective treatment. This reduces material usage and overall cost. Additionally, nanomaterials can be reused after regeneration, which improves sustainability. These advantages make nanotechnology an important tool in modern environmental engineering. Researchers are continuously exploring new nanomaterials and methods to enhance water purification processes.

Silica nanoparticles are particularly attractive because they are environmentally friendly compared to many other nanomaterials. They are non-toxic and do not free harmful substances into the environment. This makes them safe for use in water treatment applications. Their chemical stability ensures that they maintain their structure and performance over time. Furthermore, silica is abundant and relatively inexpensive, making it suitable for large-scale applications. These properties make silica nanoparticles a preferred choice for sustainable wastewater treatment solutions.

In practical applications, silica nanoparticles can be used in batch or continuous treatment systems. In batch systems, nanoparticles are mixed with contaminated water and then separated after adsorption. In continuous systems, water flows through a column packed with nanoparticles. Both methods have their advantages depending on the scale and requirements of treatment. Continuous systems are more suitable for industrial applications, while batch systems are useful for laboratory studies. The flexibility of application increases the usefulness of silica nanoparticles in real-world conditions.

Regeneration and reuse of adsorbents are important for reducing operational costs. Silica nanoparticles can be regenerated using simple chemical treatments such as washing with acids or bases. After regeneration, they can be reused for multiple cycles without significant loss of efficiency. This reduces waste generation and improves sustainability. The ability to reuse adsorbents makes the process more economical. It also reduces the need for constant production of new materials, conserving resources and energy.

Another important aspect of wastewater treatment is the removal efficiency at low concentrations of heavy metals. Traditional methods often fail to achieve high efficiency at low concentrations. However, silica nanoparticles show excellent performance even at trace levels of contaminants. This makes them suitable for meeting strict environmental standards. High removal efficiency ensures safer discharge of treated water into the environment. It also reduces the risk of long-term pollution and health hazards.

The scalability of sonochemical synthesis is an important advantage for industrial applications. The process can be scaled up by using larger ultrasonic reactors. This allows production of nanoparticles in large quantities. The ability to control reaction conditions at a larger scale ensures consistent product quality. Industries require reliable and reproducible processes, and sonochemical synthesis meets these requirements. Therefore, it has strong potential for commercial applications in wastewater treatment.

Energy efficiency is another important factor in selecting a treatment method. Sonochemical synthesis requires less energy compared to high-temperature methods. The use of ultrasonic waves reduces the need for extreme conditions. This leads to lower energy consumption and reduced operational costs. Energy-efficient processes are important for sustainable development. They help in reducing environmental impact and improving overall system performance.

Environmental safety is a major concern in wastewater treatment technologies. Many traditional methods produce secondary pollutants such as toxic sludge. In contrast, adsorption using silica nanoparticles generates minimal waste. This reduces the burden on disposal systems and minimizes environmental risks. Cleaner processes are essential for long-term sustainability. The use of eco-friendly materials and methods supports environmental protection efforts.

The integration of sonochemistry and nanotechnology represents a modern approach to environmental remediation. This combination enhances both synthesis and application of nanoparticles. Sonochemistry improves particle properties, while nanotechnology provides high efficiency in pollutant removal. Together, they offer a powerful solution for water treatment challenges. This interdisciplinary approach is gaining popularity among researchers and engineers.

Research studies have shown that sonochemically synthesized silica nanoparticles have higher adsorption capacity compared to conventionally prepared ones. This is mainly due to their improved surface area and porosity. Better structural properties lead to more active sites for adsorption. As a result, these nanoparticles can remove larger amounts of heavy metals from water. Improved performance makes them suitable for advanced treatment systems.

The use of advanced characterization techniques helps in understanding the properties of nanoparticles. Techniques such as X-ray diffraction (XRD), scanning electron microscopy (SEM), and transmission electron microscopy (TEM) provide information about structure and morphology. Fourier transform infrared spectroscopy (FTIR) helps identify functional groups on the surface. BET analysis measures surface area and porosity. These techniques are essential for evaluating the quality and performance of nanoparticles.

Understanding the relationship between nanoparticle properties and adsorption performance is important for optimizing treatment processes. By studying different parameters, researchers can improve efficiency and reduce costs. Optimization helps in identifying

the best conditions for maximum removal of heavy metals. This leads to better design of treatment systems and improved practical applications.

Heavy metal contamination is not only an environmental issue but also a socio-economic problem. Polluted water affects agriculture, fisheries, and human health. This leads to economic losses and increased healthcare costs. Providing clean water is essential for sustainable development. Therefore, effective wastewater treatment technologies are necessary to address this problem.

Government regulations and environmental policies play an important role in controlling pollution. Strict guidelines are set for the discharge of industrial wastewater. Industries are required to treat their wastewater before releasing it into the environment. However, compliance with these regulations depends on the availability of efficient treatment methods. Advanced technologies like nanotechnology can help industries meet these standards.

Public awareness about water pollution is also increasing. People are becoming more conscious of environmental issues and demanding cleaner water. This has encouraged industries and researchers to develop better treatment solutions. Sustainable technologies are gaining importance in modern society. The use of eco-friendly materials supports environmental conservation.

The development of new materials for water treatment is an active area of research. Scientists are exploring different types of nanoparticles and composites. Combining silica nanoparticles with other materials can further improve their performance. Hybrid materials offer better adsorption capacity and stability. This opens new possibilities for advanced water treatment technologies.

Future research should focus on improving the efficiency and cost-effectiveness of nanoparticle-based treatment methods. Large-scale applications require affordable and reliable processes. Continuous improvement in synthesis and application techniques will enhance performance. Collaboration between researchers and industries is important for practical implementation.

The study of adsorption isotherms helps in understanding how metal ions interact with the adsorbent surface. Models such as Langmuir and Freundlich isotherms are commonly used. The Langmuir model assumes monolayer adsorption on a uniform surface, while the Freundlich model describes adsorption on heterogeneous surfaces. These models help in determining adsorption capacity and surface properties. They are useful for designing and optimizing treatment systems. Proper analysis of isotherms provides valuable information about the adsorption mechanism.

Kinetic studies are important for understanding the rate of adsorption and the steps involved in the process. Models such as pseudo-first-order and pseudo-second-order kinetics are used to describe adsorption behavior. These models help in identifying whether the process is controlled by physical or chemical interactions. Kinetic analysis also helps in determining the rate-limiting step. Understanding adsorption kinetics is essential for designing efficient treatment systems. It ensures proper utilization of adsorbent materials.

Thermodynamic analysis provides information about the feasibility and nature of the adsorption process. Parameters such as Gibbs free energy, enthalpy, and entropy are studied. Negative values of Gibbs free energy indicate that the process is spontaneous. Enthalpy changes help determine whether the process is endothermic or exothermic. Entropy changes provide information about randomness at the solid-liquid interface. These studies help in understanding the fundamental nature of adsorption. They also support the design of efficient treatment systems.

The reuse and safe disposal of nanoparticles are important factors in wastewater treatment systems. Although silica nanoparticles are generally considered non-toxic and environmentally safe, improper disposal may still create long-term environmental concerns. After adsorption, nanoparticles contain concentrated heavy metals, which must be handled carefully. Regeneration methods such as chemical washing can be used to remove the adsorbed metals and restore the adsorption capacity of the nanoparticles. This allows repeated use of the same material, reducing waste generation. Recycling of nanoparticles not only saves cost but also supports sustainable practices. Proper disposal techniques must be followed when regeneration is not possible. This ensures that heavy metals do not re-enter the environment. Therefore, lifecycle management of nanoparticles is essential for safe and effective wastewater treatment.

Combining adsorption with other treatment methods can improve overall efficiency in wastewater treatment.

A single method may not always be sufficient to remove all types of pollutants present in complex wastewater. For example, adsorption can be combined with membrane filtration, coagulation, or biological treatment to achieve better results. Such hybrid

systems help in removing both dissolved and suspended pollutants effectively. They also improve treatment efficiency at different concentration levels. The integration of multiple methods reduces the limitations of individual techniques. This approach is especially useful in industrial wastewater treatment where multiple contaminants are present. By designing combined systems, researchers can achieve higher purification levels. Therefore, hybrid treatment systems provide a more complete solution for water purification.

The application of nanotechnology in environmental engineering is growing rapidly due to its high efficiency and advanced performance. Nanomaterials are being widely studied for water purification, air treatment, and soil remediation. Their unique properties such as high surface area, reactivity, and tunable surface chemistry make them highly effective. In water treatment, nanoparticles can remove contaminants more quickly than traditional materials. They also require smaller amounts, which reduces material usage. Continuous research is leading to the development of new nanomaterials with improved performance. These advancements are helping to solve complex environmental problems. As technology progresses, nanotechnology is expected to play a major role in sustainable environmental management.

Although silica nanoparticles have shown excellent performance in laboratory studies, their application in real wastewater systems still requires further investigation. Real wastewater contains a mixture of different pollutants, which may affect adsorption efficiency. Factors such as pH variation, presence of competing ions, and temperature changes can influence performance. Therefore, it is important to test nanoparticles under actual field conditions. Pilot-scale studies are necessary to evaluate their practical applicability. These studies help in understanding real-world challenges and limitations. Based on such results, modifications can be made to improve performance. Thus, bridging the gap between laboratory research and real applications is essential for successful implementation.

Economic feasibility is an important factor in selecting wastewater treatment technologies. Even if a method is highly efficient, it must also be affordable for large-scale use. The cost of raw materials, synthesis process, and operation must be considered. Sonochemical synthesis offers advantages such as reduced reaction time and lower energy consumption, which help in reducing costs. Additionally, the possibility of regenerating and reusing nanoparticles further improves economic efficiency. Industries prefer treatment methods that provide a balance between cost and performance. Therefore, cost analysis plays a key role in determining the suitability of nanoparticle-based treatment systems.

Environmental impact assessment is necessary to evaluate the overall benefits and risks of using new technologies. While nanotechnology offers many advantages, its long-term environmental effects must be carefully studied. It is important to ensure that nanoparticles do not cause unintended harm to ecosystems. Studies should focus on their behavior, stability, and potential toxicity in different environments. Sustainable solutions should aim to minimize negative impacts while maximizing benefits. By conducting proper assessments, researchers can develop safer and more reliable technologies. This helps in gaining public trust and promoting wider adoption of advanced treatment methods.

The development of green synthesis methods is becoming increasingly important in modern research. Green chemistry focuses on reducing the use of harmful chemicals and minimizing waste generation. Sonochemical synthesis follows these principles by using less toxic reagents and lower energy input. The process also reduces reaction time, which improves efficiency. Environmentally friendly synthesis methods are essential for sustainable development. They help in reducing pollution at the source rather than controlling it later. Therefore, green synthesis of nanoparticles is an important step toward eco-friendly wastewater treatment solutions.

The role of nanoparticles in water purification is expected to grow significantly in the future. Continuous research and technological advancements are improving their performance and applicability. Scientists are developing new materials with higher adsorption capacity and better selectivity. Innovations in synthesis methods are also making production more efficient. These developments will lead to more effective and affordable treatment systems. As water pollution becomes a global concern, the demand for advanced purification technologies will increase. Nanoparticles are likely to become a key component of next-generation water treatment systems.

Silica nanoparticles have shown great potential as adsorbents for heavy metal removal from wastewater. Their large surface area and active sites allow efficient interaction with metal ions. They are chemically stable and can withstand different environmental conditions. In addition, their non-toxic nature makes them safe for environmental applications. The ability to modify their surface

further enhances their performance. These properties make silica nanoparticles highly suitable for wastewater treatment. Continued research will help in improving their efficiency and expanding their applications.

The integration of advanced materials and innovative techniques is essential for solving complex environmental problems. Combining nanotechnology with methods such as sonochemistry creates powerful treatment solutions. This multidisciplinary approach brings together knowledge from chemistry, engineering, and environmental science. It allows the development of more efficient and sustainable technologies. By using combined approaches, researchers can overcome the limitations of individual methods. This leads to better performance and wider applicability. Therefore, innovation and integration are key factors in advancing wastewater treatment technologies.

Water pollution control has become a global priority due to increasing environmental and health concerns. Access to clean water is essential for human survival and economic development. Contaminated water affects agriculture, industry, and daily life. Governments and organizations are working to improve water quality through regulations and technologies. Sustainable treatment methods are required to meet future water demands. Advanced materials like silica nanoparticles can play an important role in achieving this goal. Ensuring clean water for future generations is a shared responsibility.

The use of sonochemically synthesized silica nanoparticles aligns with modern environmental goals and sustainability principles. This method combines efficiency, cost-effectiveness, and eco-friendliness. It reduces energy consumption and minimizes the use of harmful chemicals. The nanoparticles produced have improved properties, leading to better adsorption performance. This approach supports the development of green technologies for environmental protection. It also meets the requirements of modern wastewater treatment systems.

Heavy metal contamination in wastewater is a serious environmental and health problem because these metals do not degrade and can accumulate in living organisms. Traditional treatment methods like chemical precipitation, ion exchange, and filtration are useful but have limitations such as high cost, sludge production, and lower efficiency at low concentrations. Because of these drawbacks, there is a need for better and more sustainable solutions. Silica nanoparticles are a promising option due to their high surface area and strong adsorption capacity.

When these nanoparticles are prepared using the sonochemical method, their properties improve significantly. Ultrasound helps in producing smaller, uniform, and well-dispersed particles, which increases their efficiency in removing heavy metals. This method also reduces reaction time and minimizes the use of harmful chemicals, making it environmentally friendly. It follows the principles of green chemistry and supports sustainable development. The combination of nanotechnology and sonochemistry provides a modern and effective approach for wastewater treatment. Overall, sonochemically synthesized silica nanoparticles offer a simple, efficient, and eco-friendly solution for heavy metal removal and have strong potential for future applications in water purification.

LITERATURE SURVEY

A modified Stöber synthesis assisted by medium–high frequency ultrasound (80, 120, and 500 kHz) at different powers for the preparation of spherical SiO₂ nanoparticles. They showed that ultrasound significantly accelerated the reaction (20–60 min) even at a relatively low ammonia/TEOS molar ratio (≈ 0.84), compared to conventional Stöber syntheses. The particle sizes obtained ranged from 63 to 117 nm depending on the applied frequency and power, with values of around 84 nm at 80 kHz, 74 nm at 120 kHz, and 90 nm at 500 kHz under comparable acoustic conditions. Increasing ultrasonic power tended to reduce the nanoparticle size, while sonication time and volume had only minor effects, indicating good scalability. The authors attributed this size control to cavitation phenomena, where bubble size and collapse dynamics at different frequencies altered nucleation and growth pathways. [1]

An ultrasonic-assisted Stöber approach to rapidly synthesize silica nanoparticles for drug delivery applications. Their work demonstrated that ultrasound not only shortened synthesis time but also improved particle size control, morphology, and surface characteristics. Importantly, the nanoparticles exhibited pH-sensitive behavior, enabling controlled drug release, making them suitable for biomedical applications. This study highlights the dual role of sonication in both accelerating synthesis and tailoring physicochemical properties for functional uses. [2]

This comprehensive review revisited the classic Stöber method, summarizing its modern adaptations and innovations. The authors discussed how parameters such as TEOS concentration, solvent choice, catalyst concentration, and additives influence silica particle formation. They also covered new opportunities, including template syntheses, surface functionalization, hybrid composites, and large-scale production. In the context of sonication, the review places ultrasound as one of the modern modifications to enhance

synthesis control, uniformity, and versatility, thereby expanding the method's applicability. [3]

The foundational work of Stöber, Fink, and Bohn first introduced the sol-gel synthesis of uniform silica spheres via controlled hydrolysis and condensation of tetraethyl orthosilicate (TEOS) in alcohol-ammonia solutions. This method established the principles of nucleation and growth control that remain central to silica nanoparticle synthesis. Particle size was shown to be tunable by adjusting precursor ratios, solvent composition, and catalyst levels. All subsequent studies, including ultrasound-assisted modifications, derive their baseline comparison from this classical method. [4]

Kharchenko and collaborators presented an analytical overview of strategies used to regulate particle size in the Stöber method. Their study emphasized the role of reagent concentrations, catalyst type and level, solvent variations, and process modifications. This work serves as a useful comparative framework, showing the chemical and operational levers available for size control, which can complement physical process modifications such as ultrasound. [5]

Wang and colleagues investigated the synthesis of silica particles at higher TEOS concentrations than commonly employed in classical Stöber protocols. They demonstrated that monodisperse spheres could still be obtained under these conditions, highlighting strategies to overcome challenges such as secondary nucleation and aggregation. Their findings are particularly relevant for scaling up synthesis, showing that higher precursor concentrations can increase productivity without compromising particle quality, and potentially complement sonochemical approaches. [6]

This study evaluated low-cost bio sorbents—Moringa seed powder and banana peel powder for the adsorption of Cr (VI) from aqueous solutions. The authors optimized parameters such as pH, adsorbent dose, and contact time, reporting promising adsorption capacities. Their work demonstrates that agricultural by-products can be efficient and sustainable alternatives for wastewater treatment, setting benchmarks against which nanomaterials like functionalized silica can be compared. [7]

Shrivastava and Gupta conducted a study on the use of various agricultural wastes as adsorbents for chromium removal from wastewater. They discussed adsorption efficiencies, mechanisms, and regeneration potential of such bio-wastes. Like Badessa et al., their results highlight the practicality and cost-effectiveness of bio sorption, while also illustrating the limitations that advanced nanomaterials like engineered silica might overcome, such as higher selectivity, reusability, or controlled functionalization. [8]

This study focused on the development of biopolymer/fumed silica nanocomposites. The authors demonstrated that the incorporation of fumed silica enhanced the thermal stability and mechanical properties of the composites while retaining biodegradability. Such materials were suggested as promising candidates for eco-friendly packaging and biomedical applications. [9]

The authors investigated the influence of lignocellulose fillers on polycaprolactone (PCL)- based composites. Their findings revealed that the addition of lignocellulose fillers improved the stiffness of PCL but reduced ductility. Importantly, the composites maintained their biodegradable nature, making them suitable for sustainable packaging uses. [10]

In this work, polycaprolactone/multi-walled carbon nanotube (MWCNT) nanocomposites were synthesized and characterized. The results showed remarkable improvements in tensile strength, thermal stability, and electrical conductivity with the incorporation of MWCNTs. However, achieving uniform dispersion of nanotubes remained a major challenge for optimizing properties. [11]

This review article provided an overview of the development and applications of PCL/metal oxide nanocomposites. Metal oxides such as TiO₂, ZnO, and SiO₂ were reported to significantly improve mechanical, barrier, and antimicrobial properties. These nanocomposites were highlighted as suitable for biomedical applications including tissue engineering and drug delivery, as well as advanced packaging. [12]

The authors synthesized porous PCL-silica composites for biomedical use. Their results indicated that the addition of silica improved porosity, bioactivity, and supported cell proliferation, making the composites ideal for scaffolds in bone regeneration and tissue engineering. [13]

This review summarized advances in biodegradable polymer nanocomposites, with emphasis on the role of nanofillers such as silica, clay, and carbon nanotubes. It was highlighted that nanofillers improve mechanical, barrier, and thermal properties, although

challenges persist in achieving homogeneous dispersion and maintaining polymer– nanofiller compatibility. [14]

In this study, vitamin B1 was used to modify the surface of silica nanoparticles for improved compatibility with PVC. The modification resulted in better dispersion of silica within the polymer matrix and enhanced thermal and mechanical properties of the composites. The work presented a novel approach or developing advanced nano-composites with applications in packaging and electronics. [15]

In this study, the authors investigated the efficiency of ion exchange resins for the removal of chromium from aqueous solutions and industrial wastewater. They demonstrated that ion exchange resins were highly effective in removing both trivalent and hexavalent chromium ions. The process exhibited good selectivity, reusability, and high treatment efficiency, making ion exchange a reliable technique for wastewater treatment. However, resin regeneration and cost factors were noted as limitations for large-scale applications. [16]

This comprehensive review summarized various techniques for chromium removal from water, including adsorption, ion exchange, chemical precipitation, membrane filtration, and electrochemical methods. The authors emphasized that adsorption using low-cost natural and synthetic materials emerged as one of the most efficient and economical methods. They also highlighted the environmental challenges posed by chromium contamination and stressed the need for sustainable, scalable, and cost-effective technologies. [17]

This experimental study explored the use of low-cost adsorbents for chromium removal from wastewater. The authors tested different adsorbent materials and reported significant removal efficiency at optimal pH and contact time. The findings revealed that natural and readily available adsorbents could provide an eco-friendly and economical alternative to conventional treatment methods, making them highly suitable for large-scale wastewater management in developing regions. [18]

This study reported the synthesis of SiO₂ nanoparticles using the sol-gel method. The nanoparticles were characterized by techniques such as XRD, SEM, and UV-Vis spectroscopy to investigate their structural and optical properties. The results revealed that the synthesized particles were spherical, uniform in size distribution, and exhibited good optical transparency. The sol-gel approach was highlighted as a cost-effective and versatile method for obtaining high-purity silica nanoparticles with potential applications in optics, sensors, and catalysis. [19]

The authors synthesized and characterized SiO₂ nanoparticles and evaluated their use in chemical mechanical polishing (CMP) of steel substrates. The study demonstrated that silica nanoparticles provided effective polishing performance, resulting in smoother steel surfaces with reduced roughness. The research emphasized the importance of controlling nanoparticle size and morphology to optimize polishing efficiency. This work established SiO₂ nanoparticles as promising abrasives in precision surface finishing applications. [20]

This research focused on the ultrasonic-assisted synthesis of SiO₂@TiO₂ core-shell nanocomposite particles. The structural, morphological, and photo catalytic properties of the materials were investigated. The authors reported that the core-shell architecture enhanced photo catalytic activity under UV light due to improved charge separation between SiO₂ and TiO₂. The findings demonstrated that ultrasound-assisted synthesis provides a simple and effective route to produce core-shell nanostructures with applications in environmental remediation and photo catalysis. [21]

Suslick and co-workers explored the fundamentals of sonochemistry and its applications in material synthesis. They explained that ultrasonic waves generate acoustic cavitation, which produces localized high temperature and pressure conditions. These extreme conditions accelerate chemical reactions and improve particle formation. The study emphasized that sonochemical methods can produce nanoparticles with better uniformity and controlled size compared to conventional methods. It also highlighted that ultrasound improves dispersion and reduces agglomeration. This makes sonochemistry a powerful tool in nanoparticle synthesis. Their work laid the foundation for further research in ultrasound-assisted material preparation. [22]

Gedanken investigated the application of sonochemistry in nanomaterial synthesis and reported that ultrasonic irradiation significantly enhances nucleation rates. The study demonstrated that nanoparticles synthesized under ultrasonic conditions exhibit smaller particle sizes and improved morphology. The author also noted that sonochemical synthesis is a rapid and efficient process compared to traditional techniques. This method allows better control over reaction conditions. The findings showed that ultrasound can be used to tailor the physical and chemical properties of nanoparticles. The study contributed to understanding the advantages of sonochemical synthesis. [23]

Mason and Lorimer provided a comprehensive overview of sonochemistry and its industrial applications. They discussed how ultrasonic waves influence chemical reactions through cavitation. The study highlighted that sonochemical processes are environmentally friendly and energy-efficient. It also explained the role of ultrasound in enhancing mass transfer and reaction kinetics. The authors emphasized that sonochemistry has potential applications in wastewater treatment and material synthesis. Their work supports the use of ultrasound for sustainable technologies. [24]

Bang and Suslick studied the application of ultrasound in nanomaterial synthesis and reported that sonochemical methods improve reaction rates and product quality. They showed that nanoparticles synthesized using ultrasound have better crystallinity and uniformity. The study also highlighted the role of cavitation in controlling particle formation. Their findings confirmed that ultrasound-assisted synthesis is a reliable method for producing high-quality nanomaterials. This research contributed to the development of advanced synthesis techniques. [25]

OBJECTIVES

- To synthesize SiO₂ nanoparticles using the sonochemical (ultra-sonication) method.
- To characterize the synthesized nanoparticles using analytical techniques (XRD, SEM/TEM, FTIR, BET, etc.).
- To evaluate the adsorption/remediation efficiency of SiO₂ nanoparticles for selected heavy metals from wastewater.
- To study the influence of parameters such as adsorbent dosage on removal efficiency.

EXPERIMENTAL WORK

ULTRASOUND-ASSISTED SYNTHESIS OF SiO₂ NANOPARTICLES AND DEGRADATION PROCESS OF HEAVY METALS

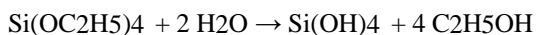
CHEMICALS REQUIRED:

Tetra Ethyl Ortho Silicate (TEOS)
Ammonium Hydroxide (NH₄OH)
Polyvinylpyrrolidone (PVP)
Ethanol (CH₃OH)

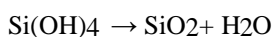
SiO₂ Nanoparticle Synthesis Process:

The synthesis of SiO₂ nanoparticles begins with the preparation of a solution containing ammonium hydroxide (NH₄OH), water (H₂O), and ethanol (EtOH) in a ratio of 10:40:80. In this mixture, NH₄OH provides hydroxide ions (OH⁻) that act as a catalyst for TEOS hydrolysis, water is necessary for the hydrolysis reaction, and ethanol serves as a solvent to dissolve TEOS and control the hydrolysis rate. The solution is then subjected to ultrasonic treatment for 10 minutes, where acoustic cavitation produces localized high temperature and pressure, accelerates Hydrolysis, prevents particle agglomeration, and ensures uniform nucleation of silica nanoparticles.

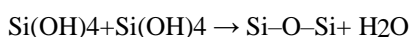
Tetraethyl orthosilicate (TEOS) is added drop wise to the sonicated solution, initiating hydrolysis according to the reaction:



The hydrolyzed silanol groups then undergo condensation to form silica networks either through self-condensation:



or via intermolecular condensation:



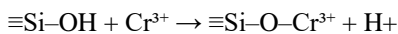
This results in the nucleation and growth of SiO₂ nanoparticles, which are allowed to age at room temperature for 12 hours to improve particle uniformity, surface stability, and complete condensation. The nanoparticles are then separated by centrifugation at 600 rpm for 10 minutes

and washed several times with ethanol and deionized water to remove unreacted TEOS, ammonia, and other byproducts. Finally, the nanoparticles are calcined at 400–600 °C for 2 hours, which removes residual organics, improves crystallinity, and enhances structural stability, yielding highly pure, crystalline SiO₂ nanoparticles with a large surface area.

Degradation Process of Heavy Metals:

For the adsorption of heavy metals, a stock solution of Cr³⁺ ions is prepared, and the pH is adjusted, typically to a slightly acidic or neutral range, since adsorption efficiency is strongly pH- dependent. The synthesized SiO₂ nanoparticles are added to the Cr³⁺ solution and continuously stirred to ensure uniform contact.

Adsorption occurs mainly through interactions between surface silanol groups (–Si–OH) and Cr³⁺ ions, which can involve ion exchange, electrostatic attraction, and complexation, as represented by:



Samples are collected at different time intervals to study the adsorption kinetics, followed by centrifugation to separate the nanoparticles with adsorbed Cr³⁺. The residual Cr³⁺ concentration in the supernatant is analyzed using techniques like AAS, ICP-OES, or UV spectroscopy, which helps determine the adsorption efficiency. Overall, the ultrasound-assisted synthesized SiO₂ nanoparticles are uniform, crystalline, and possess abundant silanol groups, making them highly effective, low-cost, and eco-friendly adsorbents for the removal of heavy metals such as Cr³⁺ from wastewater.



Figure No.1 Experimental Flow Diagram of Synthesis of Silica Nanoparticles

Exact Lab-Scale Quantities for Ultrasound Assisted Synthesis of Polyvinylpyrrolidone -Capped SiO₂ Nanoparticles

Total Reaction Volume: 100 mL reaction mixture

Table No. 1:- Chemicals and Quantities

Chemical	Quantity	Purpose
Ethanol (C ₂ H ₅ OH)	70 mL	Solvent

Chemical	Quantity	Purpose
Distilled water	10 mL	Hydrolysis medium
Ammonia solution (25%)	3 mL	Catalyst
TEOS (Si(OC ₂ H ₅) ₄)	4 mL	Silica precursor
PVP (Mw 40,000)	1 g	Stabilizing agent

Detailed Experimental Procedure

Step 1: Preparation of PVP Solution

- Weight 1 g PVP (Mw ≈ 40,000).
- Dissolve in 20 mL ethanol.
- Stir using magnetic stirrer for 20 minutes until completely dissolved.

Result: Clear PVP solution

Step 2: Preparation of Reaction Medium

In a 250 mL beaker add:

- 50 mL ethanol
- 10 mL distilled water
- 3 mL ammonia solution (25%)

Stir at 500 rpm for 10 minutes.

Purpose:

- Creates alkaline medium for TEOS hydrolysis

Step 3: Addition of PVP

Add the prepared PVP solution to the reaction mixture.

Stir for 10 minutes.

Now total volume ≈ 83 mL

Step 4: Addition of Silica Precursor

Measure 4 mL TEOS.

Add dropwise (~1 drop/sec) under stirring.

Continue stirring for 10 minutes.

Step 5: Ultrasonic Treatment

Place the reaction mixture in an ultrasonic bath or probe sonicator.

Table No. 2:- Recommended conditions

Parameter	Value
Frequency	20–40 kHz
Power	150–200 W
Time	45 minutes
Temperature	30–35 °C

Effect:

nucleation and particle formation

- Cavitation accelerates

Step 6: Aging of Sol

After sonication:

- Stir the mixture for 3 hours at room temperature.

Silica particles continue condensation and growth.

White colloidal suspension appears.

Step 7: Centrifugation

Transfer mixture into centrifuge tubes.

Centrifuge:

- 9000 rpm
- 10 minutes

White precipitate = SiO₂ nanoparticles

Step 8: Washing

Wash the precipitate with:

- Ethanol (20 mL)
- Distilled water (20 mL)

Repeat 3 times.

Purpose: Remove excess PVP and unreacted TEOS

Step 9: Drying

Dry nanoparticles in vacuum oven

Conditions:

- Temperature: 70°C
- Time: 12 hours

Final product:

PVP-capped SiO₂ nanoparticles

Yield: ~0.8 – 1.1 g

Table No. 3:- Expected Particle Characteristics

Property	Typical Value
Particle size	40 – 80 nm
Morphology	Spherical
Structure	Amorphous
Surface coating	PVP stabilized

Table No. 4:-

Characterization for Publication

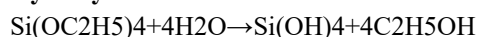
Technique	Expected Result
FTIR	Peak at 1650 cm ⁻¹ (C=O of PVP)
XRD	Broad peak at 22° (amorphous SiO ₂)
SEM/TEM	Uniform spherical particles
DLS	Narrow size distribution
TGA	5–10% PVP coating

Table No.5:- Important Parameters for Size Control

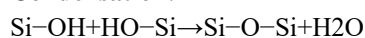
Parameter	Effect
TEOS concentration	Controls particle size
PVP amount	Controls aggregation
Ammonia concentration	Controls hydrolysis rate
Sonication power	Controls nucleation

Standard Reaction Summary:

Hydrolysis:



Condensation:



Below is an optimized parameter table commonly reported in research papers for ultrasound-assisted Stöber synthesis of SiO₂ nanoparticles (with stabilizers such as PVP). These ranges are compiled from multiple experimental studies on silica nanoparticle synthesis and parameter optimization.

Typical Optimized Reaction Composition (Example)

Table No. 6:- For ~50–80 nm silica nanoparticles

Reagent	Amount
Ethanol	70 mL
Distilled Water	10 mL
Ammonia (25%)	3 mL
TEOS	4 mL
PVP	1 g
Sonication	40 kHz, 45 min

Expected result:

- **Particle size:** 40–80 nm
- **Morphology:** spherical
- **Yield:** >80%

Key Optimization Trends (Important for Writing Research Papers)

1. TEOS Concentration

Increasing TEOS concentration produces **larger silica nanoparticles** because fewer nuclei form and more growth occurs.

2. Ammonia Concentration

Ammonia controls the **hydrolysis and condensation rate**.

- Low ammonia → small particles
- High ammonia → larger particles.

3. Water Content

Water affects hydrolysis kinetics.

Higher water concentration typically leads to **larger particle sizes** due to faster condensation.

4. Ultrasonic Parameters

Ultrasound improves mixing and nucleation.

Higher frequency and power can reduce particle size and reaction time.

Table No. 7:- Example Parameter Set Used in Sonochemical Study

Parameter	Value
Ethanol	6 mL
NH ₄ OH	0.40 mL
TEOS	0.25 mL
Water	3 mL
Sonication	120 kHz, 78 W, 20 min
Resulting size	~71 nm silica nanoparticles

Adsorption Process:

Adsorption is a physicochemical surface phenomenon in which molecules, ions, or atoms from a fluid phase (liquid or gas), known as the adsorbate, accumulate on the surface of a solid material called the adsorbent. Unlike absorption, where the substance dissolves into the bulk of another phase, adsorption occurs only at the interface, making it highly dependent on surface properties such as area, porosity, and surface energy.

In environmental engineering, adsorption plays a crucial role in the purification of water and wastewater. Industrial effluents often contain toxic heavy metals such as chromium (Cr³⁺). These metals are non-biodegradable, persistent, and can bioaccumulate in living organisms, leading to severe health issues including neurological disorders, kidney damage, and cancer. Therefore, efficient removal of these contaminants is essential before wastewater discharge.

Several conventional methods such as chemical precipitation, ion exchange, membrane filtration, and electrochemical treatment are used for heavy metal removal. However, these techniques often suffer from limitations such as high operational cost, sludge generation, incomplete removal, and sensitivity to operating conditions. Adsorption, on the other hand, is considered one of the most effective and economical methods due to its simplicity, high efficiency, and ability to remove even trace levels of contaminants.

The effectiveness of adsorption depends on the interaction between the adsorbent and adsorbate. These interactions can be classified into two main types:

Physisorption (Physical Adsorption):

Involves weak van der Waals forces. It is generally reversible and occurs at low temperatures.

Chemisorption (Chemical Adsorption):

Involves the formation of strong chemical bonds (covalent or ionic) between adsorbent and adsorbate. It is usually irreversible and more specific.

In recent years, nanomaterials have emerged as highly efficient adsorbents due to their exceptionally high surface area-to-volume ratio and enhanced surface reactivity. Among them, silicon dioxide (SiO₂) nanoparticles have attracted significant attention. These nanoparticles possess desirable properties such as:

- High surface area and porosity
- Abundant surface hydroxyl (-OH) groups for binding metal ions

- Chemical and thermal stability
- Non-toxicity and environmental compatibility

In this project, SiO₂ nanoparticles are synthesized using the sonochemical method, which employs ultrasonic irradiation to induce acoustic cavitation. This process generates localized high temperatures and pressures, leading to the formation of uniform, small-sized nanoparticles with improved surface characteristics. These enhanced properties significantly increase the adsorption efficiency of SiO₂ nanoparticles.

Purpose of Adsorption:

1. Rate of adsorption (how fast metal ions are removed)
2. Controlling mechanism (physical vs chemical adsorption)
3. Suitable model for design of treatment systems.

Role of Adsorbent Dosage in Heavy Metal Removal:

Adsorbent dosage is one of the most influential factors in determining the efficiency of pollutant removal from wastewater. In the case of SiO₂ nanoparticles, the dosage directly controls the number of available surface sites that can interact with heavy metal ions such as chromium (Cr³⁺).

When the amount of adsorbent is increased, more active binding sites become available for adsorption. These sites include surface hydroxyl groups (–OH) and siloxane bonds (Si–O–Si), which can interact with metal ions through electrostatic attraction, surface complexation, or ion exchange mechanisms.

At lower dosages, the number of active sites is limited compared to the concentration of metal ions present in the solution. As a result, only a fraction of the contaminants can be removed.

As the dosage increases, the removal efficiency improves significantly. This is because the increased surface area provides more locations for metal ions to attach. Higher dosage enhances the probability of collision between nanoparticles and metal ions, improving adsorption performance.

However, beyond a certain optimum dosage, the improvement in removal efficiency becomes negligible. This behavior is commonly observed due to:

- Particle aggregation: At higher concentrations, nanoparticles tend to cluster together, reducing the effective surface area.
- Overlapping of active sites: Some adsorption sites become inaccessible due to crowding of particles.
- Saturation effect: Once most of the metal ions are removed, additional adsorbent does not significantly improve performance.
- Thus, an optimum adsorbent dosage must be selected to achieve maximum efficiency while maintaining economic feasibility.

Surface Characteristics of SiO₂ Nanoparticles:

The adsorption performance of SiO₂ nanoparticles is strongly dependent on their surface properties. These include:

- High surface area
- Presence of functional groups
- Porous structure
- Surface charge characteristics

The large surface area of nanoparticles provides more adsorption sites compared to bulk materials. Moreover, the presence of silanol (Si–OH) groups plays a key role in binding heavy metal ions.

The porous nature of SiO₂ enhances diffusion of metal ions into the internal structure, improving adsorption capacity. Surface charge also influences interaction with positively charged metal ions, especially under suitable environmental conditions.

Characterization is essential to understand the physical and chemical properties of the adsorbent, which directly affect adsorption behavior.

Relationship Between Characterization and Adsorption Performance:

The results obtained from characterization techniques provide a clear understanding of how SiO₂ nanoparticles perform as adsorbents:

- High surface area (BET) → Increased adsorption capacity
- Functional groups (FTIR) → Strong binding with metal ions
- Amorphous structure (XRD) → More active sites
- Porous morphology (SEM/TEM) → Better diffusion and interaction

These properties collectively enhance the efficiency of SiO₂ nanoparticles in removing heavy metals from wastewater.

Importance in Wastewater Treatment:

The use of SiO₂ nanoparticles as adsorbents offers several advantages:

- High efficiency in removing toxic metals
- Easy availability and low cost
- Chemical stability
- Environmentally friendly nature

Optimizing adsorbent dosage and understanding material characteristics are essential steps in developing an effective wastewater treatment system.

OBSERVATIONS

Table No. 8:- Variations in concentration of precursor solution

Power of Probe [Watt]	TEOS [in Molar Unit]	NH ₄ OH [in Molar Unit]	C ₂ H ₅ OH [in Molar Unit]	H ₂ O [in Molar Unit]	Wt. of SiO ₂ Nanoparticles produced [gm]
250 Watt [20 mm diameter]	0.0269	0.0592	1.370	0.5551	1.1
	0.0358	0.0789	1.256	0.7399	2.6
	0.0448	0.0985	1.142	0.9248	3.2
	0.0537	0.1182	1.028	1.1096	4
	0.0627	0.1381	0.9137	1.2945	7.5
120Watt [12 mm diameter]	0.0269	0.0592	1.370	0.5551	1.7
	0.0358	0.0789	1.256	0.7399	2.8
	0.0448	0.0985	1.142	0.9248	4.3
	0.0537	0.1182	1.028	1.1096	6.2
	0.0627	0.1381	0.9137	1.2945	5

Discussion: The SiO₂ weight increases when TEOS, NH₄OH, and probe power are higher because they speed up hydrolysis and condensation, forming more silica. It decreases when these values are lower, because less silica is formed.

Table No. 9:- Variations in duty cycle of ultrasonic probe

Power of Probe [Watt]	On/Off Time in %	TEOS [in Molar Unit]	NH4OH [in Molar Unit]	C2H5OH [in Molar Unit]	H2O [in Molar Unit]	Wt. of SiO2 Nanoparticles produced [gm]
250 Watt [20 mm diameter]	50	0.0269	0.0592	1.370	0.5551	2.3
	55	0.0269	0.0592	1.370	0.5551	1.6
	60	0.0269	0.0592	1.370	0.5551	2.3
	65	0.0269	0.0592	1.370	0.5551	2
	70	0.0269	0.0592	1.370	0.5551	1.2
120Watt [12 mm diameter]	50	0.0269	0.0592	1.370	0.5551	1.7
	55	0.0269	0.0592	1.370	0.5551	2
	60	0.0269	0.0592	1.370	0.5551	1.6
	65	0.0269	0.0592	1.370	0.5551	1.7
	70	0.0269	0.0592	1.370	0.5551	1.7

Discussion: The weight of SiO₂ increases at 50–60% on/off time because cavitation is strong and stable, producing more silica. It decreases at 65–70% on/off time because excessive cavitation causes overheating and breaks the silica network, reducing the final yield.

Parameter	Optimized Range	Typical Value Used in Papers	Effect on Nanoparticles
TEOS concentration	0.20 – 0.30 M	~0.25 M	Higher TEOS → larger particles
Ammonia concentration	0.097 – 0.29 M	~0.19 M	Controls hydrolysis rate and nucleation
Water concentration	2 – 5 M	~4–5 M	Affects particle growth and size
Ethanol volume fraction	60 – 80% of solution	~70%	Solvent medium for Stöber reaction
PVP concentration	0.5 – 2 wt%	~1 wt%	Prevents aggregation
Reaction temperature	25 – 45 °C	~30 °C	Higher temperature → smaller particles but more polydispersity
Ultrasonic frequency	20 – 120 kHz	40 kHz	Controls cavitation intensity
Ultrasonic power	100 – 300 W	150–200 W	Improves nucleation and dispersion
Sonication time	20 – 60 min	30–45 min	Ensures uniform particle formation
Reaction time (aging)	2 – 24 h	3–6 h	Allows condensation and particle growth
pH	10 – 11	~10.5	Required for TEOS hydrolysis

Table No. 10:- Optimized Parameter for Ultrasound-Assisted Synthesis of SiO₂ Nanoparticles (with PVP)

Discussion: The optimized conditions for preparing SiO₂ nanoparticles using ultrasound with PVP. TEOS mainly controls the size of particles, while ammonia and water help in controlling the reaction and growth. Ethanol works as a solvent, and PVP prevents particles from sticking together. Ultrasonic parameter improve mixing and help in forming uniform particles. Proper time and basic pH are important to complete the reaction and get stable nanoparticles.

ANALYSIS

1. XRD Analysis:

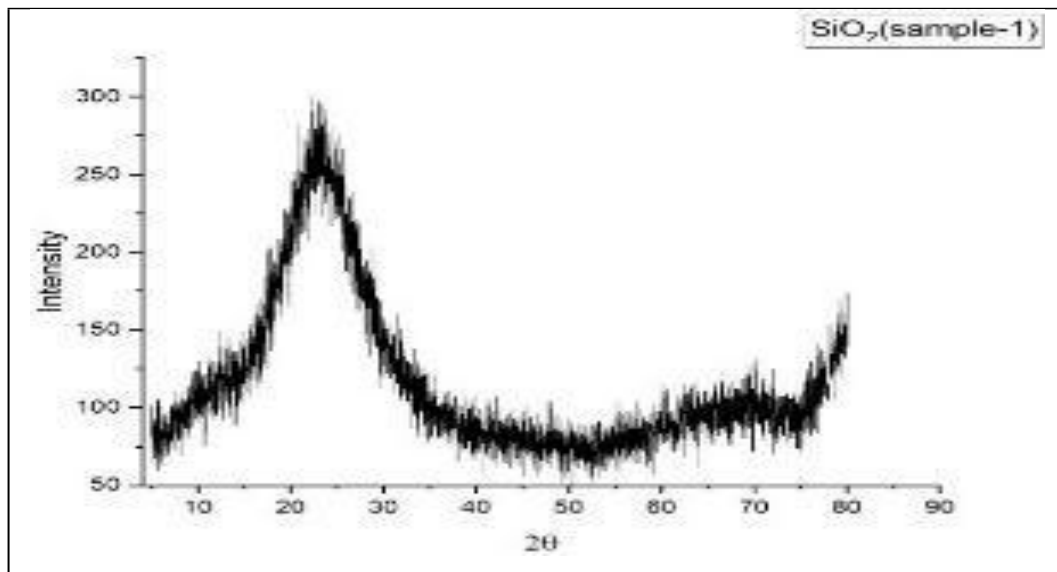


Figure No.2: XRD Analysis of Silica Nps

The X-Ray Diffraction (XRD) analysis was carried out to confirm the structural properties of the synthesized silica (SiO₂) nanoparticles. The obtained XRD pattern shows a broad diffraction peak around $2\theta \approx 22^\circ$, which is a characteristic feature of amorphous silica.

The broad hump instead of sharp crystalline peaks indicates the non-crystalline (amorphous) nature of the synthesized nanoparticles. This confirms that the sol-gel method successfully produced silica nanoparticles without long-range order in their atomic arrangement.

The absence of distinct crystalline peaks also suggests:

- Uniform dispersion of Si-O-Si network,
- No formation of unwanted crystalline silica phases,

Controlled synthesis suitable for further application in hyperthermia or other nanomedicine fields. Thus, the XRD results verify that the product obtained is amorphous SiO₂ nanoparticles, which aligns with expected structural characteristics for sol-gel-derived silica.

2. Polyvinylpyrrolidone (PVP)-XRD Analysis: -

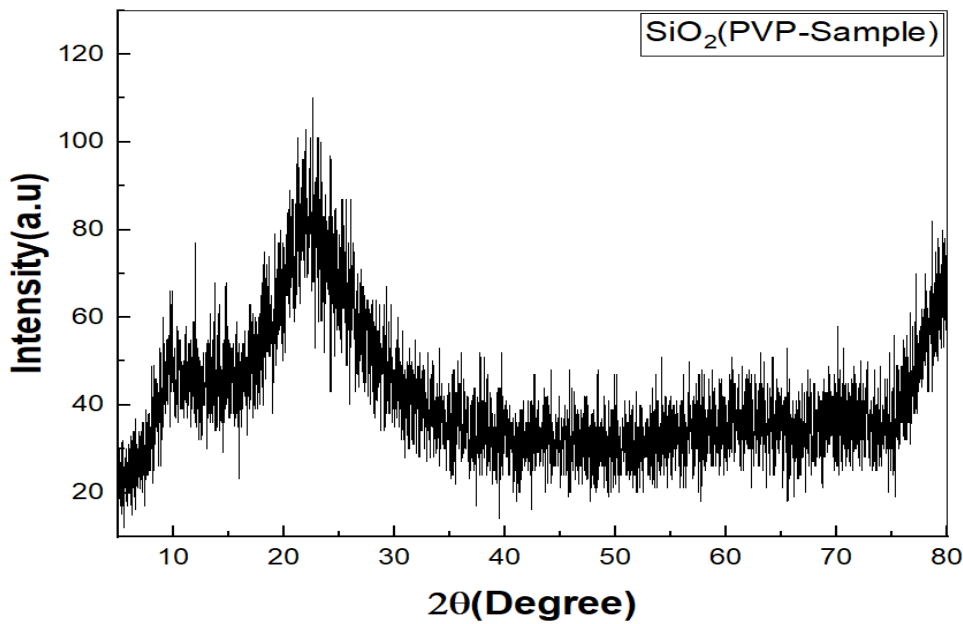


Figure No.3: XRD Analysis of Silica PVP Nps (1)

The X-Ray Diffraction (XRD) analysis of silica Polyvinylpyrrolidone (PVP) nanoparticles with sharp peaks, indicating a crystalline structure. The peaks are present at different 2θ values, which shows proper arrangement of atoms in the material. However, the intensity of peaks is comparatively lower, suggesting moderate crystallinity.

The graph also shows some noise, which indicates less uniform particle distribution. The peaks are not very well defined, showing that crystal growth is not fully developed. This means the particles are formed but not highly ordered. The structure is crystalline but with lower quality. The sample shows basic crystal formation with some irregularity.

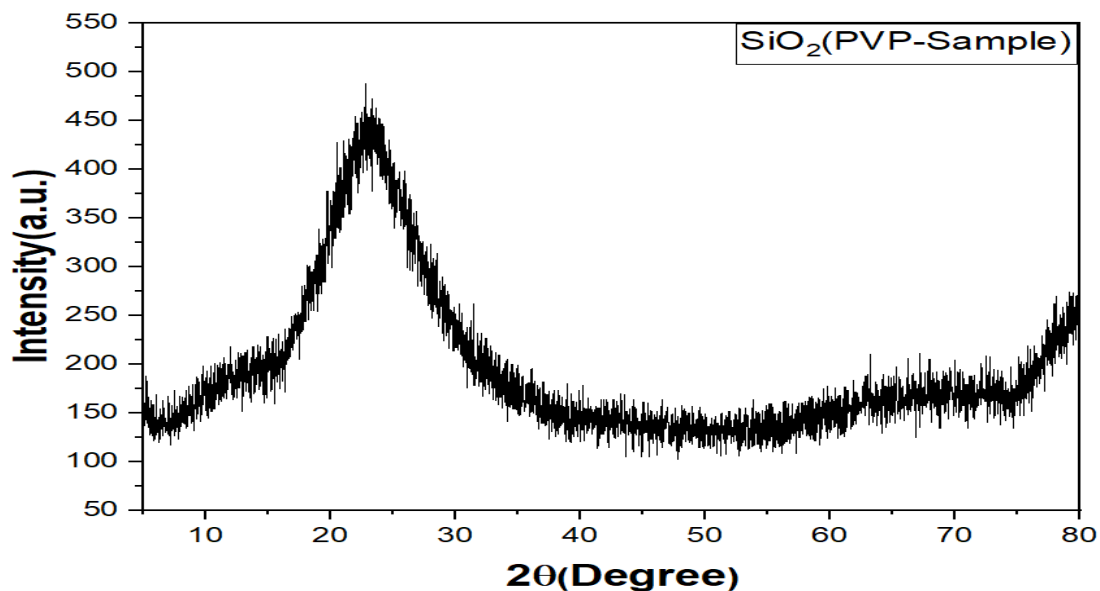


Figure No.4: XRD Analysis of Silica PVP Nps (2)

The X-Ray Diffraction (XRD) analysis of silica Polyvinylpyrrolidone (PVP) nanoparticles with sharp peaks, indicating a crystalline structure. The peak intensity is higher and more clear compared to the first graph. This indicates better crystallinity and improved particle formation. The peaks are well-defined and the graph has less noise, showing uniform particle distribution. The crystal structure is more stable and properly developed. This suggests better synthesis conditions in this sample. The particles are more regular in size and arrangement.

3. FTIR Analysis:

FTIR spectroscopy will be conducted to identify the functional groups present in the sample.

This will confirm:

- Presence of Si–O–Si stretching vibrations,
- Hydroxyl (–OH) groups,
- Completion of condensation reactions during synthesis.

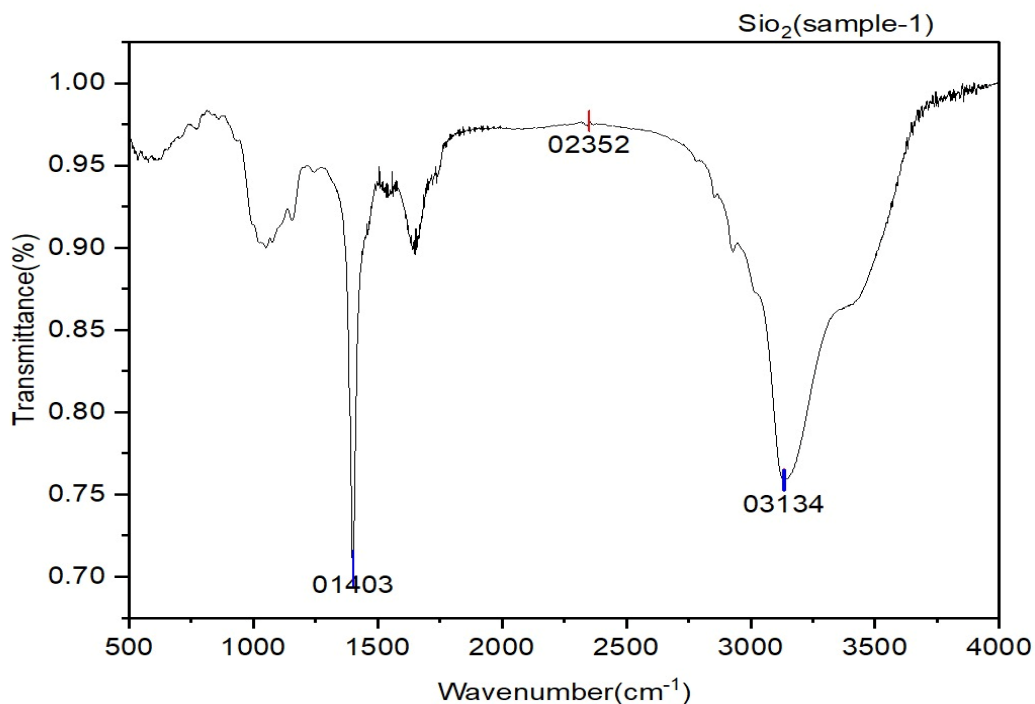


Figure No.5: FTIR Analysis of Silica Nps

The Fourier Transform Infrared (FTIR) spectrum of SiO₂ nanoparticles shows characteristic absorption bands confirming the formation of silica with typical surface functionalities. A broad peak observed around 3134 cm⁻¹ is attributed to O–H stretching vibrations, indicating the presence of adsorbed water molecules and silanol (Si–OH) groups on the silica surface. The peak near 2352 cm⁻¹ corresponds to atmospheric CO₂ absorption and does not represent the silica structure.

Another band around 1403 cm⁻¹ can be assigned to O–H bending vibrations or possible traces of residual impurities. The most significant feature of the spectrum lies in the region of 1000–1100 cm⁻¹, which is associated with strong Si–O–Si asymmetric stretching vibrations, confirming the formation of the silica network. Additional bands, if observed near 800 cm⁻¹ and 450–500 cm⁻¹, correspond to symmetric stretching and bending vibrations of Si–O–Si bonds.

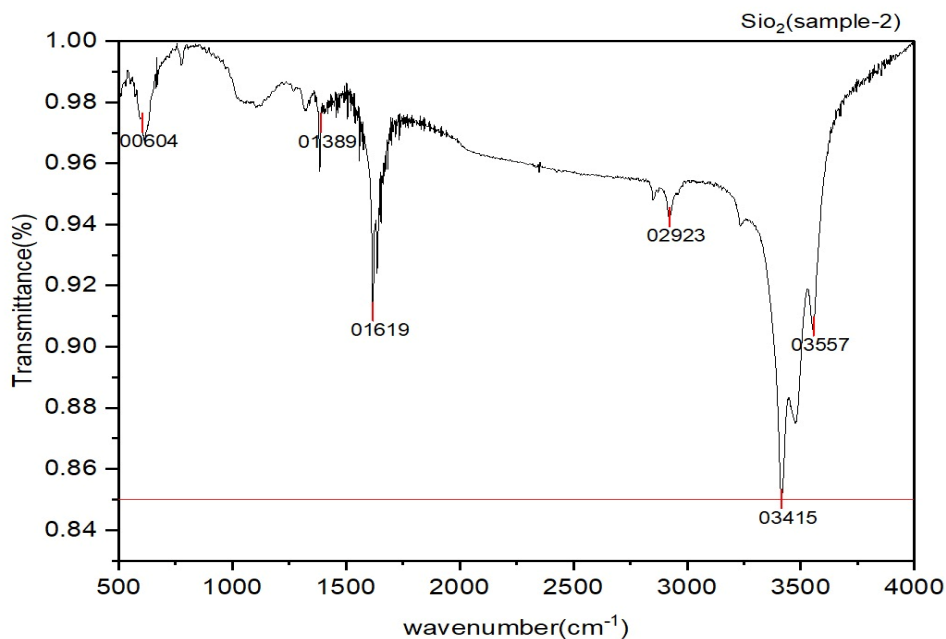


Figure No.6: FTIR Analysis of Silica Nps

The FTIR spectrum of SiO₂ nanoparticles exhibits several characteristic absorption bands indicating the presence of silica along with surface functional groups and minor impurities. A broad and intense peak observed around 3415–3557 cm⁻¹ is attributed to O–H stretching vibrations, confirming the presence of adsorbed water molecules and silanol (Si–OH) groups on the silica surface. The band near 2923 cm⁻¹ corresponds to C–H stretching vibrations, suggesting the presence of residual organic compounds, possibly from precursors or synthesis reagents. A prominent peak at 1619 cm⁻¹ is assigned to O–H bending vibrations of molecular water. The band around 1389 cm⁻¹ may be related to C–H bending or minor impurities.

The peak near 604 cm⁻¹ is associated with Si–O bending vibrations, which is a typical feature of silica materials. Although not strongly highlighted, the region around 1000–1100 cm⁻¹ is expected to show Si–O–Si asymmetric stretching, confirming the formation of the silica network structure.

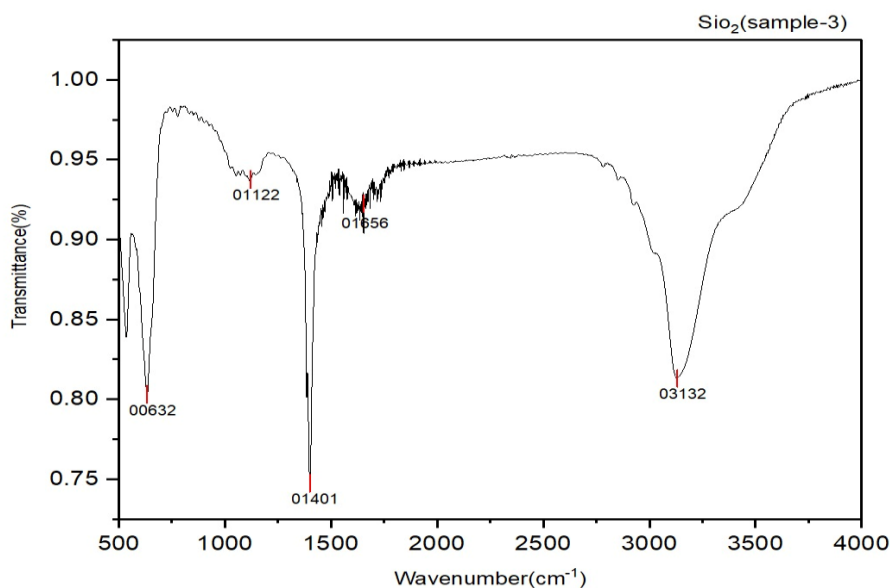


Figure No.7: FTIR Analysis of Silica Nps

The FTIR spectrum of SiO₂ nanoparticles shows characteristic absorption bands confirming the formation of silica along with surface hydroxyl groups and minor residual components.

A broad absorption band observed around 3132 cm⁻¹ is attributed to O–H stretching vibrations, indicating the presence of adsorbed water molecules and silanol (Si–OH) groups on the surface. The strong peak near 1401 cm⁻¹ is associated with O–H bending vibrations or possible contributions from residual organic species.

Another band around 1656 cm⁻¹ corresponds to the bending vibration of molecular water (H–O–H). The peak at approximately 1122 cm⁻¹ is characteristic of Si–O–Si asymmetric stretching vibrations, which confirms the formation of the silica network structure. Additionally, the band near 632 cm⁻¹ is assigned to Si–O bending vibrations, a typical feature of silica materials.

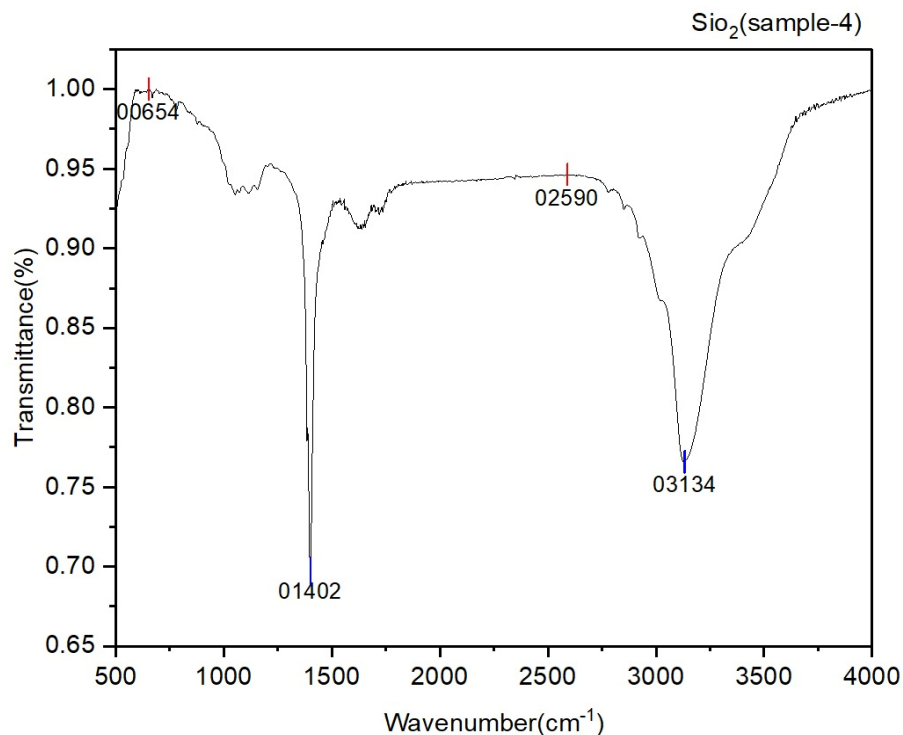


Figure No.8: FTIR Analysis of Silica Nps

The FTIR spectrum of SiO₂ (sample-4) displays characteristic absorption bands confirming the formation of silica along with surface hydroxyl groups and minor atmospheric contributions. A broad absorption band observed around 3134 cm⁻¹ is attributed to O–H stretching vibrations, indicating the presence of adsorbed water molecules and silanol (Si–OH) groups on the silica surface. The peak near 2590 cm⁻¹ is likely due to atmospheric CO₂ or weak overtone/combination bands and is not directly related to the silica structure.

A strong and sharp band at approximately 1402 cm⁻¹ can be assigned to O–H bending vibrations or possible residual impurities. The band around 654 cm⁻¹ corresponds to Si–O bending vibrations, which is a typical feature of silica materials. Although not explicitly marked, the region near 1000–1100 cm⁻¹ is expected to show intense Si–O–Si asymmetric stretching vibrations, confirming the formation of the silica network.

4. SEM Analysis:

Scanning Electron Microscopy (SEM) will be used to study:

- Surface morphology,
- Aggregation behaviour,
- Approximate particle size distribution

This will help in visualizing the topography of silica nanoparticles.

5. TEM Analysis:

Transmission Electron Microscopy (TEM) will provide:

- Accurate nanoparticle size,
- Particle shape,
- Degree of agglomeration

TEM will help confirm whether the synthesized nanoparticles are spherical and within the expected nanometre range.

CONCLUSION

Sonochemical synthesis is a modern and effective method for preparing SiO₂ nanoparticles for the removal of heavy metals from wastewater. In this method, ultrasound waves create tiny bubbles in the liquid, and when these bubbles collapse, they generate high energy. This energy helps to speed up chemical reactions and forms nanoparticles quickly. Because of this process, the particles produced are usually very small, uniform in shape, and well dispersed.

These silica nanoparticles have a high surface area, which makes them very useful for removing heavy metals such as chromium from water. The large surface area provides more active sites for adsorption, so metal ions can easily attach to the surface of the nanoparticles. This improves the efficiency of the treatment process compared to traditional methods, which are often slower and less effective.

These SiO₂ nanoparticles can be easily modified with different functional groups like amino or thiol groups. These modifications increase the attraction between the nanoparticles and heavy metal ions, leading to better removal performance. Due to this flexibility, silica nanoparticles can be designed for specific types of contaminants, making them suitable for different wastewater treatment needs.

Sonochemical synthesis offers a fast, efficient, and environmentally friendly approach for producing high-quality silica nanoparticles. It reduces reaction time, improves material properties, and enhances heavy metal removal efficiency. This method has strong potential for large-scale applications in wastewater treatment and can help in protecting both the environment and human health in the future.

FUTURE SCOPE

The future scope of sonochemical synthesis of SiO₂ nanoparticles for heavy metal removal is very wide and promising. One important direction is improving the efficiency of the ultrasound process by carefully controlling parameters like frequency, intensity, temperature, and reaction time. Better control can help produce nanoparticles with more uniform size, higher surface area, and improved adsorption capacity. Researchers can also work on reducing production cost so that this method can be easily used in large-scale wastewater treatment plants.

Another important area is the development of advanced surface modification techniques. By attaching different functional groups such as amino, thiol, or carboxyl groups, the selectivity of silica nanoparticles toward specific heavy metals can be increased. This means the material can be designed to remove only targeted pollutants like arsenic, lead, or mercury with higher efficiency. Combining SiO₂ nanoparticles with other materials such as magnetic particles, polymers, or carbon-based materials can improve their performance and allow easy separation after treatment.

Reusability and regeneration of nanoparticles is also a key focus for future research. Scientists need to develop simple and effective methods to recover used nanoparticles and reuse them multiple times without losing their adsorption capacity. This will reduce waste generation and overall treatment cost. Long-term stability and durability of these nanoparticles in different environmental conditions should also be studied to ensure reliable performance.

Future direction is testing this technology in real wastewater systems rather than only laboratory conditions. Real wastewater contains many different pollutants, which may affect the performance of nanoparticles. Therefore, pilot-scale and industrial-scale studies are required to understand practical challenges. Integration of this method with existing treatment technologies like filtration, biological treatment, or membrane systems can further improve efficiency.

Environmental safety is also an important concern. Future research should focus on understanding the possible environmental impact of nanoparticles after use. Proper disposal methods and eco-friendly synthesis techniques should be developed to avoid secondary pollution. Using green synthesis methods along with sonochemical techniques can make the process more sustainable.

These continuous research and technological improvements, sonochemical synthesis of SiO₂ nanoparticles has the potential to become a highly effective, economical, and environmentally safe method for heavy metal removal from wastewater. This approach can play a significant role in solving water pollution problems and ensuring safe water for future generations.

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