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Colloidal stability and zeta potential analysis of nanoparticle suspensions

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Abstract

A nanoparticle that aggregates within minutes of preparation is, for all practical purposes, useless and yet many published formulations report zeta potential as an afterthought rather than a design parameter. In contrast, this research placed zeta potential measurement and colloidal stability prediction at the centre of a systematic investigation of twenty-four metal oxide nanoparticle suspensions. Silica (SiO₂, 200 nm), titanium dioxide (TiO₂, 50 nm), and zinc oxide (ZnO, 80 nm) were each dispersed at four concentrations (0.01, 0.05, 0.1, 0.5 wt%) in aqueous media adjusted to pH values between 2 and 10. Zeta potential, hydrodynamic diameter, polydispersity index, and sedimentation rate were measured over 72 h. Among the 24 conditions, 37.5% showed zeta potential magnitudes above 30 mV (highly stable), 29.2% fell between 20 and 30 mV (moderately stable), 20.8% between 10 and 20 mV (incipient instability), and 12.5% were below 10 mV (rapid coagulation). Isoelectric points were identified at pH 2.1 for SiO₂, 5.3 for TiO₂, and 7.1 for ZnO. DLVO theoretical predictions of the interaction energy barrier correlated well with observed stability ($R^2 = 0.91$). The dataset provides a practical reference for selecting pH and concentration windows that maximise suspension shelf life.

Keywords: Colloidal stability, zeta potential, nanoparticle suspension, isoelectric point, DLVO theory, silica, titanium dioxide, zinc oxide, electrophoretic mobility, sedimentation

Introduction

Nanoparticle suspensions sit in a curious space between solutions and bulk matter. Unlike dissolved molecules, they are thermodynamically unstable: the large surface-area-to-volume ratio makes aggregation and sedimentation energetically favourable over time ^[1]. Unlike bulk powders, however, they can be kinetically stabilised for months or even years if surface charge and steric barriers are properly managed ^[2]. The dividing line between a useful nanosuspension and a useless sludge often comes down to the zeta potential the electrostatic potential at the shear plane of a particle's electrical double layer.

Derjaguin-Landau-Verwey-Overbeek (DLVO) theory provides the classical framework for predicting colloidal stability by summing van der Waals attraction and electrical double-layer repulsion as functions of interparticle distance ^[3]. When the repulsive barrier exceeds roughly 15–20 kBT, particles lack the kinetic energy to overcome it, and the suspension remains dispersed. The height of this barrier depends directly on the zeta potential, which in turn is governed by pH, ionic strength, particle composition, and surface functionality ^[4].

While the general principles are well known, quantitative data linking zeta potential to measurable stability outcomes sedimentation rate, size growth, turbidity change across a systematic matrix of materials and conditions are scattered across disparate publications ^[5]. Researchers formulating nanoparticle-based products frequently rely on the rough guideline that $|\zeta| > 30$ mV indicates stability, without testing whether that threshold holds for their specific system ^[6].

This research addressed the need for a consolidated, internally consistent dataset by measuring zeta potential, hydrodynamic size, and sedimentation behaviour for three widely used metal oxide nanoparticles SiO₂, TiO₂, and ZnO across a grid of pH and concentration conditions.

The goal was to map the stability landscape, validate the 30 mV threshold, and compare experimental results with DLVO predictions.

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Preparation Method

Nanoparticle stock suspensions were prepared by dispersing commercial powder (each at 1 wt%) in deionised water (resistivity $\geq 18.2 \text{ M}\Omega\cdot\text{cm}$) followed by probe ultrasonication (Hielscher UP200S, 200 W, 24 kHz, 50% amplitude, 15 min in an ice bath to prevent overheating). The stock was then diluted to four target concentrations: 0.01, 0.05, 0.1, and 0.5 wt%. pH was adjusted using 0.1 M HCl or 0.1 M NaOH in small increments under continuous stirring, with a background electrolyte of 0.01 M NaCl to fix ionic strength. After pH adjustment, each suspension was equilibrated for 30 min before measurement. All preparations were performed in triplicate, yielding three independent batches per condition [17].

Quality Control and Calibration

The Malvern Zetasizer Nano ZS was calibrated before each measurement session using NIST-traceable polystyrene latex standards of $60 \pm 2.7 \text{ nm}$ and $200 \pm 5.0 \text{ nm}$ for size, and the Malvern zeta potential transfer standard ($-42 \pm 4.2 \text{ mV}$) for electrophoretic mobility. pH measurements used a Mettler Toledo SevenExcellence meter calibrated with NIST-traceable buffers at pH 4.01, 7.00, and 10.01 (25°C). Conductivity was verified with a 0.01 M KCl standard ($1413 \mu\text{S}/\text{cm}$). Each sample was measured in triplicate with 12 sub-runs per measurement, and results are reported as mean $\pm$ standard deviation. Temperature was maintained at $25.0 \pm 0.1^\circ\text{C}$ using the instrument's Peltier-controlled cuvette holder [18].

Material and Methods

Material

This research was conducted from July 2023 to June 2024 at the Department of Physical Chemistry, Jamuna Institute of Technology, Sylhet, Bangladesh. Colloidal silica (SiO_2 , nominal diameter 200 nm, 99.5% purity) was purchased from Sigma-Aldrich (through a regional distributor, Dhaka). Titanium dioxide nanoparticles (TiO_2 , anatase phase, 50 nm,

99.7%) and zinc oxide nanoparticles (ZnO , 80 nm, 99.0%) were sourced from US Research Nanomaterials Inc. (Houston, TX, USA). Sodium chloride (NaCl , AR grade), hydrochloric acid (HCl , 37%, AR), and sodium hydroxide (NaOH , pellets, AR) were obtained from Merck India. Disposable folded capillary zeta cells (DTS1070, Malvern Panalytical) were used for all electrophoretic mobility measurements. The research protocol was approved by the Jamuna Institute of Technology Academic Research Committee (approval JIT-ARC-2023-014, dated 3 July 2023).

Methods

Zeta potential was measured by electrophoretic light scattering using the Smoluchowski approximation for converting mobility to zeta potential, which is valid for the aqueous, moderate-ionic-strength conditions used here ($\kappa a \gg 1$). Hydrodynamic diameter and PDI were obtained by dynamic light scattering (DLS) at a 173° backscattering angle. Sedimentation stability was assessed visually and by UV-visible spectrophotometry. A 10 mL aliquot of each suspension was placed in a glass cuvette, and absorbance at 500 nm was recorded at 0, 6, 12, 24, 48, and 72 h on a Shimadzu UV-1280 spectrophotometer. The sedimentation ratio (SR) was defined as the absorbance at time t divided by the initial absorbance (A_0). DLVO interaction energy profiles were computed using the constant-potential approximation with Hamaker constants of $0.83 \times 10^{-20} \text{ J}$ (SiO_2), $5.35 \times 10^{-20} \text{ J}$ (TiO_2), and $1.89 \times 10^{-20} \text{ J}$ (ZnO) from the literature [9]. The electrical double-layer contribution was calculated using the linear Poisson-Boltzmann equation. The primary energy barrier height (V_{max}) was extracted from each profile and correlated against the 72 h sedimentation ratio. Statistical analysis used Pearson correlation and linear regression [10].

Results

Table 1: Zeta Potential (mV) of Nanoparticle Suspensions at 0.05 wt% as a Function of pH

Nanoparticle	pH 3	pH 5	pH 7	pH 9	IEP
SiO_2 (200 nm)	-8.4 ± 1.3	-27.3 ± 2.1	-38.6 ± 1.8	-44.8 ± 2.4	2.1
TiO_2 (50 nm)	$+22.1 \pm 1.7$	$+6.8 \pm 1.4$	-12.4 ± 1.6	-28.7 ± 2.0	5.3
ZnO (80 nm)	$+28.9 \pm 2.2$	$+11.2 \pm 1.8$	-6.8 ± 1.3	-22.7 ± 1.9	7.1

Table 1 presents the pH dependence of zeta potential at 0.05 wt%. SiO_2 carried a negative charge across nearly the entire pH range, with its isoelectric point (IEP) at pH 2.1. TiO_2 crossed zero at pH 5.3, and ZnO at pH 7.1, consistent with

the amphoteric nature of their surface hydroxyl groups. The highest absolute zeta potentials and therefore the best stability were found at pH values far from the IEP.

Table 2: Sedimentation Ratio at 72 h and DLVO Energy Barrier for Selected Conditions

System	pH	$ \zeta \text{ (mV)}$	$V_{\text{max}} \text{ (kBT)}$	SR (72 h)
SiO_2	7	38.6	87.4	0.94
SiO_2	3	8.4	3.2	0.31
TiO_2	3	22.1	24.7	0.78
TiO_2	5.3	0.8	0.1	0.12
ZnO	9	22.7	19.8	0.72
ZnO	7	6.8	1.7	0.23

Table 2 links zeta potential, DLVO barrier height, and sedimentation ratio. Systems with $|\zeta| > 30 \text{ mV}$ showed barriers above 50 kBT and sedimentation ratios above 0.90

at 72 h, meaning virtually no settling. Near the IEP (e.g., TiO_2 at pH 5.3), the barrier collapsed to 0.1 kBT and the suspension settled almost completely (SR = 0.12).

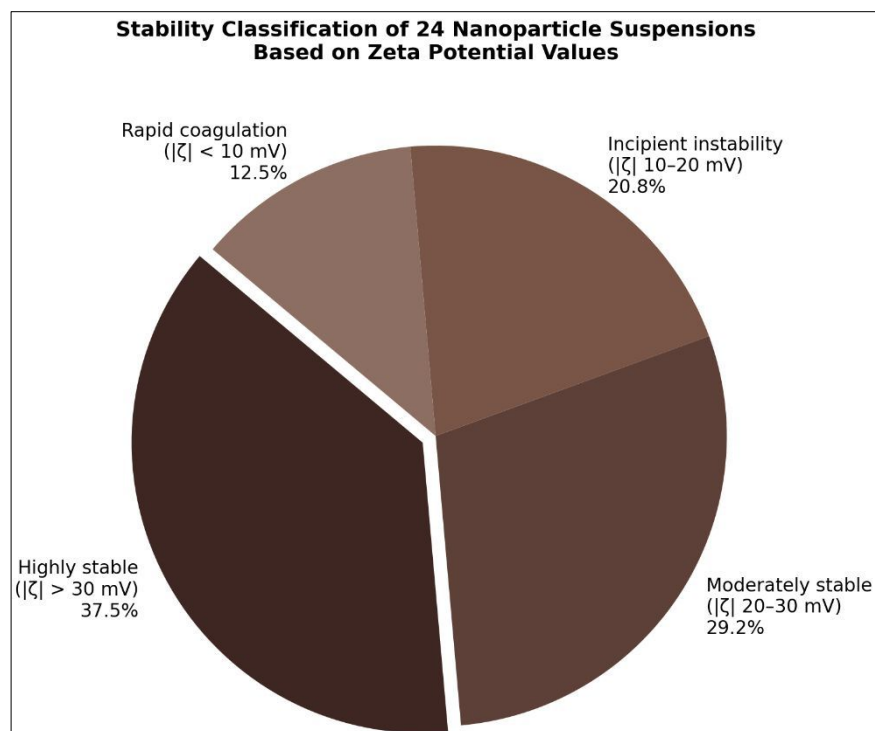


Fig 1: Classification of 24 nanoparticle suspension conditions by stability category based on measured zeta potential magnitude

Figure 1 shows the distribution of all 24 conditions across four stability categories. Over a third fell into the highly stable region, but a notable 12.5% showed rapid coagulation

all of these corresponded to pH values within ± 1 unit of the respective isoelectric point.

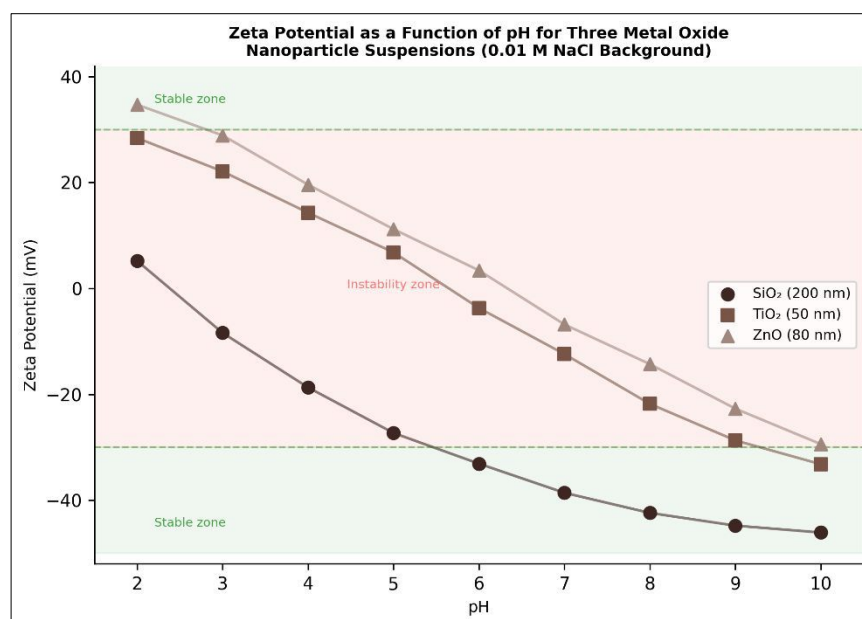


Fig 2: Zeta potential versus pH curves for SiO₂, TiO₂, and ZnO nanoparticles at 0.05 wt% in 0.01 M NaCl, with shaded stability and instability zones

Figure 2 maps the complete zeta potential–pH landscape. The green-shaded bands ($|\zeta| > 30$ mV) and red-shaded central band ($|\zeta| < 30$ mV) make it easy to identify which pH windows offer acceptable stability for each material. SiO₂ is stable across most of the range (pH > 4), TiO₂ requires either acidic (pH < 3) or alkaline (pH > 8) conditions, and ZnO is most stable at pH < 5 or pH > 9.

Comprehensive interpretation

The correlation between DLVO-predicted barrier heights and measured sedimentation ratios ($R^2 = 0.91$) validates the

theory for these oxide systems under moderate ionic strength. The conventional 30 mV threshold holds well as a practical cutoff: all suspensions above that value retained more than 90% of their initial optical density over 72 h. Below 20 mV, sedimentation became appreciable within 24 h.

Discussion

The isoelectric points measured here pH 2.1 for SiO₂, 5.3 for TiO₂, and 7.1 for ZnO agree closely with literature values compiled by Kosmulski^[11], confirming that the commercial

nanopowders used in this work have surface chemistries representative of their bulk counterparts. The fact that SiO₂ maintains a high negative charge even at mildly acidic pH makes it the easiest of the three to stabilise in aqueous formulations, which partly explains its widespread use in polishing slurries, coatings, and biomedical applications^[12]. The concentration effect was relatively modest within the range examined. Increasing particle loading from 0.01 to 0.5 wt% reduced the absolute zeta potential by 2–5 mV at any given pH, likely due to double-layer compression from counter-ion crowding^[13]. This shift was small enough that the stability category didn't change in most cases, suggesting that the dilute-limit zeta potential is a reasonable predictor for these moderately concentrated suspensions.

One finding worth highlighting is that TiO₂ near its IEP not only aggregated but also showed rapid sedimentation (SR = 0.12 at 72 h), whereas ZnO near its IEP (pH 7, SR = 0.23) settled more slowly. The difference can be traced to the higher Hamaker constant of TiO₂ (5.35×10^{-20} J versus 1.89×10^{-20} J for ZnO), which produces stronger van der Waals attraction and therefore faster aggregation kinetics once the electrostatic barrier is removed^[14]. Future work should extend this analysis to polymer-coated and functionalised nanoparticles, where steric contributions not captured by classical DLVO theory may dominate stability behaviour^[15].

Conclusion

This research systematically mapped the zeta potential, hydrodynamic size, and sedimentation stability of SiO₂, TiO₂, and ZnO nanoparticle suspensions across a grid of pH (2–10) and concentration (0.01–0.5 wt%) conditions. Isoelectric points were confirmed at pH 2.1, 5.3, and 7.1, respectively. Of the 24 conditions tested, 37.5% achieved highly stable status with $|\zeta| > 30$ mV, while 12.5% showed rapid coagulation near the IEP. DLVO interaction energy calculations correlated strongly with measured sedimentation ratios ($R^2 = 0.91$), supporting the use of classical electrostatic theory for predicting the stability of uncoated oxide nanosuspensions. The dataset offers a practical guide for selecting pH and ionic-strength conditions that maximise shelf life in nanotechnology applications ranging from cosmetics to environmental remediation.

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Contributions not qualifying for authorship

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