

Peer-Review

Kumaraswamy, Tanesha, Sachith M Veerapur, Ojaswin Sastry, Karthik Nagendra, Swetha Nataraj, and Ramya P Bantwal. 2025. "Engineering Cerium Oxide Nanoparticles for Tunable Wettability Properties." *Journal of High School Science* 9 (3): 454–74. <https://doi.org/10.64336/001c.144173>.

This manuscript will require re-experimentation. Its conclusions are not justified by the methods/data.

1. centrifugation will not remove surfactant even if above the CMC unless ultracentrifuged at g's of 100000. Washing with water and/or water/alcohol mixtures will set up an equilibrium with the rinsing solvent such that an inordinately large number of multiple rinses are required to completely remove unbound surfactant. The only way to completely remove unbound surfactant is dialysis (see references below, in particular reference https://uknowledge.uky.edu/cgi/viewcontent.cgi?article=1073&context=cme_facpub). Provide evidence that there was no unbound surfactant CTAB or citric acid when these prepared Ce-nanoparticle suspensions were analyzed (including analysis for wetting angle or reduction of surface tension).

2. Report centrifugation as the number of times the force of gravity is exceeded on the sample for a certain time so that it is extrapolable to any model/make of centrifuge at any RPM.

3. Report molarity of all ingredients in the final preparation mixture so that the mole ratios of relevant species can be calculated; i.e. moles of citric acid of CTAB per cm² on nanoparticle. This enables the calculation of whether the coverage was complete as a monolayer or whether the surfactant /citric acid existed as > monolayer on each nanoparticle. Calculate and report these numbers as they will add to the depth and utility of the manuscript. See references below.

4. Provide evidence that you have not changed the morphology, surface characteristics (including zeta potential), binding of surfactant or citric acid of the nanoparticles by "dispersing them in ethanol, drop-cast onto silicon substrates, and dried under vacuum overnight..." Note that both CTAB and citric acid are soluble in ethanol. How was the silicon substrate prepared ? (see reference below).

5. Provide evidence that a physical mixture of citric acid and/or CTAB with the nanoparticles (without the hydrothermal reaction step) does not result in the same analytical results for any/all of the tests performed (including the contact angle measurements). You would still obtain the same results with XRD, particle size, FTIR and Raman spectroscopy with physical mixtures. Therefore, you will need to provide evidence that there is a difference in adsorption energy between citric acid and/or CTAB with the nanoparticle when mixed physically versus when synthesized with the hydrothermal reaction step.

6. You did not perform DSC, TGA or ¹³C-SSNMR on the sample. These techniques are the only ones that would have definitively informed about the adsorption of citric acid/CTAB on the nanoparticles (see references below, especially https://uknowledge.uky.edu/cgi/viewcontent.cgi?article=1073&context=cme_facpub), therefore your methods do not allow differentiation of adsorbed versus non-adsorbed citric acid/CTAB. Please provide such evidence. Also see points 1 and 5.

References:

<http://dx.doi.org/10.1016/j.ceramint.2015.05.044>

<https://doi.org/10.1080/07388551.2018.1426555>

https://uknowledge.uky.edu/cgi/viewcontent.cgi?article=1073&context=cme_facpub

[https://citeseerx.ist.psu.edu/document?](https://citeseerx.ist.psu.edu/document?repid=rep1&type=pdf&doi=e82780bb0c0bb91193837a32e77ec1201b8b6f12)

[repid=rep1&type=pdf&doi=e82780bb0c0bb91193837a32e77ec1201b8b6f12](https://citeseerx.ist.psu.edu/document?repid=rep1&type=pdf&doi=e82780bb0c0bb91193837a32e77ec1201b8b6f12)

We thank the reviewer for the insightful questions. We have addressed this question, and point by point response is given below.

1. Centrifugation will not remove surfactant even if above the CMC unless ultracentrifuged at g's of 100000. Washing with water and/or water/alcohol mixtures will set up an equilibrium

with the rinsing solvent such that an inordinately large number of multiple rinses are required to completely remove unbound surfactant.

The only way to completely remove unbound surfactant is dialysis (see references below, in particular reference https://uknowledge.uky.edu/cgi/viewcontent.cgi?article=1073&context=cme_facpub). Provide evidence that there was no unbound surfactant CTAB or citric acid when these prepared Ce-nanoparticle suspensions were analyzed (including analysis for wetting angle or reduction of surface tension).

We agree with the reviewer that ultracentrifugation is ideal, repeated washing with water/ethanol (1:1 v/v) disrupts micelle formation (above CMC) and facilitates surfactant removal due to solubility differences (Journal of Molecular Liquids 187 (2013) 320–325). However, we carried out zeta potential studies and presented them below.

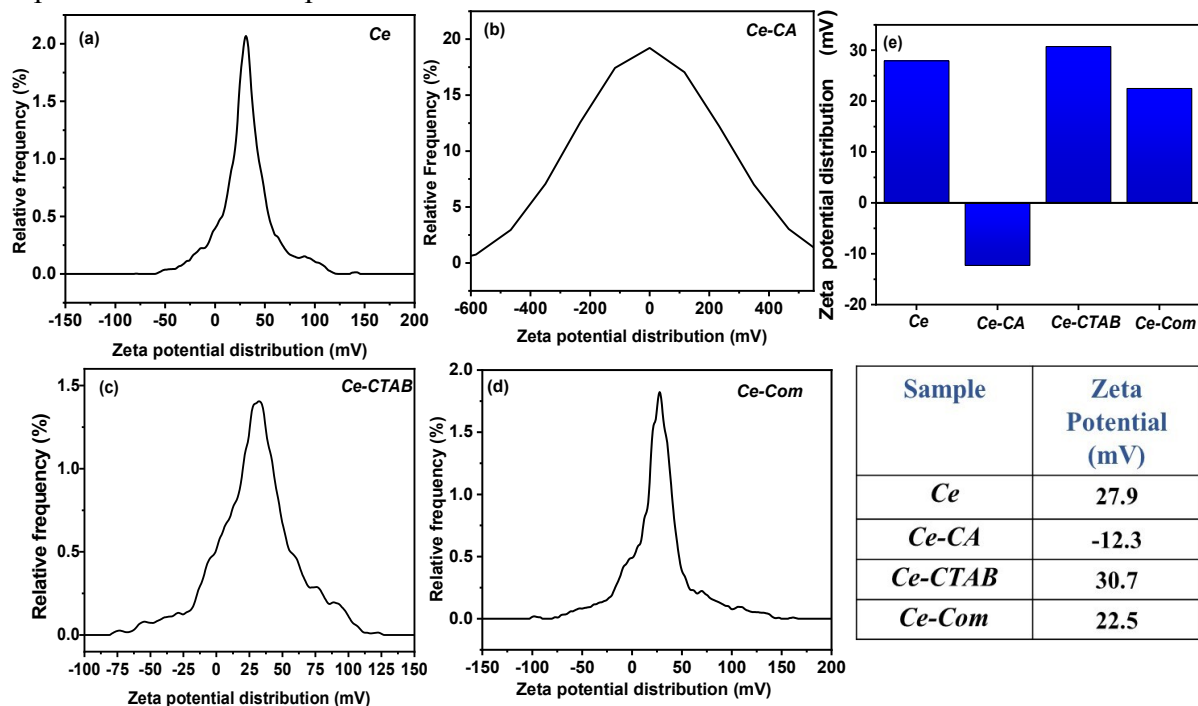


Figure 10. Zeta potential analysis of (a) *Ce*, (b) *Ce-CA*, (c) *Ce-CTAB*, (d) *Ce-Com*, (e) Bar diagram showing the zeta potential distribution.

CTAB-coated nanoparticles showed a positive zeta potential (observed: +30.7 mV), while free CTAB in solution would have caused an even higher value ($>+40$ mV) due to excess cations. Citric acid-coated nanoparticles exhibit a negative zeta potential (-12.3 mV), but free citric acid would further decrease it (<-25 mV). The stable, moderate zeta values suggest bound surfactants only, not free excess.

We have added the following paragraph to the revised manuscript.

Zeta potential analysis was performed to evaluate the characteristics of the surface charge of the nanoparticles, and the results are shown in Figure 10. For the measurements, an aqueous dispersion of each nanoparticle sample was prepared in type 1 water at a concentration of 1 mg/mL. The experiments were conducted under ambient conditions to determine the nature of nanoparticle surface charges. (1)

The zeta potential of *Ce* nanoparticles was found to be +27.9 mV, indicating a positively charged surface at neutral pH. In contrast, the *Ce-CA* sample exhibited a zeta potential of -12.3 mV, while *Ce-CTAB* showed a higher positive value of +30.7 mV, suggesting strong surface charge contributions from the stabilizing agents. The negative zeta potential of *Ce-CA* is attributed to the anionic nature of citric acid, whereas the positive charge of *Ce-CTAB* arises from the cationic surfactant CTAB (2). The

commercial Ce sample (*Ce-Com*) displayed a zeta potential of +22.5 mV, further supporting the influence of surface modifications on colloidal stability.

2. **Report centrifugation as the number of times the force of gravity is exceeded on the sample for a certain time so that it is extrapolable to any model/make of centrifuge at any RPM. The relative centrifugal force (RCF) accurately reflects the amount of force applied on the samples, irrespective of the model/ make of the instrument, whereas RPM suggests only about the speed of rotation which is subject to variation across different model/ make of the instrument.**

We thank the reviewer for asking this query in the revised manuscript; we have added the RCF value. The RCF value is 9558g for the instrument used; the centrifugation was carried out at 9000 rpm for 10 minutes. We have attached the image from the instrument below.



3. **Report molarity of all ingredients in the final preparation mixture so that the mole ratios of relevant species can be calculated; i.e. moles of citric acid of CTAB per cm² on nanoparticle. This enables the calculation of whether the coverage was complete as a monolayer or whether the surfactant /citric acid existed as > monolayer on each nanoparticle. Calculate and report these numbers as they will add to the depth and utility of the manuscript. See references below.**

The concentration of CeO₂ is 10mg/ mL. The molarity of CTAB is 10.37nM/ nm² and citric acid is 8.92 nM/nm² as calculated by considering the shape of NPs as rectangle (from TEM image) and a distribution of 30 particles.

The coverage of nanoparticles for Ce-CTAB is 1.85 nM/ nm² and for Ce-CA the coverage is 2.846 nM/nm², which suggests that the coverage is not a monolayer; it is a multilayer coverage.

4. **Provide evidence that you have not changed the morphology, surface characteristics (including zeta potential), binding of surfactant or citric acid of the nanoparticles by “dispersing them in ethanol, drop-cast onto silicon substrates, and dried under vacuum overnight...” Note that both CTAB and citric acid are soluble in ethanol. How was the silicon substrate prepared? (see reference below).**

We have used a bath sonicator to disperse the samples; the duration of sonication was 2 minutes. A shorter duration of exposure to sound waves will not impact the morphology or size of nanoparticles. The change in morphology will be observed only after sonicating for a very long time, i.e, more than 30 mins.

Although citric acid and CTAB are soluble in ethanol, the molecules are not freely available; they are bound to the nanoparticles, as suggested by the FTIR and zeta potential results. Cerium oxide was dispersed in ethanol and sonicated for 2 min at room temperature; it was immediately drop-cast on the silicon substrate and dried under vacuum. The same protocol was followed for all the samples.

- Provide evidence that a physical mixture of citric acid and/or CTAB with the nanoparticles (without the hydrothermal reaction step) does not result in the same analytical results for any/all of the tests performed (including the contact angle measurements). You would still obtain the same results with XRD, particle size, FTIR and Raman spectroscopy with physical mixtures. Therefore, you will need to provide evidence that there is a difference in adsorption energy between citric acid and/or CTAB with the nanoparticle when mixed physically versus when synthesized with the hydrothermal reaction step.

The physical mixtures of the compounds when mixed with water/ ethanol dissolves the surfactants but not the nanoparticles, this will create an uneven sol recrystalizes upon heating, hence the studies like contact angle cannot be performed when it is a physical mixture, it has to be dispersed and drop casted to conduct this study. The contact angle measurements were done at 5 sample positions, and the average was considered as final; hence, it proves that the dispersion was even throughout the coating area.

- You did not perform DSC, TGA or ^{13}C -SSNMR on the sample. These techniques are the only ones that would have definitively informed about the adsorption of citric acid/CTAB on the nanoparticles (see references below, especially https://uknowledge.uky.edu/cgi/viewcontent.cgi?article=1073&context=cme_facpub), therefore your methods do not allow differentiation of adsorbed versus non-adsorbed citric acid/CTAB. Please provide such evidence. Also see points 1 and 5.

We thank the reviewer for asking this query, and we have done TGA analysis for all four samples. All the samples were analyzed for their thermal properties using thermogravimetric analysis (TGA) and are shown below.

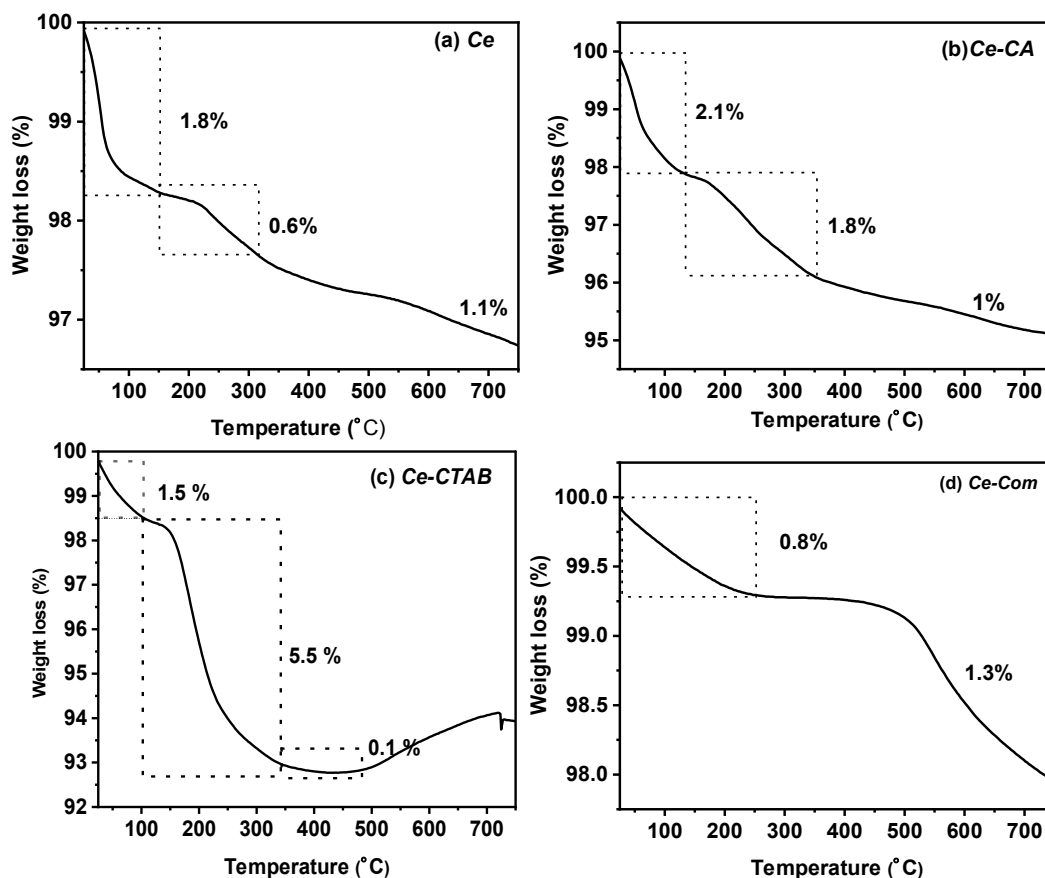
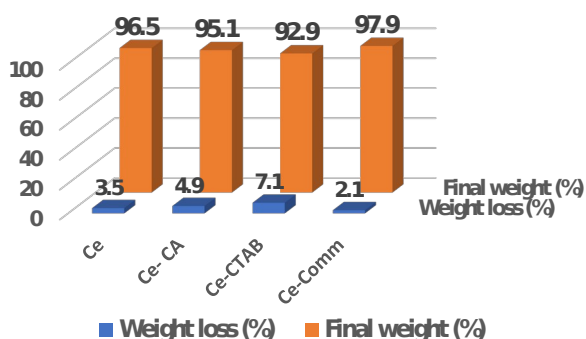


Figure 11. Thermogravimetric analysis of (a) *Ce*, (b) *Ce-CA*, (c) *Ce-CTAB*, (d) *Ce-Com*.

TGA was carried out in an ambient atmosphere, and the samples were subjected to heating from room temperature up to 750°C with an increase of temperature at the rate of 10°C per minute. The weight loss was observed in three stages: the loss of weight from 100°C to 150°C is attributed to the loss of surface water molecules, (3) and the loss between 150°C and 600°C is due to organic compounds. (4) The thermal stability of cerium oxide is very high, and the melting point is around 2500°C. (5) The initial weight loss in the samples was observed as 1.8%, 2.1%, 1.5%, and 0.8% for *Ce*, *Ce-CA*, *Ce-CTAB*, and *Ce-Comm*, respectively, at temperatures between 100-200°C, attributed to the loss of surface water molecules. The *Ce* sample shows a loss of 0.6% between 250°C and 400°C due to the loss of organic compounds and a loss of 1.1% between 500°C and 700°C, which is also attributed to the loss of organic matter (6). The *Ce-CA* shows a loss of 1.8% at 200°C to 400°C is due to the loss of organic compounds, i.e., citric acid, and a 1% loss at 550°C to 650°C due to decomposition of organic species. For the *Ce-CTAB* sample, there is a sharp curve between 150°C and 350°C, with 5.5% loss due to the loss of organic compounds, i.e., CTAB. The wide curve at 350°C and 550°C with a loss of 0.1% could be due to the decomposition of remaining organic matter. *Ce-Com* shows a loss of 1.3% at 500°C could be due to the organic materials in the sample.



Sl. No	Sample	Weight loss (%)	Final weight (%)
1	<i>Ce</i>	3.5	96.5
2	<i>Ce-CA</i>	4.9	95.1
3	<i>Ce-CTAB</i>	7.1	92.9
4	<i>Ce-Com</i>	2.1	97.9

Figure 12. Bar diagram representing TGA analysis of various samples.

The TGA results clearly says that the amount of surfactant bound to the nanoparticles is highly influencing the surface properties of the material, the graph trend says that the maximum loss has occurred in the sample *Ce-CTAB* which suggests that the functional groups were highly bound to the particles, and comparing this with the contact angle studies this sample has shown the most hydrophobic behavior compared to the other samples. The *Ce-CA* results also say that the functional groups are bound to the nanoparticles and have contributed to the surface activity in contact angle measurements. The loss of weight in *Ce-CA* and *Ce-CTAB* clearly suggests the loss of bound surfactants, which are absent in the *Ce* and *Ce-Com* samples.

References

1. Singh RP, Sharma K, Mausam K. Dispersion and stability of metal oxide nanoparticles in aqueous suspension: A review. *Mater Today Proc.* 2019;26(xxxx):2021–5.
2. Habib IY, Kumara NTRN, Lim CM, Mahadi AH. Dynamic light scattering and zeta potential studies of ceria nanoparticles. *Solid State Phenom.* 2018;278 SSP:112–20.
3. Hancock ML, Yokel RA, Beck MJ, Calahan JL, Jarrells TW, Munson EJ, et al. The characterization of purified citrate-coated cerium oxide nanoparticles prepared via hydrothermal

- synthesis. Appl Surf Sci. 2021;535.
4. Kommula B, Prabhu B R, Kopperi H, Bhat VS, Hegde G, John NS. Diverse Morphologies of Nb₂O₅ Nanomaterials: A Comparative Study for the Growth Optimization of Elongated Spiky Nb₂O₅ and Carbon Nanosphere Composite. Part Part Syst Charact. 2024;41(3):1–10.
 5. Aristova NM. Thermodynamic Properties of Cerium Dioxide in the Condensed State. High Temp. 2022;60(6):756–60.
 6. Chaudhary S, Sharma P, Kumar R, Mehta SK. Nanoscale surface designing of Cerium oxide nanoparticles for controlling growth, stability, optical and thermal properties. Ceram Int . 2015;41(9):10995–1003.

I thank the authors for attempting to address my comments and appreciate the work put in. However, I am not convinced.

1. In my comments, I stated that the only way to remove unbound surfactant is dialysis. This does not appear to have been done. It is relatively simple to obtain a dialysis cassette with a defined MWCO, load it with your nanoparticle suspension, and spin in a sink medium such as water in a large beaker. You have provided zeta potential measurements as an indicator of chemisorption. Zeta potential will not confirm chemisorption; it is physical adsorption to the surface of the nanoparticle only because of charge difference.

2. Thank you for performing the TGA. However, your TGA shows approx. 2% organic material adsorbed. In contrast, the paper I cited earlier: https://uknowledge.uky.edu/cgi/viewcontent.cgi?article=1073&context=cme_facpub shows a 15% weight loss. These authors calculated that their particle (similar size as your, about 7 nm) were covered with a nanometer of chemisorbant (see eq. 5 in their manuscript). It follows that if a 15% weight loss is required for a monolayer, then your weight loss of 2% effectively means that negligible surfactant is chemisorbed to your particles.

I will therefore need to see confirmatory evidence that you have actually succeeded in chemisorbing various molecules to your Cerium Oxide nanoparticles.

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Ans: As suggested by the reviewer, we have carried out dialysis of the CeO₂ nanoparticles. The dispersion of CeO₂ nanoparticles is filled in regenerated cellulose membranes dialysis tubings with a circular internal diameter of 22 mm, and a MWCO of 10,000 Da (Thermo Fisher Scientific Catalog number 68100). The dialysis is carried out in type 1 water for 72 hours under 300 rpm stirring at room temperature. We have replaced dialysate every 24 hours. The setup of dialysis is shown in the figure below.

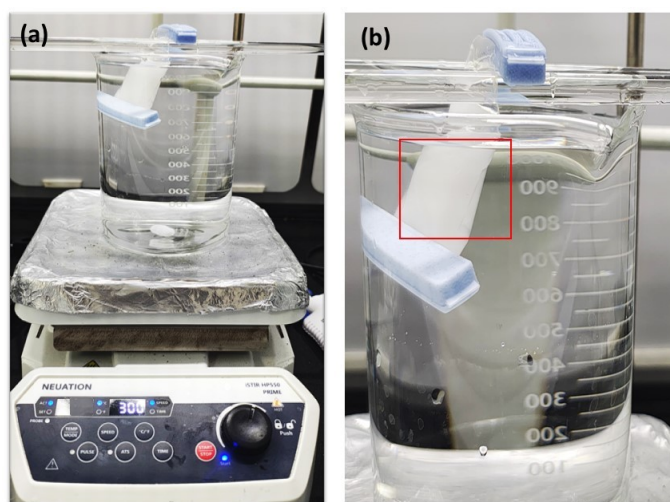


Figure 1. (a) Photograph of dialysis set up for CeO₂ nanoparticles. (b) Zoomed photograph of the dialysis set up.

Further, we have carried out TGA to see the organic matter content on the nanoparticles and the results are shown below.

Thank you for performing the TGA. However, your TGA shows approx. 2% organic material adsorbed. In contrast, the paper I cited earlier: https://uknowledge.uky.edu/cgi/viewcontent.cgi?article=1073&context=cme_facpub shows a 15% weight loss. These authors calculated that their particle (similar size as your, about 7 nm) were covered with a nanometer of chemisorbant (see eq. 5 in their manuscript). It follows that if a 15% weight loss is required for a monolayer, then your weight loss of 2% effectively means that negligible surfactant is chemisorbed to your particles. I will therefore need to see confirmatory evidence that you have actually succeeded in chemisorbing various molecules to your Cerium Oxide nanoparticles.

Ans: We thank the reviewer for their insightful comment. As suggested, we have performed thermogravimetric analysis on freshly prepared samples of *Ce-CA* and *Ce-CTAB* after dialysis purification. The TGA data provided below are included in the revised manuscript.

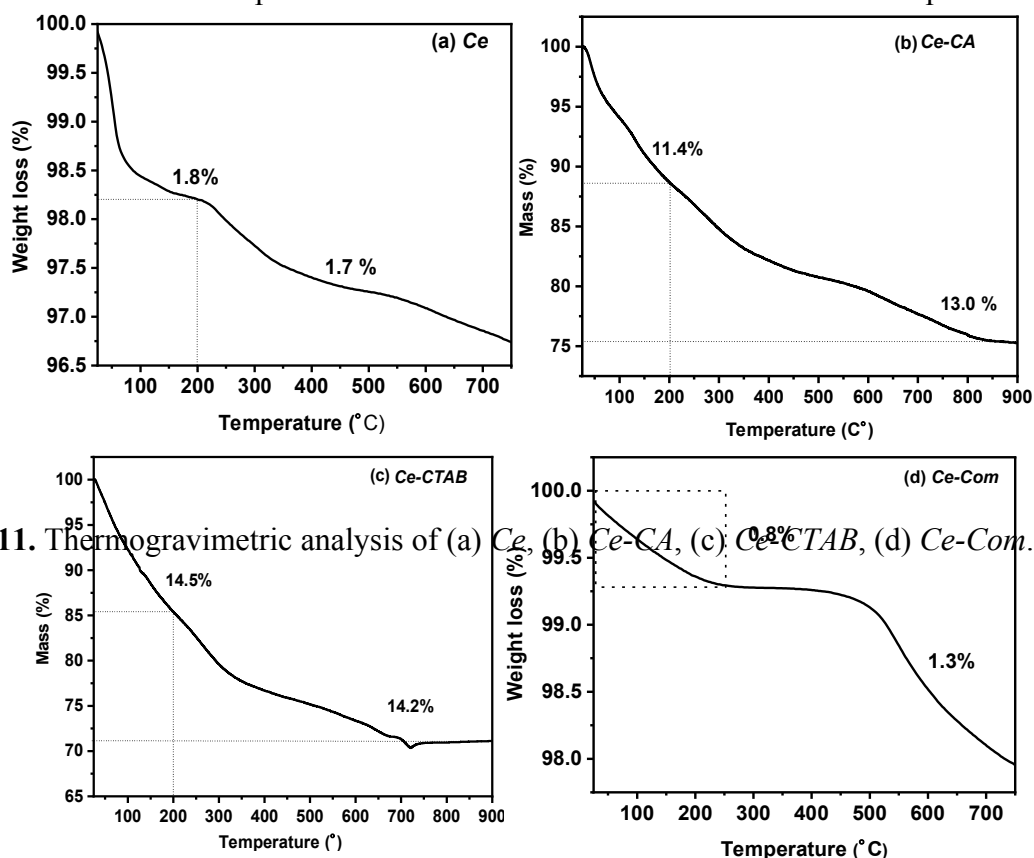


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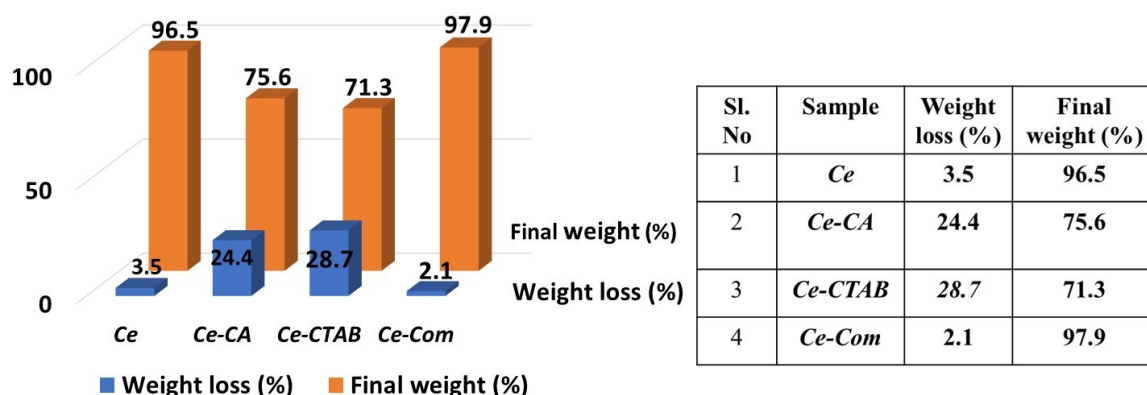


Figure 12. Bar diagram representing TGA analysis of various samples.

TGA was carried out in an ambient atmosphere, and the samples were subjected to heating from room temperature up to 900°C with an increase of temperature at the rate of 10°C per minute. The loss of weight from 100°C to 200°C is attributed to the loss of surface water molecules (31), and the loss between 200°C to 600°C is due to organic compounds. (32) The thermal stability of cerium oxide is very high, and the melting point is around 2500°C. (33)

The initial weight loss in the samples was observed as 1.8%, 11.4%, 14.5%, and 0.8% for *Ce*, *Ce-CA*, *Ce-CTAB*, and *Ce-Com*, respectively, at temperatures between 100 -200°C, attributed to the loss of surface water molecules.

The *Ce* sample shows a loss of 1.7% between 200°C and 700°C due to the loss of organic compounds. (34) The *Ce-CA* shows a loss of 13.0% at 200°C to 750°C is due to the loss of organic compounds, i.e., citric acid. For the *Ce-CTAB* sample, there is a 14.2% loss due to the loss of organic compounds, i.e., CTAB. The *Ce-Com* sample shows a loss of 1.3% at 500°C could be due to the organic materials present in the sample.

Further, the final weight is 96.5%, 75.6% and 71.3%, and 97.9% for *Ce*, *Ce-CA*, *Ce-CTAB*, and *Ce-Com*, respectively. By carrying out TGA, we can see that there is a significant amount of chemisorbed surfactant molecules on the *Ce-CA* and *Ce-CTAB* samples.

Thank you for addressing my comments and performing the dialysis experiment. It does seem like you do have some degree of chemisorption of the ligands on the nanoparticles. With this evidence, some more explanation/corrections is/are necessary in the manuscript along the lines below

1. Why does the contact angle increase when the nanoparticles are functionalized with citric acid? Your explanation with reference 28 does not seem to answer the question because there is no reference to citric acid or contact angles in that reference (please make sure the references you cite actually back up your evidence; this is automatically checked during proofreading). In the manuscript that I sent to you earlier, the authors diagram the possible configuration of CA on the surface. If the -COOH and -OH functional groups are projected into water, the loss in bulk water's entropy through micellization would be more than offset by the negative enthalpic contributions between the C=O hydrogen acceptors and O-H hydrogen donors with water, making the free energy change negative and thus decreasing the contact angle. Please provide some explanation for your anomalous result in the manuscript.

2. Do NOT hyperlink the references in the text to those in the reference section of the manuscript.

3. The references should follow the Journal's format. All references should follow a consistent format (APA or equivalent). A live link (DOI) is necessary for each reference. When there are < 6 authors, all authors must be listed, when there are > 6 authors, the first 6 must be listed followed by an et al. Do

NOT use the software's automatic numbering feature to number references. Instead, number them manually.

4. The quality of English is generally poor. See for example: ".....The TGA results clearly says that the amount of surfactant bound to the nanoparticles is highly influencing the surface properties of the material, the graph trend says that the maximum loss has occurred in the sample Ce- CTAB which suggests that the functional groups were highly bound to the particles, and comparing this with the contact angle studies this sample has shown the most hydrophobic behavior compared to the other samples....."

Please run the manuscript through grammarly or equivalent to improve grammar, sentence structure and syntax. The manuscript must be written in past perfect tense, third person. The use of opinionated superlative words must be avoided.

5. More details in procedure - all around - seem necessary. For example, in the dialysis experiment, how much suspension was aliquoted into the membrane? How much sink water? After dialysis, how was the sample dried (temp., time, oven?)... You mention that an MWCO of 10kDa was used. This corresponds to a diameter of approximately 2-5 nm. Explain why some of your nanoparticles may not have filtered through this membrane into the water sink and if yes, describe the implications. Please make sure the procedure described for the various experiments in the manuscript contains enough detail in order for it to be reproduced in other labs.

6. For all experiment, where applicable, describe how many replicates were performed.

Thank you for addressing my comments and performing the dialysis experiment. It does seem like you do have some degree of chemisorption of the ligands on the nanoparticles. With this evidence, some more explanation/corrections is/are necessary in the manuscript along the lines below

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"We thank the reviewer for their insightful comment. The observed increase in contact angle after citric acid (CA) functionalization is attributed to the orientation of CA molecules on the CeO₂ surface. Here, the carboxylate (-COO⁻) groups of CA chelate strongly with Ce³⁺/Ce⁴⁺ sites on the nanoparticle surface, causing the hydrophobic hydrocarbon chains (-CH₂) to face outward toward the water interface. This molecular arrangement is supported by FTIR data, which confirms the presence of -CH₂ groups on the functionalized surface. While carboxylates are inherently hydrophilic, their binding geometry dictates wettability. In our system, the chelation-induced folding of -COO⁻ groups toward CeO₂ (rather than the water phase) and the exposure of -CH₂ groups dominate the interfacial energetics, leading to increased hydrophobicity. Although CA also contains hydroxyl (-OH) groups, their contribution to wettability is outweighed by the shielding effect of the hydrophobic -CH₂ moieties. Thus, the net effect is a higher contact angle, consistent with the observed behaviour."

The observed increase in contact angle upon citric acid (CA) functionalization aligns with prior studies on Ce-based films, where citrate-modified surfaces exhibit higher hydrophobicity compared to chloride-derived CeO₂. For instance, Dunja Marunčić et al. demonstrated that Ce-citrate films on AISI 4130 steel yielded water contact angles (WCAs) of 50–65° (vs. 35–40° for Ce-chloride), with prolonged immersion further increasing WCAs to ~71.7° after 96 h. This trend was attributed to the formation of a denser, more compact inhibitory film in Ce-citrate systems, likely due to the chelating action of citrate.

Marunčić, D., Jegdić, B., Pejić, J., Milošević, M., Marinković, A., & Radojković, B. (2022). *Analysis of inhibitory properties of Ce-citrate as a green corrosion inhibitor of low alloy steel in neutral chloride solution. Materials and Corrosion*, 73(8), 1286-1297. DOI: 10.1002/maco.202213079.

In the revised manuscript, we have removed Ref 28. In the revised manuscript, we have added this paragraph and cited the above manuscript.

2. **Do NOT hyperlink the references in the text to those in the reference section of the manuscript.**

We have removed the hyperlink in the revised manuscript.

3. **The references should follow the Journal's format. All references should follow a consistent format (APA or equivalent). A live link (DOI) is necessary for each reference. When there are < 6 authors, all authors must be listed; when there are > 6 authors, the first 6 must be listed, followed by an et al. Do NOT use the software's automatic numbering feature to number references. Instead, number them manually.**

We thank the reviewer for the query. In the revised manuscript, we have used the APA style of reference, and the DOI is also included.

4. **The quality of English is generally poor. See for example: ".....The TGA results clearly says that the amount of surfactant bound to the nanoparticles is highly influencing the surface properties of the material, the graph trend says that the maximum loss has occurred in the sample Ce- CTAB which suggests that the functional groups were highly bound to the particles, and comparing this with the contact angle studies this sample has shown the most hydrophobic behavior compared to the other samples....."**

Please run the manuscript through Grammarly or equivalent to improve grammar, sentence structure and syntax. The manuscript must be written in past perfect tense, third person. The use of opinionated superlative words must be avoided.

We have run through the Grammarly software (paid version) and corrected the grammar.

5. **More details in procedure - all around -seem necessary. For example, in the dialysis experiment, how much suspension was aliquoted into the membrane? How much sink water? After dialysis, how was the sample dried (temp., time, oven? You mention that an MWCO of 10kDa was used. This corresponds to a diameter of approximately 2-5 nm. Explain why some of your nanoparticles may not have filtered through this membrane into the water sink, and if yes, describe the implications. Please make sure the procedure described for the various experiments in the manuscript contains enough detail in order for it to be reproduced in other labs.**

A 1 mg/mL dispersion of CeO₂ nanoparticles was prepared in type 1 water. For purification, 20 mL of the dispersion was loaded into regenerated cellulose membranes dialysis tubings with a circular internal diameter of 22 mm, and a MWCO of 10,000 Da (Thermo Fisher Scientific Catalog number 68100). The dialysis is carried out in one litre (sink) of type 1 water for 72 hours under 300 rpm stirring at room temperature. We have replaced dialysate every 24 hours. The dialysate volume was 50x the CeO₂ dispersion volume. After the dialysis, the CeO₂ nanoparticles are centrifuged at 9558g for 15 minutes. Then the CeO₂ powder is dried in a hot air oven at 80°C for 4 hours.

Our TEM data shows that the nanoparticles exist as agglomerates rather than single particles. Moreover, it confirms that they exist as dimers, trimers, or chains, and these agglomerates exceed the membrane pore size, leading to retention of the nanoparticles in the membrane. As per the suggestion of the reviewer, we have enriched the experimental part with additional information.

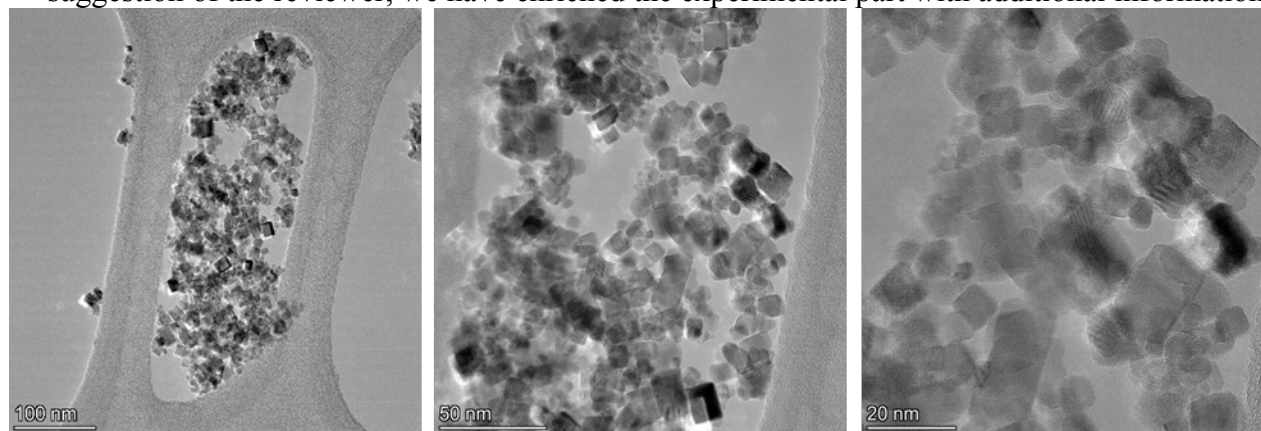


Figure 1. TEM image of the *Ce-CA* with various magnifications.

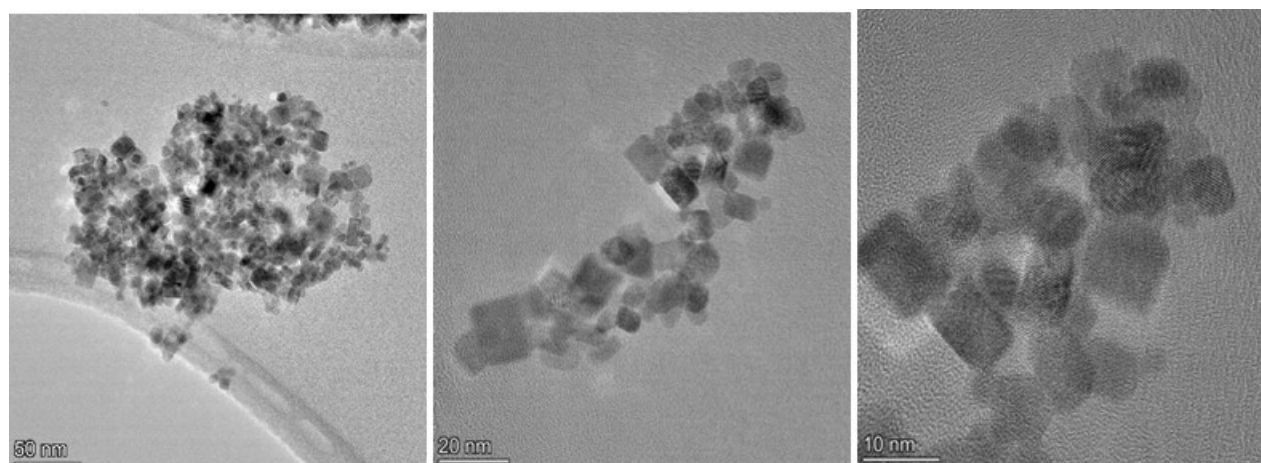


Figure 2. TEM image of the *Ce-CTAB* with various magnifications.

We have provided additional TEM images for the reviewers' reference. The TEM data indeed show that the nanoparticles exist as agglomerates rather than single particles. Moreover, it confirms that they exist as dimers, trimers, or chains, and these agglomerates exceed the membrane pore size, leading to retention of the nanoparticles in the membrane.

6. **For all experiments, where applicable, describe how many replicates were performed.**

All experiments are repeated 5 times to obtain accurate and consistent data.

Please make the formatting and grammatical changes as described. Please communicate the entire procedure in enough detail so that your experiments can be reproduced.

1. References need to appear sequentially in the manuscript. The reference section should be numbered manually and the references should correspond with the numbers in the text.

2. Corrected tense, grammar and sentence structure using your paragraph. The corrected paragraph appears below yours. As communicated to you earlier, please use past perfect tense - not present continuous.

“We have synthesized CeO₂ nanoparticles employing a simple, one-pot hydrothermal approach using cerium nitrate and sodium hydroxide as precursors, along with various capping agents in water. We have used citric acid (CA) and cetrimonium bromide (CTAB) as capping agents. The capping agent’s concentration is 0.025 M. We have taken 0.1 M Cerium nitrate solution and 1M sodium hydroxide solution. The reaction time of 18 h and the temperature of 150 °C were maintained for all the reactions. The powder obtained from the reaction mixture was washed with an ethanol-water (50:50) mixture. The products are centrifuged at a relative centrifugal force (RCF) value of 9558g and dried in the hot air oven at 80°C for 4 hours.”

Text should be written in past perfect.

“Capped CeO₂ nanoparticles were synthesized employing a simple, one-pot hydrothermal approach using 0.1 M cerium nitrate and 1 M sodium hydroxide as precursors, and using 0.025 M citric acid (CA) and 0.025 M cetrimonium bromide (CTAB) as capping agents. The reaction proceeded at a temperature of 150 C for 18 hours. The powder obtained was washed with a 50:50 EtOH:water solution, subsequently centrifuged at 9558 g for X minutes? and then dried on a tray? in a hot air oven at 80 C for 4 hours. The product was stored in moisture proof vials at 4-8 C until analyzed?”

Please make the formatting and grammatical changes as described. Please communicate the entire procedure in enough detail so that your experiments can be reproduced.

7. **References need to appear sequentially in the manuscript. The reference section should be numbered manually, and the references should correspond with the numbers in the text.**

We have done the above changes in the revised manuscript.

8. **Corrected tense, grammar and sentence structure using your paragraph. The corrected paragraph appears below yours. As communicated to you earlier, please use past perfect tense - not present continuous.**

“We have synthesized CeO₂ nanoparticles employing a simple, one-pot hydrothermal approach using cerium nitrate and sodium hydroxide as precursors, along with various capping agents in water. We have used citric acid (CA) and cetrimonium bromide (CTAB) as capping agents. The capping agent’s concentration is 0.025 M. We have taken 0.1 M Cerium nitrate solution and 1M sodium hydroxide solution. The reaction time of 18 h and the temperature of 150 °C were maintained for all the reactions. The powder obtained from the reaction mixture was washed with an ethanol-water (50:50) mixture. The products are centrifuged at a relative centrifugal force (RCF) value of 9558g and dried in the hot air oven at 80°C for 4 hours.”

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We thank the reviewer for guiding us; we have incorporated the suggestions in the revised manuscript.

This paper has reached content publication status but cannot be published until significant improvements to the language are made. Please have a native English speaker evaluate and correct this manuscript for tense, grammar, sentence structure and composition. I have corrected the abstract but do not have the time to go through this entire manuscript to make language changes. Please write in past perfect tense. Do not

leave out propositions, write in an active voice, use clauses and modifiers correctly, and pay attention to commas and semicolons.

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We sincerely appreciate the time you took to review our work and to correct the abstract. We have taken your feedback with the utmost seriousness. In direct response to your instructions, we have tried to rewrite the manuscript using the past perfect or simple past tense, maintaining an active voice. We had also paid close attention to punctuation, particularly commas and semicolons, to improve readability and precision. These revisions have been applied consistently to the entire manuscript, and we believe the paper has reached the required publication standard.

Thank you for addressing my comments. Accepted. I have added significant content (after figures 12 and 13), please check carefully if you are in agreement with the added content. Please also provide clarification where-ever I have **BOLDED** and **UNDERLINED** content in the manuscript. You can upload this as a separate file in the discussion section.
