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Research on the Synthesis of Nanocomposite Colloids by Micro EDM

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*Abstract– In this study, a micro electrical discharge machine (micro-EDM) was employed to fabricate zinc oxide–titanium dioxide (ZnO–TiO_2) nanocomposite colloids using the Electrical Spark Discharge Method (ESDM) in a deionized water dielectric. High-purity (99.99%) zinc and titanium wires were used as electrodes, and the discharge process was conducted under ambient temperature and atmospheric pressure conditions. Systematic experiments were performed to investigate the effects of various discharge pulse parameters (Ton–Toff) and discharge voltages. The particle size and zeta potential of the colloids were measured using a laser scattering particle size analyzer (Zetasizer), and their optical properties were characterized with a UV-Visible (UV-Vis) spectrophotometer. The results showed that the colloid fabricated under a Ton–Toff of 50–50μs and a discharge voltage of 160V exhibited the best suspension stability, achieving a zeta potential of 29.8mV and an average particle size of approximately 76.57*nm. *Overall, this study confirms that ESDM enables the rapid and environmentally friendly synthesis of high-purity nanocomposite colloids without the need for chemical additives, demonstrating significant potential for applications in biomedical materials and environmental fields.* Keywords– *Electrical Spark Discharge Method, Micro Electrical Discharge Machine, Nano zinc oxide-titanium dioxide colloid, Zeta Potential , UV-Vis Spectroscopy.*

INTRODUCTION

Titanium and zinc metals exhibit excellent properties even before nanostructuring. Titanium is renowned for its lightweight characteristics, corrosion resistance, and tolerance to extreme temperatures, while zinc demonstrates strong electrochemical activity and corrosion resistance. After nanostructuring into zinc oxide (ZnO) and titanium dioxide (TiO_2), the crystal sizes are reduced to the nanoscale, significantly increasing their surface area and surface energy. These changes alter their electronic structures and surface activities, resulting in pronounced nanoscale effects[1]. Such effects have led to the widespread application of ZnO and TiO_2 in fields such as photocatalysis and biomedicine[2].Titanium dioxide (TiO_2) is one of the most widely used photocatalytic materials. Under ultraviolet (UV) light irradiation, TiO_2 generates a photocatalytic effect by absorbing photon energy and forming electron-hole pairs. The holes can further generate oxygen molecules or hydroxyl radicals (OH radicals) with strong oxidative abilities, while the free electrons can produce hydrogen peroxide (H_2O_2) or superoxide anions (O_2^-) in the presence of oxygen, both exhibiting high oxidative potential[3]. Through these mechanisms, TiO_2 can effectively decompose harmful organic substances and purify environmental pollutants.Although ZnO and TiO_2 exhibit similar functionalities in the field of photocatalysis, their applications differ slightly. ZnO is primarily used in antibacterial agents and ultraviolet protection products (such as sunscreens)[4], whereas TiO_2 is extensively applied in bone implant coatings[5]-[6], pollutant degradation, and antimicrobial textiles[7]-[8]. Studies have shown that heterogeneous composite structures such as ZnO-TiO_2 can synergistically enhance light absorption and catalytic efficiency, promoting advancements in green energy and smart materials[9]-[11].This study employs the Electrical Spark Discharge Method (ESDM) to fabricate nanocolloids. ESDM is a purely physical process that generates high-temperature electric arcs through high-voltage pulsed discharges in a dielectric liquid, vaporizing the metallic electrode material, which is then rapidly quenched and condensed into nanoparticles. The entire process occurs within the dielectric fluid, effectively preventing nanoparticle dispersion and enabling the production of stable nanocolloids without the addition of chemical dispersants. Compared with conventional chemical synthesis methods, ESDM is more environmentally friendly and efficient, and has been successfully applied to the fabrication of nanocolloids of pure metals such as gold, silver, titanium, and their composites[12].

In this study, ZnO-TiO_2 nanocomposite colloids were fabricated using ESDM, and the optimal fabrication parameters were investigated. The colloidal properties were analyzed to validate the technical feasibility and contributions of this method.

PAGE LAYOUT

In this study, ZnO-TiO_2 nanocomposite colloids were fabricated using a laboratory-built micro-EDM. The fabrication setup is shown in Fig. 1. A beaker containing dielectric fluid and a stirring rod was placed on an electromagnetic stirrer. The upper electrode was a zinc wire, and the lower electrode was a titanium wire. During fabrication, both electrodes were immersed in the dielectric fluid and connected to the positive and negative terminals of a pulsed power supply. A control system monitored the current signal between the electrodes and drove a stepper motor to maintain an appropriate interelectrode gap, ensuring stable spark discharge generation[12]-[13].

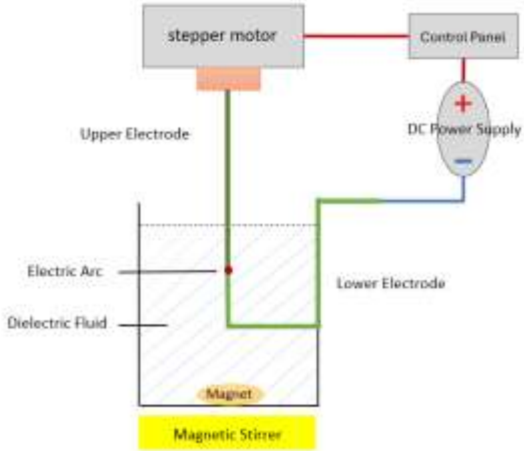
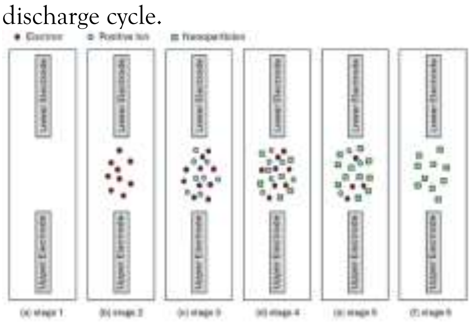


Fig. 1 Schematic diagram of the electrical discharge machine.

The electrical spark discharge process can be divided into Ton and Toff stages, as illustrated in Fig. 2:
(1)Preparation stage: The interelectrode gap is maintained in an insulating state, and no gap current (Igap) flows.
(2)Initiation stage: As the electrodes approach each other, the electric field strength increases, leading to dielectric breakdown and the formation of an initial electron flow, establishing a discharge channel[14].(3)Ionization stage: The high-intensity electric field accelerates electrons, causing collisions with dielectric fluid molecules and generating a large number of positive ions and free electrons. During this stage, Igap rapidly increases, and the gap voltage (Vgap) significantly decreases[14].(4)Spark discharge stage: Electrons and ions collide with the electrodes at high speed, creating localized high temperatures that vaporize the electrode materials. The vaporized metals rapidly condense in the dielectric fluid to form nanoparticles. During this phase, Vgap remains constant and Igap stays at its peak value.(5)Termination stage: No voltage is applied between the electrodes, and both the voltage and current drop to zero. The dielectric fluid returns to its insulating state, with nanoparticles suspended in the fluid.(6)Insulation recovery stage: The dielectric fluid fully restores its insulating properties, preparing for the next discharge cycle.



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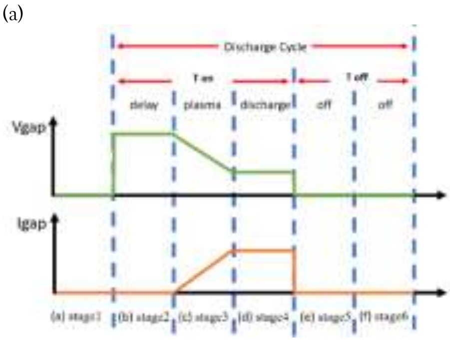


(b)

Fig. 2 Schematic diagram of the electrical spark discharge process. (a) Steps of EDM(b) Relationship Between Vgap and Igap During the Discharge Process

The experimental micro-EDM apparatus used in this study is shown in Fig. 3. The discharge voltage range was set between 90V and 180V, and the Ton–Toff time was adjustable between 10–100μs. These parameters were used as variable factors in the fabrication of the nanocolloids. The user interface of the EDM system is shown in Figure 4.The materials used in the experiments included 99.99% pure zinc and titanium wires as electrodes. The dielectric fluid was deionized water(DW) with a volume of 80mL and a conductivity of 4–10μS/m. All fabrication processes were carried out under ambient temperature and atmospheric pressure conditions, with a fabrication time of 2 minutes.

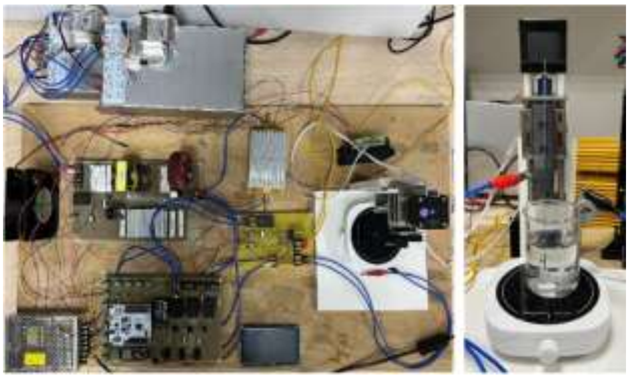


Fig. 3 Photograph of the experimental micro-EDM apparatus.

Fig. 4 User Interface of the micro-EDM System.

RESULTS AND DISCUSSION

In this study, zinc oxide–titanium dioxide ($ZnO–TiO_2$) nanocomposite colloids were fabricated using an EDM system via the ESDM. A laser scattering particle size analyzer (Zetasizer Nano System, Zetasizer) was used to measure the particle size and surface charge (zeta potential) of the nanoparticles[15]. A higher zeta potential indicates better colloidal suspension stability, and a colloid with an absolute zeta potential value exceeding 30mV is generally considered to have good stability [16]. Ultraviolet-visible spectroscopy (UV-Vis) was used to analyze the absorbance intensity and characteristic peaks of the samples, providing information on their transmittance and reflectance properties[17].In this study, 10 different Ton–Toff pulse cycle parameter sets and 8 different discharge voltage settings were designed, all based on a 50% duty cycle, to fabricate $ZnO–TiO_2$ nanocolloids. The goal was to determine the optimal fabrication parameters by analyzing the colloidal properties. A high-quality nanocolloid requires good suspension stability, with the absolute value of the zeta potential (either positive or negative) typically exceeding ±30mV, indicating sufficient electrostatic repulsion between particles to prevent aggregation[18]. This study focused on zeta potential analysis to identify the nanocolloids with the best suspension stability. The fabrication parameters, materials used, and environmental conditions are summarized in Table 1.

TABLE I
PREPARATION PARAMETERS FOR $ZnO–TiO_2$ NANOCOLLOIDS

Experimental parameter	Values
Volume of the dielectric fluid	Deionized Water: 80 mL
Electrode diameter	pure magnesium wire (99.9%): 1 mm
Ton–Toff	10-10、20-20、30-30、40-40、50-50、60-60、70-70、80-80、90-90、100-100 (μs)
Peak Current setting (IP)	IP1、IP2、IP3、IP4、IP5、IP6、IP7
Discharge Voltage	110 V、120 V、130 V、140 V、150 V、160

	V、170 V、180 V
Preparation time	2 min
Temperature	25°C (room temperature)
Atmospheric pressure	1 atm

In the Ton–Toff parameter experiments, the discharge voltage was fixed at 120V, and colloid samples were fabricated using Ton–Toff values ranging from 10–10μs to 100–100μs in 10 different pulse cycle settings. The zeta potential (V_ζ) and particle size distribution analysis results for these colloids are shown in Table 2. As seen in Table 2, the particle sizes of all samples were less than 100nm; however, the absolute values of $|V_\zeta|$ did not exceed 30mV, indicating that none of the samples achieved good suspension stability. Among them, the sample fabricated at 50–50μs exhibited the highest $|V_\zeta|$ value and was therefore selected as the baseline parameter for the subsequent discharge voltage experiments.

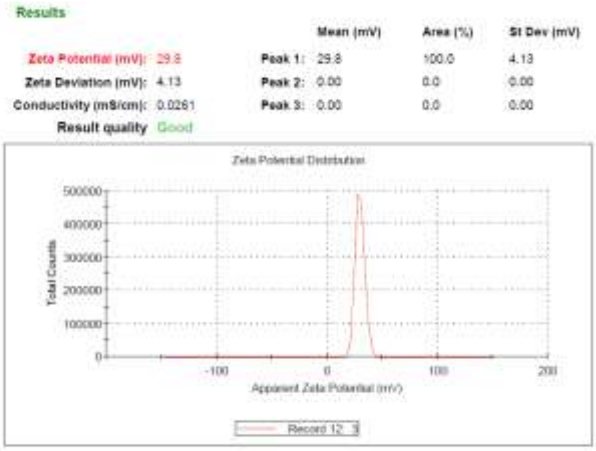
TABLE II
COLLOIDAL PROPERTIES UNDER DIFFERENT TON–TOFF PARAMETERS (DISCHARGE VOLTAGE = 120V)

Ton-Toff settings	**V_ζ(mV)**	**Particle size–number distribution**	
		>100nm(%)	**Particle size(d.nm)**
10 -10μs	19.5	99.8	56.18
20 -20μs	19.2	99.7	72.23
30 -30μs	17.0	99.9	59.16
40 40μs	16.6	100.0	54.01
50 -50μs	22.7	99.9	51.83
60 -60μs	19.1	100.0	52.61
70 -70μs	20.8	99.9	52.76
80 -80μs	20.1	99.7	70.41
90 90μs	20.5	99.9	48.51
100 -100μs	21.0	99.7	61.34

For the discharge voltage parameter experiments, the Ton–Toff was fixed at 50–50μs, while the discharge voltage was varied from 110V to 180V, increasing by 10V for each colloid sample. The values and particle size distribution analysis results are summarized in Table 3.As shown in Table 3, as the discharge voltage increased, the absolute value of the zeta potential | | also increased. When the discharge voltage reached 160V, the colloid sample achieved the best performance, with a zeta potential of 29.8mV, as shown in Fig. 5(a). The corresponding particle size was 76.57nm. According to the UV-Vis analysis, the colloid exhibited a characteristic absorption wavelength at 238nm, with a maximum absorbance of 2.236, as illustrated in Fig. 5(b) and Fig. 5(c).

TABLE III
COLLOIDAL PROPERTIES UNDER DIFFERENT DISCHARGE VOLTAGE PARAMETERS (TON–TOFF = 50–50 μS)

Discharge voltage	**V_ζ (mV)**	**Particle size–number distribution**	
		>100nm(%)	**Particle size(d.nm)**
110V	22.7	99.8	64.05
120V	23.0	99.9	54.36
130V	23.7	99.8	77.86
140V	24.9	99.9	59.29
150V	25.2	99.8	70.37
160V	29.8	99.8	76.57
170V	26.0	99.9	60.41
180V	27.5	99.8	67.47



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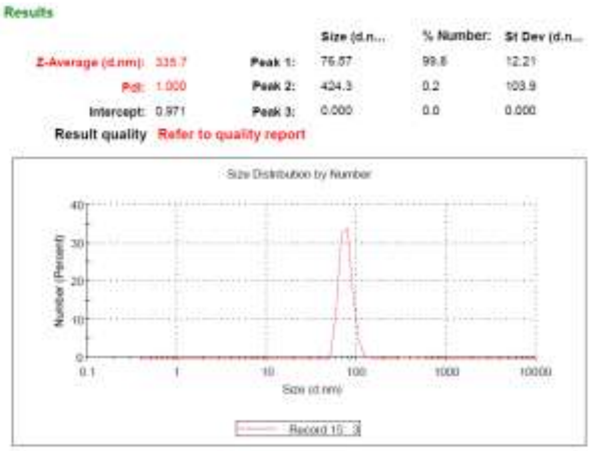

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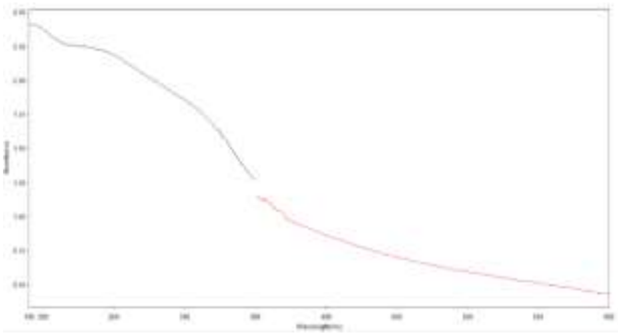

(c)

Fig. 5 Characterization of Samples Prepared at a Discharge Voltage of 160V (a) Zeta Potential, (b) Size Distribution, (c) UV-Vis Spectroscopy Results

CONCLUSIONS

In this study, a laboratory-built micro-EDM was utilized under ambient temperature and atmospheric pressure conditions to fabricate nanocolloids using deionized water as the dielectric fluid. Metal electrodes were melted by electrical arc discharge to generate the nanocolloids. Compared to other fabrication methods, this physical fabrication approach is faster and simpler, and because both the fabrication and collection processes are carried out entirely in DW, the issue of nanoparticle dispersion into the environment is effectively avoided. By varying the discharge pulse parameters, this study investigated the optimal pulse settings for achieving the best suspension stability. The characteristics of the fabricated samples were analyzed using a laser scattering particle size analyzer (Zetasizer Nano System) and a UV-Visible spectrophotometer (UV-Vis). The conclusions of this study are as follows:
(1) This study successfully demonstrated that high-purity and compositionally simple $ZnO–TiO_2$ nanocomposite colloids can be fabricated via ESDM under ambient conditions, effectively avoiding contamination from chemical reagents.

(2) The ESDM process offers significant advantages over traditional chemical synthesis methods, being simpler, more environmentally friendly, and more efficient in fabrication.
(3) Titanium particles were relatively larger and difficult to process into the nanoscale range, indicating that further optimization of the fabrication parameters is necessary.
(4) The zeta potentials of the fabricated colloids in this study did not exceed 30 mV; therefore, further improvements are needed to enhance the suspension stability of the colloids.

COMPETING INTERESTS

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

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