

# Stability analysis of platinum nanoparticles prepared by ESDM in deionised water

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Electrical discharge machine was used to prepare platinum nanocolloid by electric spark discharge method (ESDM) at room temperature. Preparation of platinum nanocolloid needed only traditional electrical discharge machine, platinum wire (99.5%) and magnet mixer. Then verify whether platinum nanoparticles be could successfully prepared under different preparation and their suspension stability. The optical properties and zeta potential were measured by UV-vis and Zetasizer, and their characteristic peaks (surface plasmon resonant), while suspension stability was analysed. The results of transmission electron microscope showed size, shape and dispersibility of platinum nanoparticles with a size of mostly <10 nm. X-ray diffraction was used to measure crystal lattice structure and component of platinum nanoparticles. In this work, platinum nanocolloid in conditions of normal and centrifuge were analysed. Zeta potential of normal colloid was decreased from -29.5 to -18.8 mV after precipitation, while zeta potential of the centrifuged colloid still maintained at -40 mV, confirming the good suspension stability of platinum nanocolloid prepared by ESDM after centrifugation.

**1. Introduction:** Platinum has the properties of good ductility, high density and low oxidation activity. It has extremely high corrosion resistance at high temperatures. In addition, its production is very scarce, so the price is very expensive. The current application of platinum includes conductive electrodes to increase their sensitivity to reactants [1–4], contact resistance and conductivity of individual platinum nanowires embedded in coplanar waveguide structures are investigated at high frequencies [5]. In terms of biomedical, platinum nanoparticle through biosynthesis was used as a therapeutic drug for breast cancer [6]. Platinum nanoparticle was used as a catalyst to enhance the antibacterial effect of silver nanoparticles [7]. There are many others application such as magnetic beads in the separation of platinum nanoparticles [8], platinum nanofilm in temperature sensors [9] and surface ionisation gas detection on platinum and metal oxide surfaces [10].

Although platinum can be applied to industrial, biomedical and catalyst, they are mostly prepared by chemical methods. If there are any chemical agents in platinum nanoparticles, they may cause damage to the human body and pollute environments. Therefore, it is an important issue to look for the preparation of platinum nanocolloid without pollution and toxicity. Different from chemical preparation methods [11–13], electric spark discharge method (ESDM) is a fast and easy preparation of platinum nanoparticles. Easy-collection and mass-production of nanoparticles are huge advantages. The main principle of ESDM is to apply a periodic high-frequency voltage pulse between the electrode and the workpiece. Any conductive materials can be used for ESDM [14]. Through the discharge arc between the work piece and electrode immersed in the dielectric fluid, the surface of metal was eroded and melted into nanoparticles at high temperature [15]. In this Letter, only traditional electric discharge machine and magnet mixer were used. Deionised water (DI water) was used as dielectric fluid, and platinum wire was used as material. Without adding any chemicals, platinum nanocolloid with an average size of <10 nm can be prepared in a green process [16]. The suspension stability (i.e. zeta potential) verified whether nanoparticles can stably suspend in nanocolloid. Therefore, platinum nanoparticles prepared under different conditions were analysed to confirm the difference in suspensibility of the nanoparticles.

## 2. Experiment

2.1. Experimental system: Electrical discharge machine system was used in this Letter, as shown in Fig. 1. The related control

panel settings are shown in Fig. 2: 1 –  $T_{on}-T_{off}$  (discharge pulse modulation), 2 – high-voltage (HV) switch, 3 – current segment setting (IP), 4 – capacitor, 5 – servo control knob (Servo), and 6 – sensitivity control knob (Sens). Preparation of platinum nanocolloid through ESDM is a fast and safe way. This physical method required only DI water and platinum wires and could operate at room temperature [17]. For the preparation of platinum nanocolloid, parameters of the control panel were set appropriately as shown in Table 1. Due to the hardness of platinum, the current segment was set to IP7. While the current segment was set in seven sections, in IP1, the current is the minimum. When an IP section increased, the discharge current between the electrodes was getting higher, the processing speed increased, but the fineness reduced, making the metal surface much rougher. Although it could increase the concentration of colloid, the average size of nanoparticles became larger. When the discharge began, the distance between anode and cathode approached gradually to 30  $\mu\text{m}$ , which is arc discharge [16], as shown in Fig. 3. (a) Prepare for discharge, (b) discharge start, (c) ionisation, (d) arc melt and (e) end of discharge and ready for next discharge. With high temperature up to 8000 K [18], the material was pulled away from the surface and instantly condensed into nanoparticles [19].

In order to make the nanocolloid disperse evenly in the preparation process, a magnet mixer was used. After the discharge, platinum nanocolloid was obtained and waited for the next discharge. The advantage of preparing platinum nanoparticles through ESDM is that no chemical agent is needed, and electric energy was converted into heat for the removal of the metal surface. Fixed discharge cycle was finely etched [20, 21] to obtain a good dispersion, suspension and small size of platinum nanoparticles. This manufacturing method of green energy has become an important issue in nanotechnology [22, 23].

2.2. Preparation of platinum nanocolloid through electrical discharge machine: Discharge pulse modulation ( $T_{on}-T_{off}$ ) was used to control the start and end of discharge, which affected the rate and quality of discharge. HV switching button was used to raise the voltage from 140 to 240 V, suitable for metals of high melting point. Current segment setting (IP) was used to increase the current between electrodes for the use of large-area processing or puncturing. The capacitor was used to increase the pulse modulation of the voltage at  $T_{on}$ , which was used to

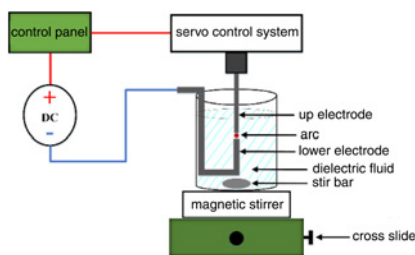


Fig. 1 Diagram of electrical discharge machine



Fig. 2 Control panel of electrical discharge machine

1 –  $T_{on}-T_{off}$  (discharge pulse modulation), 2 – HV switch, 3 – current segment setting (IP), 4 – capacitor, 5 – Servo control knob (Servo), 6 – Sensitivity control knob (Sens)

Table 1 Environmental parameters of platinum nanoparticles preparation

pulse cycle	$T_{on}-T_{off}=30:30$ ( $\mu s$ )
material	platinum, 99.5%
diameter of materials	anode:0.8 mm cathode:1 mm
current segment setting (IP)	7
voltage	140 V
dielectric fluid	DI water
discharge time	1 min

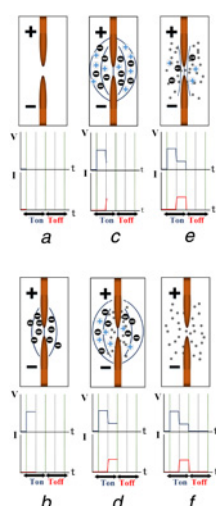


Fig. 3 Diagram of ESDM principle

a Waiting  
b Discharge phenomenon  
c Ionisation  
d Arc effect  
e Discharge cut-off  
f Restore the insulation

increase the conductivity of metals that are more difficult to discharge. Servo control knob (Servo) was used to control the rotation speed of the closed-loop system servo motor, affecting the process of electrode gap control. Sensitivity control knob (Sens) was used to correct the feedback sensitivity of a servo control system, which affected the stability of discharge machining.

In the preparation of platinum nanocolloid, 99.5% of platinum was used as the electrodes. In order to discharge smoothly, a diameter of 0.8 mm platinum wire was used as an anode, while a diameter of 1 mm platinum wire was used as the cathode. The discharge time was 1 min and 100 ml of DI water (conductivity = 1.8  $\mu S/cm$ ) was used as the dielectric fluid. In the parameter setting section,  $T_{on}-T_{off}$  was set to 30:30 ( $\mu s$ ) and the duty cycle of 50% was found to be the best. The accumulation of sediments will lead to a short circuit of electrodes. Therefore, the current segment was set to IP7, which is the optimal setting for preparation of platinum nanocolloid.

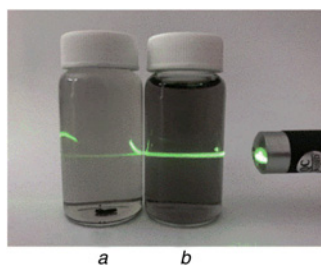
Servo control knob and sensitivity control knob were adjusted to the middle, while discharge quality depends on the gap between electrodes. If the gap is too large, the discharge circuit will open and no discharge arc exists. On the contrary, if the gap is too small, the discharge circuit will short and fail to discharge. The setting of electrode-gap-control is well optimised, so there is no platinum sediment attached to the electrodes. After the discharge, the colour of the dielectric liquid turned to black, which is the colour of platinum nanocolloid.

2.3. Comparison between normal and centrifuged platinum nanocolloids: After precipitation for a few days, the colour of colloid changed from black to transparent. It was preliminarily inferred that the suspension of the nanoparticle is not good enough. In order to analyse this phenomenon, platinum nanocolloid was collected into two scintillation vials for the next preparation. One of the bottles was centrifuged named as a centrifuge, while the other bottle was named normal. The optical properties, suspension stability, particle size distribution, and the crystal lattice structure of two bottles were measured by using various instruments. After the precipitation, two samples were tested to find the differences between before and after precipitation. The optical properties of platinum nanocolloids and centrifuged one were analysed by UV-vis spectrum analyser (9423 UVA1002E Helios Omega) [24, 25]. Zeta potential was measured by using a Zetasizer (NanoZS90, Malvern) to determine suspension of nanocolloid. Normal platinum nanocolloid and centrifuged platinum nanocolloid were dropped on the copper mesh, observing particle size and surface distribution through transmission electron microscope (TEM) (HRTEM, JEM-2100F) [26, 27]. After the preparation and precipitation, large particles under the bottom sediment were dropped onto the slides and dried for the analysis of crystal lattice structure by X-ray diffraction (XRD) (EMPYREAN) [28].

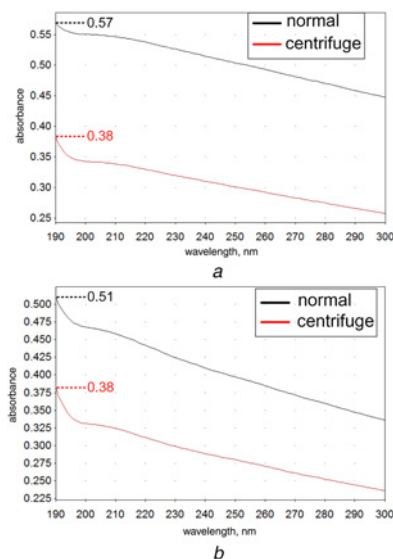
To understand the differences between EDM and other process methods, the results were compared with those of the chemical method [29], which focused on TEM images, dispersion, and size of nanoparticles.

3. Results and discussion: The platinum nanocolloid prepared in this Letter showed the presence of nanoparticles through the Tyndall effect, as shown in Fig. 4. The colour of platinum nanocolloid is black, and the colour of which was precipitated is transparent and colourless.

Preparation of platinum nanocolloid through EDM was characteristic with different particle size and heavier weight. Large particles tend to adsorb small particles, affecting the suspension stability. Therefore, nanocolloid was centrifuged to distinguish between different sizes of nanoparticles. UV-vis absorption spectrum of platinum nanocolloid was shown in Fig. 5, (a) comparison of general and centrifuged, (b) comparison of general and



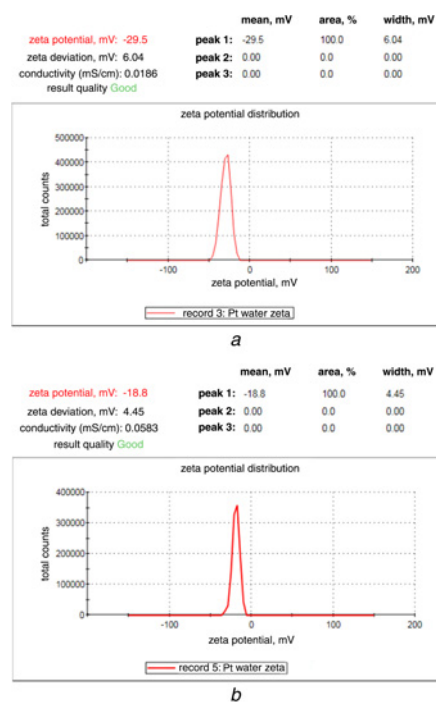
**Fig. 4** Tyndall effect of  
a Platinum nanoparticles after precipitation  
b Platinum nanoparticles



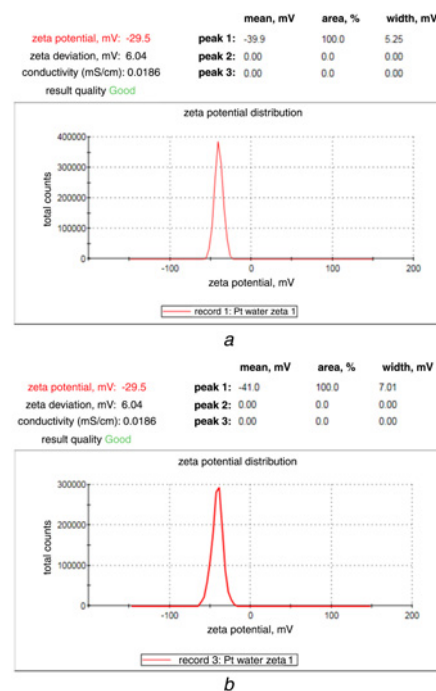
**Fig. 5** UV-vis spectrum of  
a Before centrifuge  
b After centrifuge

centrifuged (after precipitation). Absorbance decreased from 0.57 to 0.38 after colloidal centrifugation, as shown in Fig. 5a. After centrifugation, platinum nanocolloid was precipitated by large particles leaving small particles suspended in the colloid. A number of particles suspended were much less than normal one, indicating the effects of large particles on absorbance. Comparing Figs. 5a and b, it was found that normal absorbance decreased from 0.57 to 0.51. After centrifugation, the absorbance maintained at 0.38 without declination. The normal one is easy to precipitate because large particles will affect the suspension of small particles. Whereas the centrifugal one has filtered out large particles, so there is no precipitation occurred. In accordance with the result, it was presumed that after centrifugation the suspension of small particles is less likely to affect the number of suspended nanoparticles due to precipitation.

The absolute value of Zeta potential  $>30$  mV indicated the good suspension of nanoparticles. Higher concentration of the suspended particles will result in smaller spacing between them. When the space between particles became smaller, the Van der Waals forces increased. Greater Van der Waals force and settlement led to more seriously accumulation and precipitation. Zeta potential of platinum nanocolloids was shown in Fig. 6a as  $-29.5$  mV, in Fig. 6b as  $-18.8$  mV (after precipitation). Zeta potential of centrifuged platinum nanocolloids was shown in Fig. 7a as  $-39.9$  mV, in Fig. 7b as  $-40$  mV (after precipitation). The analysis showed that zeta potential of normal one tends to decrease, indicating the difficulty of suspension. But zeta potential of the centrifuged one does not change, indicating the stable suspension. Which



**Fig. 6** Zeta potential of normal colloid  
a Before precipitation  
b After precipitation

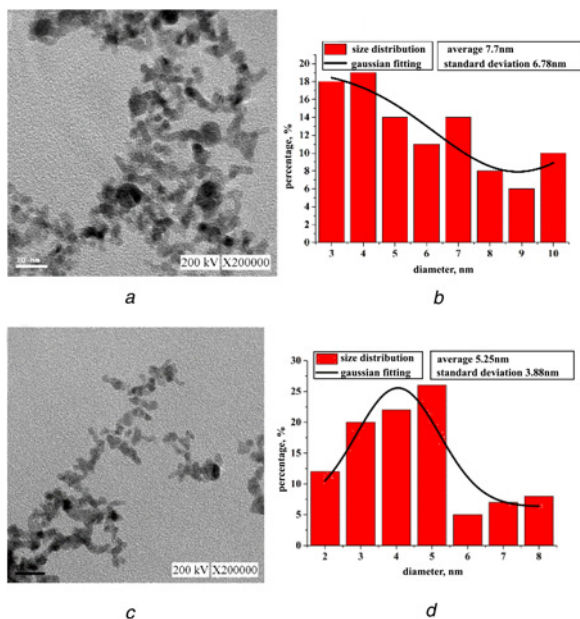


**Fig. 7** Zeta potential of centrifuged colloid  
a Before precipitation  
b After precipitation

proved the large particles of platinum adsorb small particles into a settlement and affect the suspension stability of colloid.

Size distribution of platinum nanoparticles can be clearly seen by TEM. The aggregated nanoparticles have a deep colour, while the dispersed part is much lighter. According to the TEM image and statistics chart, the size distribution of nanoparticles can be compared between general and centrifugal colloids, as shown in Fig. 8a, which shows the TEM of normal platinum nanoparticles

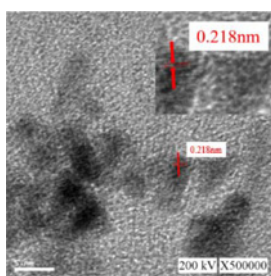




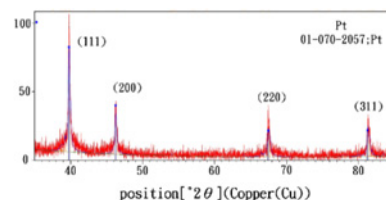
**Fig. 8** TEM of platinum nanoparticles  
*a* Normal platinum nanoparticles  
*b* Size distribution of normal platinum nanoparticles  
*c* Centrifuged platinum nanoparticles  
*d* Size distribution of centrifuged platinum nanoparticles

(10 nm in scale), and Fig. 8*b* shows the size distribution of normal platinum nanoparticles. Most platinum nanoparticles had a diameter of <10 nm, but the colloid is not centrifuged, dispersion of it is poor and there is a lot of nanoparticles aggregated in dark colour. Because van der Waals' force (attractive) is greater than double layer force (repulsive), platinum nanoparticles agglomerate into a larger size, so the size distribution was observed evenly. Fig. 8*c* shows the TEM of centrifuged platinum nanoparticles (10 nm in scale) and Fig. 8*d* shows the size distribution of centrifuged platinum nanoparticles. Size of platinum nanoparticles is also <10 nm. After centrifugation, the dispersion is better and the area of dark part is reduced. Because of less agglomeration, the average size of platinum nanoparticles is much smaller, so the average size was smaller with a diameter of 4 nm. The lattice line distance of platinum nanoparticles was 0.218 nm (5 nm in scale), as shown in Fig. 9.

XRD was used to measure the characteristics of materials, such as structure, phase and crystal orientation, as shown in Fig. 10, which shows the XRD pattern of platinum nanoparticles with four peaks, the peak position ( $2\theta$ ) and Miller indices. The crystal structures are confirmed as platinum (111), (200), (220) and (311), respectively. Four peaks, position ( $2\theta$ ) and Miller indices were measured for the identification of platinum nanoparticles, with the phase sequences (111), (200), (220) and (311), respectively.



**Fig. 9** Crystal lattice structure of platinum nanoparticles



**Fig. 10** XRD of platinum nanoparticles

**Table 2** Differences between colloids in general state and centrifugal state

Measurement	Nano colloids	
	Normal	Centrifugal
UV-vis absorption	0.57	0.38
UV-vis absorption (after precipitation)	0.51	0.38
zeta potential	−29.5 mV	−39.9 mV
zeta potential (after precipitation)	−18.8 mV	−41 mV
size distribution	$7.7 \pm 6.78$ nm	$5.25 \pm 3.88$ nm

**Table 3** Comparison of platinum nanoparticles between ESDM and chemical method in the aspect of TEM image, dispersion and size distribution

Measurement	Method	
	ESDM	Chemical
TEM (size distribution)	inconsistent	consistent
stability	bad (normal) good (centrifugal)	good (functional group)
particle size	$7.7 \pm 6.78$ nm (normal) $5.25 \pm 3.88$ nm (centrifugal)	<4 nm

According to the results of the above experiments, differences between colloids in a normal state and a centrifugal state were compared, as shown in Table 2.

Platinum nanoparticles prepared by ESDM in this Letter were compared with that of chemical preparation, as shown in Table 3. In the section of TEM, the size distribution of nanoparticles through ESDM is inconsistent, while that of chemical is more consistent. The dispersion of nanoparticles through ESDM changes by states. Under normal condition, dispersion of nanoparticle is good; whereas, dispersion of nanoparticle after centrifugation is good. In addition, a stabilising model of platinum nanoparticles through chemical method was established according to the results of HPLC and FT-IR, demonstrating the good dispersion of nanocolloid. Size of platinum nanoparticles prepared through chemical method has a size of <4 nm, while that of ESDM in both normal state and centrifugal state is larger.

**4. Conclusion:** In this Letter, platinum nanocolloid was prepared through platinum by ESDM for the first time. The conclusions were shown as follows:

- Platinum nanoparticles were successfully prepared by ESDM in DI water, and most of the particles had a size of <10 nm in diameter.
- Preparation of platinum nanoparticles by ESDM in DI water is a non-polluting, safe and rapid method. Without the

addition of any chemical agents, it is more likely to apply to the human body in the future.

- (iii) According to the results of UV-vis, average absorbance of normal platinum nanocolloid was 0.57 and dropped to 0.51 after precipitation. The absorbance of centrifuged platinum nanocolloid was 0.38 and there is no change after precipitation. It is demonstrated that the presence of large particles affects the absorbance, whereas the number of nanoparticles was not affected by the precipitation after centrifugation.
- (iv) In normal platinum nanocolloid, large particles adsorbed small particles into agglomeration, so zeta potential decrease from  $-29.5$  to  $-18.8$  mV, with worse suspension. Large particles and small particles in centrifuged platinum nanocolloid were separated, so there is no agglomeration and settlement. Zeta potential of the colloid maintained at  $-40$  mV, which is the evidence of good suspension.
- (v) The difference between normal and centrifuged platinum nanoparticles was measured through TEM. Due to the agglomeration of normal nanoparticles, the average particle size was larger in size distribution of 3–10 nm. Because of the separated particles, there is no agglomeration in centrifuged nanoparticles with smaller diameter accounted for the majority of 4 nm.
- (vi) Results of XRD analysis were used to confirm the crystal lattice structure and phase of platinum nanocolloid prepared by EDM.
- (vii) Properties of platinum nanoparticles prepared by ESDM were compared with that of the chemical method. According to the results of the comparison, size of nanoparticle through chemical method has higher uniformity, and the molecular simulation result shows a good dispersion with the size of  $<4$  nm.

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