International Journal of Environmental Sciences
ISSN: 2229-7359
Vol. 11 No. 13s,2025
https://theaspd.com/index.php`

A Study Of Fabricating High-Melting-Point Nano Niobium Colloid By Micro-EDM

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Abstract– This study presents, for the first time, the successful fabrication of a nano-niobium colloid using a laboratory-developed micro electrical discharge machining (micro-EDM) system. A systematic comparison was conducted to evaluate the colloid's particle size distribution and suspension stability under different dielectric fluids. By employing the electrical spark discharge method at ambient temperature and atmospheric pressure, nano-niobium colloids were synthesized under varying process parameters using two different dielectric fluids. In this process, deionized water and distilled water served as the dielectric media. The high-temperature arc discharge melted the metal material, and the detached fragments combined with the dielectric fluid to form nano-sized colloidal particles. The fabricated samples were analyzed using UV-Vis spectroscopy and a Zetasizer to verify the effects of different dielectric fluids on the fabrication outcomes. The fabrication voltage was fixed at 140 V, the dielectric fluid volume was maintained at 120 mL, and the discharge duration was set to 3 minutes. When deionized water was used as the dielectric fluid and the discharge pulse duration (Ton-Toff) was set to 60–60 μs, the resulting colloid exhibited a characteristic absorption peak at 192 nm with an absorbance of 0.286. The average particle size was measured to be 64.92 nm, and the zeta potential was recorded as −58.3 mV. Similarly, when distilled water was used under the same discharge conditions, the colloid exhibited a characteristic absorption peak at 192 nm with an absorbance of 0.159. The average particle size was measured to be 58.38 nm, and the zeta potential was recorded as −53.0 mV. The fabrication of nano-niobium colloids using the electrical spark discharge method is simple and rapid, requiring no additional chemical reagents. This process minimizes environmental and human health hazards to effectively zero during fabrication.
Keywords–Electrical Spark Discharge Method, Nano Niobium Colloid, Micro-EDM, Dielectric Fluid, Colloid Stability.

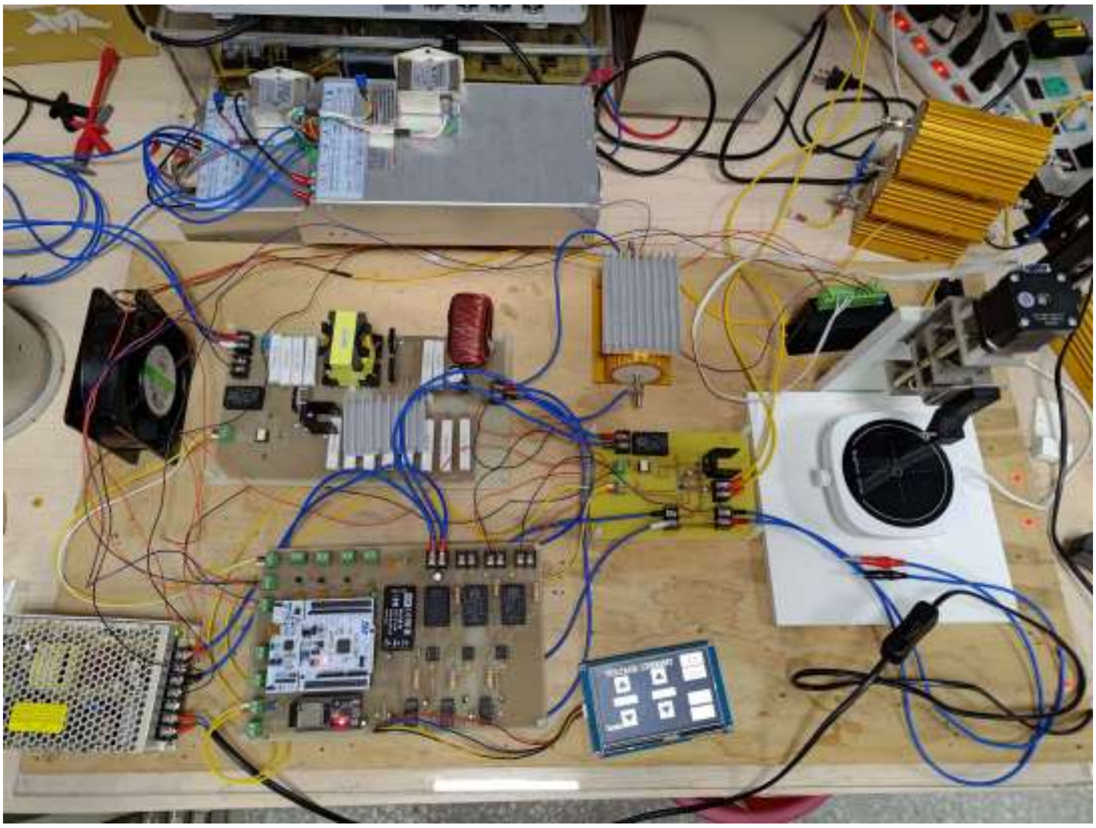
INTRODUCTION

Nanotechnology is generally categorized into three major domains: nano-devices, nanomaterials, and nano-characterization techniques. Nano-devices are developed by either bottom-up assembly from atoms or molecules, or through top-down approaches such as microfabrication that reduce the scale of functional components. Common forms of nanomaterials include nanotubes, nanorods, nanobelts, nanorings, nanofibers, and nanowires [1]–[3]. Nano-characterization and analysis techniques are essential for investigating structural, physical, and chemical properties at the nanoscale.Materials with structural dimensions ranging from 1 to 100 nanometers in any spatial direction are classified as nanomaterials. This size range bridges the macroscopic and microscopic domains and is commonly referred to as the field of nanotechnology. When a metal is reduced to the nanoscale, it exhibits a variety of distinct physical and chemical properties. This is primarily due to the significant increase in surface area and the higher proportion of atoms exposed on the particle surface. As a result, the surface activity of the material increases, giving rise to unique behaviors not observed in the bulk form. The reduction in particle size, increase in surface-to-volume ratio, and changes in electronic bandgap collectively alter the physical, thermal, electrical, chemical, and optical properties of nanomaterials compared to their bulk counterparts [4]–[6].Niobium is a silver-gray transition metal characterized by a high melting point (approximately 2,477 °C), moderate density (approximately 8.57 g/cm^3), and excellent ductility. It also exhibits good corrosion resistance and electrical conductivity. At room temperature, niobium readily forms a stable oxide layer that provides surface protection [7]. Due to these properties, niobium is widely used in the steel industry for producing high-strength low-alloy steels and superalloys to enhance heat resistance and corrosion tolerance. These niobium-containing alloys are extensively applied in the automotive, aerospace, and nuclear industries [8].In recent years, niobium-based nanomaterials such as nano-niobium colloids and nano-sized Nb_2O_5 have attracted increasing attention due to their unique optical, electrical, and catalytic properties. Nano-niobium has demonstrated significant potential for applications in superconducting electronic components, such as superconducting magnets and superconducting quantum interference device (SQUID) sensors. It is also utilized in high-performance energy storage devices, including supercapacitors and lithium-ion battery electrodes. Furthermore, nano-niobium materials are being explored for use in electromagnetic interference (EMI) shielding, sensors, catalysts, optoelectronic devices, and biomedical materials, highlighting both their research potential and industrial value [9].

RESEARCH METHOD

Compared to conventional electrical discharge machining (EDM) systems, the laboratory-developed micro-EDM system used in this study offers several notable advantages:Innovative user interface design: A human–machine interface (HMI) is employed in place of a traditional button-based control panel, enabling a more compact system design and improving operational intuitiveness. This allows researchers to precisely control experimental parameters with greater ease.Precise discharge voltage control: A forward converter with electrical isolation is integrated into the system, allowing the discharge voltage to be set via the HMI. The STM32-F446RE microcontroller reads the feedback voltage and adjusts the duty cycle accordingly, enabling high-precision discharge voltage regulation.Real-time monitoring of discharge success rate: A current sensing integrated circuit (IC) is employed to monitor discharge events and success rate in real time. This enables researchers to evaluate and adjust experimental conditions promptly, thereby improving the efficiency and stability of nano-colloid fabrication.Real-time data management and analysis: By incorporating Internet of Things (IoT) technology, fabrication parameters, discharge success counts, and spectral data are automatically uploaded to a private database, facilitating centralized data management and subsequent analysis.Expanded applicability to various metals: By increasing the adjustable discharge voltage range and raising the maximum discharge voltage, the system has been successfully validated for the fabrication of nano-colloids from high-melting-point metals such as niobium. Compact, lightweight, and cost-effective design: The system is small in size, lightweight, and portable, making it suitable for laboratory environments. Additionally, its manufacturing cost is significantly lower compared to conventional industrial EDM machines. The physical appearances of the industrial EDM machine and the developed micro-EDM system are shown in Fig. 1(a) and Fig. 1(b), respectively.

(a)



(b)

Fig. 1. Actual appearance of electrical discharge machining systems: (a) industrial EDM machine, (b) laboratory-developed micro-EDM system.

A niobium wire with 99.99% purity was selected as the electrode material to ensure that no extraneous elements would interfere with the analysis of the colloid. In terms of experimental conditions, the discharge voltage was set to 140 V, the volume of dielectric fluid was 120 mL, and the total discharge duration was 3 minutes. Various combinations of Ton-Toff parameters were applied during fabrication.

The fabricated colloidal samples were analyzed using a UV-Vis spectrophotometer to determine their absorption peak positions and intensities. A Zetasizer was employed to measure particle size and zeta potential, which were used to evaluate the suspension stability and the size distribution of the nano-metal particles.This study adopted a multivariable control and measurement approach, leveraging the experimental advantages of the micro-EDM platform to explore the optimal parameters for fabricating nano-niobium colloids via electrical spark discharge. The results aim to establish a solid experimental foundation for future application-oriented research.The electrical discharge machining (EDM) system processes conductive materials through spark discharges. Its operating principle is based on the conversion of electrical energy into thermal energy. One key advantage of EDM is that material removal occurs without direct contact between the electrode and the workpiece, as illustrated in Fig. 2.

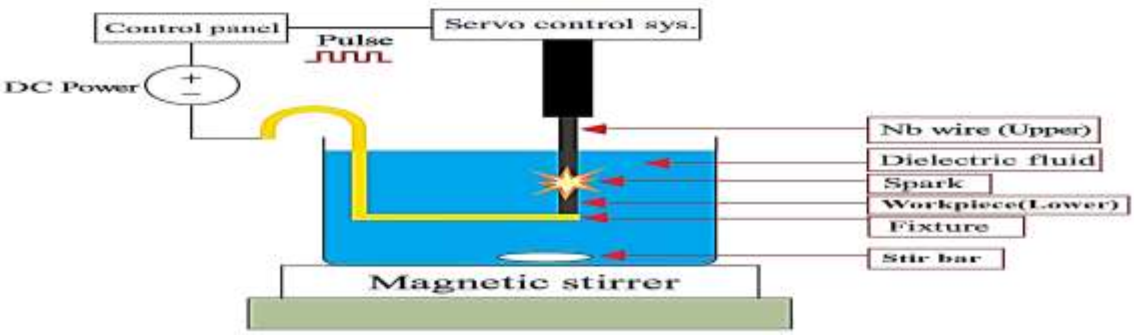


Fig. 2. Schematic diagram of the electrical discharge machining (EDM) system.

The dielectric fluid selected for the experiment was either deionized water or a highly insulating liquid [11]. In the experimental setup, conductive metallic materials were placed between the upper and lower electrodes of the EDM system, with both electrodes submerged in the dielectric fluid. A servo control system was used to adjust the distance between the upper and lower electrodes, bringing them to a narrow gap of approximately 30 μm or less.At this stage, a periodic pulsed DC voltage is applied between the two electrodes. When the electric field strength exceeds the dielectric breakdown strength of the fluid, a discharge occurs at the point of minimum gap, forming a plasma channel and a discharge column—this phenomenon is known as arc discharge [12]. During the discharge, an extremely high temperature of approximately 5,000–6,000 K is generated instantaneously [13], causing localized melting of both the electrode and the workpiece. Simultaneously, the surrounding dielectric fluid vaporizes and expands, generating an expansion pressure of approximately 40–50 kg/cm² [14]–[15].The molten metal material is ejected into the dielectric fluid due to impact forces and rapidly cools and solidifies into fine metal particles, which remain suspended in the fluid. As the Ton-Toff discharge cycles repeat, continuous material removal occurs between the two electrodes, eventually leading to the formation of a nano-metal colloid in the dielectric medium. To enhance colloidal dispersion, a magnetic stirring system was installed at the bottom of the container to promote uniform distribution of the suspended nanoparticles.The electrical spark discharge process is illustrated in Fig. 3. The voltage and current across the electrodes are denoted as Vgap and Igap, respectively, while Ton and Toff represent the on-time and off-time of the voltage pulse.

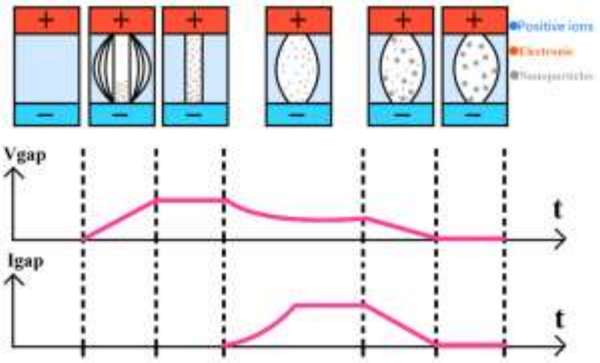


Fig. 3. Electrical spark discharge sequence of the EDM system.

Preparation for Discharge: Conductive materials were placed on the upper and lower electrodes, which were then immersed in the dielectric fluid. After connecting the electrodes to the power supply, it was necessary to confirm proper alignment between the electrode surfaces to avoid discharge failure. The upper electrode moved slowly toward the lower electrode, gradually narrowing the gap. As a result, the gap voltage (Vgap) increased slowly, but the electric field strength remained below the dielectric breakdown threshold, keeping the fluid in an insulating state.Discharge Initiation: When the inter-electrode distance was reduced to approximately 0.1–0.05 μm, the electric field strength exceeded the dielectric breakdown strength. This triggered dielectric breakdown, and electrons were emitted from the surface of the lower electrode toward the upper electrode. Atoms or molecules in the dielectric fluid were ionized by the resulting energy, leading to the gradual formation of a discharge channel.Ionization Process: As the electric field exceeded the dielectric's insulation strength, the fluid's insulating properties collapsed. Electrons traveled from the lower to the upper electrode, colliding with neutral atoms in the gap. These collisions excited outer valence electrons, generating positive ions and additional free electrons, thereby reinforcing the discharge channel.Melting and Particle Ejection: The free electrons and positive ions traveled toward the upper and lower electrodes, respectively. Upon impact with the electrode surfaces, their kinetic energy was instantly converted into thermal energy, forming an arc. This caused localized melting or vaporization, leading to explosive ejection of metal particles from the electrode surfaces into the dielectric fluid, where they rapidly solidified into nanoparticles.Discharge Termination: As the Toff phase began, both Vgap and Igap gradually decreased. With the cessation of electron emission from the electrodes, the discharge channel quickly collapsed. The generated nanoparticles remained suspended in the dielectric fluid as the system transitioned back to an insulating state.Restoration of Insulation: When the Toff phase ended, both Vgap and Igap returned to zero. The inter-electrode gap regained its dielectric insulation, allowing the nanoparticles to remain suspended in the fluid while awaiting the next discharge pulse cycle.To further illustrate the rationale behind the selection of dielectric fluids, the electrical conductivity of both deionized water and distilled water was measured under identical ambient conditions, as shown in Fig. 4. The slight difference in conductivity is expected to influence the dielectric breakdown threshold and the ionization environment during spark discharge.

(a)

(b)

Fig. 4. Measured electrical conductivity of dielectric fluids: (a) deionized water, (b) distilled water.

In theory, a lower dielectric fluid conductivity generally contributes to a more stable and concentrated discharge process, thereby favoring the formation of stable colloids. However, in this study, the niobium colloids fabricated with distilled water (with higher conductivity) exhibited better zeta potential results. This outcome may be attributed to differences in material-specific plasma interactions, surface oxidation behavior, or trace impurities in the fluid that affect nucleation dynamics. These findings suggest that conductivity alone may not sufficiently predict colloidal stability in high-melting-point metallic systems.

RESEARCH RESULTS AND DISCUSSION

In this study, nano-niobium colloids were fabricated using the electrical spark discharge method via a micro-EDM system. A Zetasizer was employed to measure the nanoparticle size and zeta potential of the colloid. When the absolute value of the zeta potential exceeds 30 mV, the colloidal nanoparticles are considered to be in a stable suspended state. A UV-Vis spectrophotometer was used to examine the absorption intensity and characteristic peaks of the samples; this instrument provides insights into the material's transmittance and reflectance properties.

The analysis results of nano-niobium colloids synthesized under different discharge parameters using deionized water as the dielectric fluid are shown in Table I. Similarly, the results for colloids prepared with distilled water as the dielectric fluid are presented in Table II.

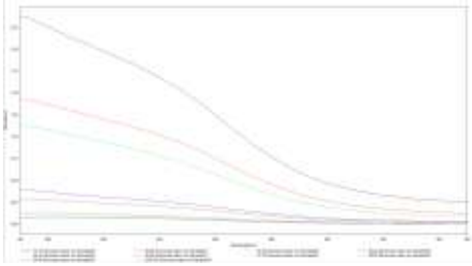
TABLE I.EXPERIMENTAL DATA OF NANO-NIOBIUM COLLOIDS PREPARED WITH DEIONIZED WATER

Deionized water										
Ton-Toff	10-10	20-20	30-30	40-40	50-50	60-60	70-70	80-80	90-90	100-100
Size(d.nm)	83.53	72.98	85.01	77.38	73.57	64.92	70.46	60.45	56.24	74.37
Potential(mv)	-54.4	-60.0	-62.0	-51.2	-56.0	-58.3	-28.3	-32.6	-30.2	-29.3
Absorbance	2.363	1.432	1.133	0.39	0.292	0.286	0.138	0.121	0.075	0.085
Wavelength(nm)	192	192	192	192	192	192	192	192	192	192
time(min)	3	3	3	3	3	3	3	3	3	3
Volume of the dielectric fluid(ml)	120	120	120	120	120	120	120	120	120	120

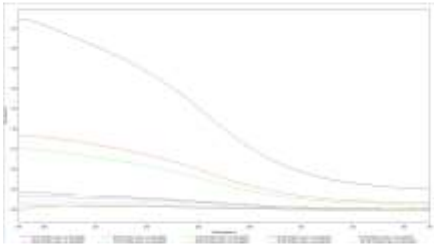
TABLE II.EXPERIMENTAL DATA OF NANO-NIOBIUM COLLOIDS PREPARED WITH DISTILLED WATER

Distilled water										
Ton-Toff	10-10	20-20	30-30	40-40	50-50	60-60	70-70	80-80	90-90	100-100
Size(d.nm)	85.39	80.02	71.23	76.05	69.87	58.38	71.2	59.87	53.06	64.72
Potential(mv)	-52.4	-54.8	-55.7	-57.6	-64.6	-53.0	-51.9	-57.2	-55.1	-47.7
Absorbance	2.368	0.922	0.751	0.214	0.197	0.159	0.097	0.048	0.042	0.072
Wavelength(nm)	192	192	192	192	192	192	192	192	192	192
time(min)	3	3	3	3	3	3	3	3	3	3
Volume of the dielectric fluid(ml)	120	120	120	120	120	120	120	120	120	120

UV-Vis analysis results showed that all nano-niobium colloid samples exhibited a characteristic wavelength at 192 nm. The absorbance values of colloids synthesized under different discharge parameters are illustrated in Fig. 5(a) and Fig. 5(b).Nano-niobium colloid samples prepared under Ton-Toff discharge cycles ranging from 10–10 to 100–100 μs were analyzed using a Zetasizer. Among the tested parameters, the Ton-Toff 60–60 μs condition yielded both small particle size and high suspension stability. According to the analysis results, the colloid prepared with deionized water exhibited an average particle size of 64.92 nm and a zeta potential of −58.3 mV, as shown in Fig. 6(a) and Fig. 6(b). In comparison, the colloid synthesized with distilled water showed an average particle size of 58.38 nm and a zeta potential of −53.0 mV, as illustrated in Fig. 7(a) and Fig. 7(b).

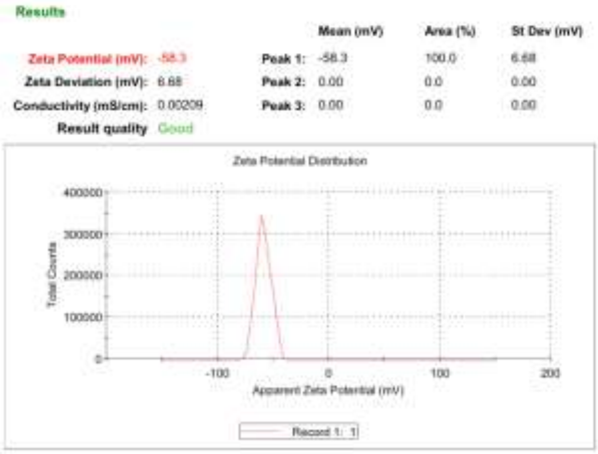


(a)

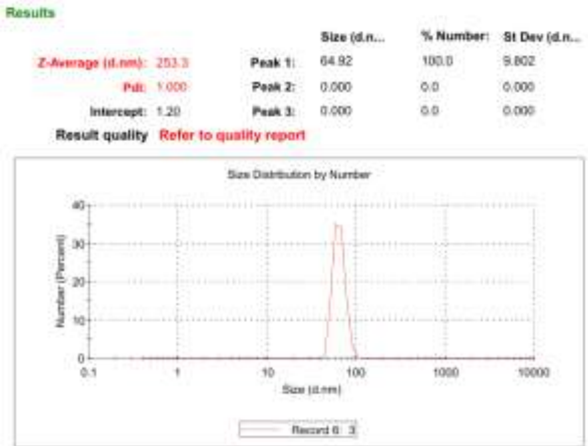


(b)

Fig. 5. UV-Vis analysis of nano-niobium colloids: (a) prepared with deionized water, (b) prepared with distilled water

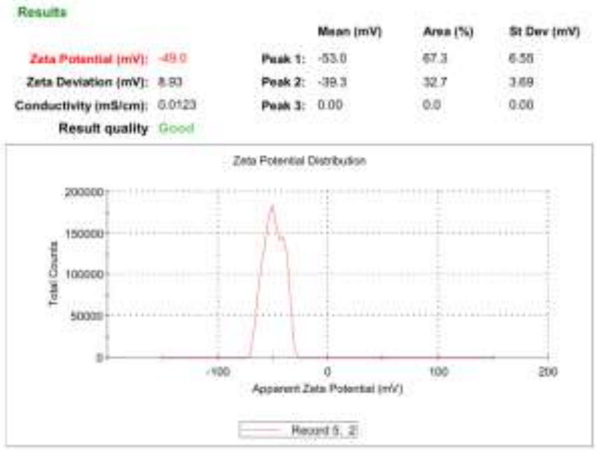


(a)

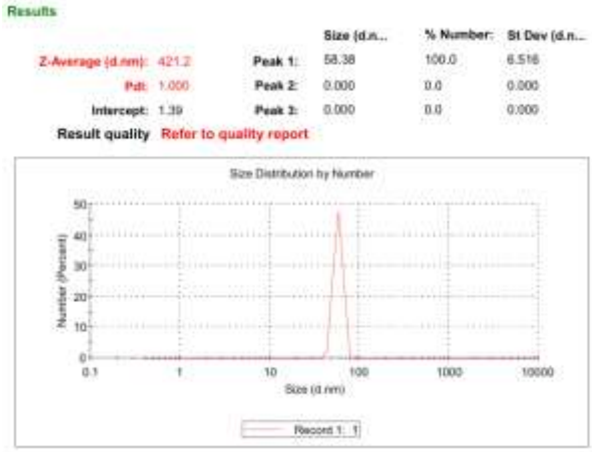


(b)

Fig. 6. Zetasizer analysis of nano-niobium colloids prepared with deionized water: (a) zeta potential, (b) particle size distribution



(a)



(b)

Fig. 7. Zetasizer analysis of nano-niobium colloids prepared with distilled water: (a) zeta potential, (b) particle size distribution

Comparative Discussion with Previous ZnO–TiO_2 Research

In a previous study conducted by the same research group, zinc oxide–titanium dioxide (ZnO–TiO_2) colloids were successfully synthesized via spark discharge using an industrial-scale EDM machine under ambient conditions. In contrast, the current study utilized a self-developed micro-EDM platform, which is significantly more compact and cost-efficient. Despite this difference in equipment scale, the nano-niobium colloids fabricated in this work exhibited comparable or superior colloidal stability.Although niobium and zinc oxide–titanium dioxide represent fundamentally different material systems—the former being a metallic nano-colloid and the latter a semiconductor oxide composite nano-colloid—the fabrication process using the same micro-EDM platform successfully yielded stable colloidal suspensions in both cases. This result demonstrates the micro-EDM system's robustness and versatility in adapting to materials with diverse electronic structures, conductivity, melting points, and chemical activities. Such findings provide a strong experimental foundation for future extensions toward a broader range of high-melting-point metals and functional oxide materials.Furthermore, although both studies adopted the same ±30 mV zeta potential threshold for evaluating colloidal stability, the niobium colloids in this study demonstrated superior suspension stability under optimized discharge parameters—despite the inherent challenges of processing high-melting-point metals.The successful fabrication of both oxide-based and metallic colloidal systems using the same micro-EDM platform underscores its adaptability and positions it as a promising tool for synthesizing a wider spectrum of functional nanomaterials.These results validate the micro-EDM system's capability to fabricate functional nanomaterials with high reproducibility and stability, even when handling refractory metals like niobium. The ability to achieve such performance using a miniaturized and low-cost setup further highlights its potential for versatile and scalable nano-colloid synthesis.

CONCLUSIONS

Under ambient temperature and pressure, nano-colloids were fabricated by melting metallic electrodes through arc discharge, using deionized water and distilled water as dielectric fluids. Compared to industrial-scale EDM systems, the micro-EDM system offers a more compact form factor, lower cost, and a wider range of selectable discharge voltage levels. By varying the discharge pulse parameters, the relationship between particle size and suspension stability was investigated. Finally, the characteristics of the fabricated samples were analyzed using a Zetasizer and a UV-Vis spectrophotometer. The conclusions of this study are summarized as follows:

1. The study confirms that the electrical spark discharge method is a stable and effective technique for fabricating nano-metal colloids.
2. Niobium metal particles are relatively large and difficult to process into the nanoscale range, requiring multiple attempts to identify suitable fabrication parameters.
3. Compared with previous studies, the results verify that changing the dielectric fluid does not necessarily lead to significant differences in colloid fabrication outcomes.
4. The study found that a Ton–Toff setting of 60–60 μs yielded colloids with both smaller particle size and relatively high suspension stability. Using deionized water as the dielectric fluid, the colloids exhibited an average particle size of 64.9 nm and a zeta potential of −58.3 mV. In comparison, colloids prepared with distilled water showed an average particle size of 58.4 nm and a zeta potential of −53.0 mV.
5. The fabrication of nano-niobium colloids using the electrical spark discharge method is simple and rapid. The process does not require the addition of chemical agents, thereby minimizing potential hazards to both the environment and human health.

In addition to structural evaluation, the optoelectronic and photocatalytic properties of the nano-niobium colloids will be explored through performance testing in potential applications such as photoelectrodes, sensors, and energy storage devices. Finally, process optimization strategies including real-time discharge monitoring, closed-loop control systems, and batch automation will be investigated to assess the feasibility of scaling up toward industrial production and commercialization.

COMPETING INTERESTS

The authors declare that there is no conflict of interest regarding the publication of this paper.

FUNDING

The Ministry of Science and Technology (NSTC 113-2622-E-027-024 -).

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