International Journal of Progressive Sciences and Technologies (IJPSAT)
ISSN: 2509-0119.

https://ijpsat.org/
Vol. 55 No. 2 February 2026, pp. 456-464

Effect of Ablation Time on the Optical Properties and Functional Groups of Tantalum Oxide Nanoparticles Synthesized in Polyethylene Glycol Medium via Pulsed Laser Ablation

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Abstract- Tantalum oxide nanoparticles (Ta_2O_5 NPs) were synthesized in polyethylene glycol (PEG 4000) liquid medium using pulsed laser ablation (PLAL) technique to study the effect of ablation time on their physicochemical properties. The synthesis process was carried out by ablation of pure tantalum metal using Nd:YAG laser (1064 nm, 78 mJ, 7 ns, 10 Hz) for 30 and 60 min. Characterization tests were carried out using UV–Vis and FTIR spectroscopy. The results showed that increasing the ablation time caused the colloidal suspension to become more turbid, indicating a higher concentration of nanoparticles. UV–Vis spectroscopy showed a characteristic absorption peak in the range of 285–290 nm, with the absorption intensity increasing from approximately 0.20 a.u. at 30 min to 0.40 a.u. at 60 min, without significant peak shift. FTIR analysis confirmed the formation of Ta_2O_5 through the Ta–O–Ta vibration band and demonstrated the interaction between the nanoparticle surface and PEG functional groups, including ether (C–O–C) and hydroxyl (–OH) groups, indicating effective steric stabilization. Overall, longer ablation time enhanced the formation of the oxide network and the stability of the Ta_2O_5–PEG colloidal system.

Keywords: Ablation Time, Tantalum Oxide Nanoparticles, Polyethylene Glycol Medium, Pulsed Laser Ablation

I. INTRODUCTION

Tantalum oxide nanoparticles (Ta_2O_5 NPs) have attracted considerable research interest due to their superior functional characteristics, including a high atomic number, excellent chemical stability, and a wide band gap. At the nanoscale, Ta_2O_5 exhibits unique optical properties, characterized by strong absorption in the ultraviolet region associated with electronic band transitions, while maintaining transparency in the visible light region [7] [8] [14] [16]. These properties make Ta_2O_5 nanoparticles highly promising for advanced technological applications, particularly in fields requiring materials with high density and stable optical performance.

One of the rapidly developing methods for the synthesis of metal oxide nanoparticles is pulsed laser ablation in liquid (PLAL) [2] [11] [13] [15]. This method offers significant advantages, as it enables the production of high-purity nanoparticles without the need for chemical precursors, reducing agents, or additional surfactants [3] [11]. Moreover, PLAL allows direct control of synthesis parameters through the adjustment of laser energy, the type of liquid medium, and ablation time, all of which influence particle yield, particle size, size distribution, and the optical properties of the resulting nanoparticles [3] [4] [15]. Longer ablation durations generally increase nanoparticle concentration; however, they may also promote interparticle interactions and agglomeration, leading to changes in the optical properties and surface chemical characteristics of the nanoparticles [3] [11] [15].

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The selection of the liquid medium in the PLAL process plays a crucial role in laser-induced plasma cooling, particle growth kinetics, and long-term colloidal stability [2]. Polyethylene glycol (PEG) is widely employed as a liquid medium due to its biocompatibility and non-ionic nature. PEG acts as an effective stabilizing agent through steric hindrance, preventing nanoparticle agglomeration. Furthermore, the presence of hydroxyl (–OH) functional groups along the PEG chains facilitates strong surface interactions with metal oxide nanoparticles, which can be confirmed through functional group analysis using infrared spectroscopy [10] [12] [16].

Ablation duration is a critical synthesis parameter that determines the number of laser pulses delivered to the solid target. Variations in ablation time directly influence nanoparticle concentration, interparticle collision frequency, and the evolution of optical and surface chemical properties of the colloidal system. Although increasing ablation time generally enhances nanoparticle concentration, excessively long ablation durations may promote particle agglomeration or alter surface interactions with the polymer medium [3]. To investigate these phenomena, spectroscopic approaches such as Ultraviolet–Visible (UV–Vis) and Fourier Transform Infrared (FTIR) spectroscopy are widely employed [5] [9]. UV–Vis spectroscopy provides information on absorption characteristics and allows estimation of the optical band gap of Ta_2O_5 nanoparticles, while FTIR spectroscopy identifies specific chemical bonds formed between the nanoparticles and PEG chains [9]. However, studies focusing on the effect of short ablation time variations on the spectroscopic characteristics of Ta_2O_5 nanoparticles synthesized in a PEG medium remain relatively limited.

Therefore, this study aims to synthesize tantalum oxide nanoparticles in a polyethylene glycol medium using the pulsed laser ablation technique, with a focus on ablation durations of 30 minutes and 60 minutes. Comprehensive characterization using UV–Vis and FTIR spectroscopy is conducted to elucidate the effect of ablation time on the early-stage formation and physicochemical properties of Ta_2O_5 nanoparticles. The findings of this research are expected to provide fundamental insights and baseline data for the development of tantalum-based functional materials for biomedical and optoelectronic applications.

II. RESEARCH METHOD

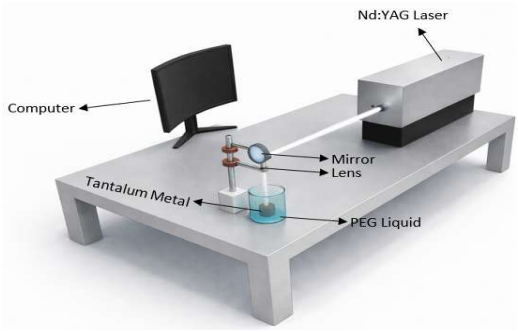

Figure 1. Schematic of tantalum nanoparticle synthesis tool in PEG medium

The primary material used in this study was a high-purity tantalum (Ta) metal target with a purity of 99.99%. The liquid medium employed as the synthesis environment was a polyethylene glycol (PEG) polymer solution with a molecular weight of 4000 Da (PEG 4000). The main equipment used for the synthesis process was a Nd:YAG (Neodymium-Doped Yttrium Aluminum Garnet) laser system operating at a fundamental wavelength of 1064 nm, with a pulse energy of 78 mJ, a pulse duration of 7 ns, and a repetition rate of 10 Hz.

2.1 Synthesis of Tantalum Oxide Nanoparticles

The synthesis of tantalum oxide nanoparticles was carried out using the pulsed laser ablation in liquid (PLAL) technique. A tantalum metal plate and 10 mL of PEG 4000 solution were placed in a glass beaker and positioned beneath a convex focusing lens with a focal length of 10 cm. The Nd:YAG laser beam (1064 nm, 78 mJ, 7 ns, 10 Hz) was first redirected using a silver-coated mirror and then focused onto the tantalum target surface through the convex lens to ensure effective ablation.

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The tantalum target was irradiated for two different ablation durations, namely 30 minutes and 60 minutes. During the ablation process, the beaker was gently moved to allow uniform laser exposure over the entire surface of the tantalum plate and to promote homogeneous nanoparticle formation. The interaction between the laser pulses and the tantalum target in the liquid medium resulted in plasma generation, followed by rapid cooling and condensation, leading to the formation of tantalum oxide nanoparticles dispersed in the PEG medium.

2.2 Characterization Techniques

The physicochemical properties of the synthesized tantalum oxide nanoparticle colloids were analyzed using two main spectroscopic techniques:

2.2.1 UV-Visible Spectroscopy

The characterization of tantalum oxide nanoparticles in a polyethylene glycol (PEG) medium was conducted using an Ultraviolet–Visible (UV–Vis) spectrophotometer to evaluate the light absorbance of the tantalum oxide nanoparticles. The UV–Vis measurements were performed over an electromagnetic wavelength range of 200–700 nm.

2.2.2 Fourier Transform Infrared (FTIR)

FTIR analysis was conducted to identify the functional groups present in the tantalum oxide nanoparticle colloids synthesized in the PEG medium. The measurements were carried out in the wavenumber range of 400–4000 cm^{-1}. This characterization aimed to verify molecular interactions and the capping mechanism between PEG polymer chains and tantalum oxide nanoparticles, which are essential for ensuring steric stabilization of the colloidal system.

III. RESULT AND DISCUSSION

3.1 Analysis of tantalum oxide nanoparticle synthesis in a polyethylene glycol (PEG) medium

Figure 2. Colloidal tantalum oxide nanoparticles in a polyethylene glycol (PEG) medium with different synthesis times: (a) 30 minutes and (b) 60 minutes

Figure 2 shows the colloidal tantalum oxide nanoparticles synthesized in a polyethylene glycol (PEG) medium using the pulsed laser ablation in liquid (PLAL) method with ablation times of (a) 30 minutes and (b) 60 minutes. Both samples exhibit milky white to grayish colloidal suspensions, indicating the successful formation of tantalum oxide nanoparticles as a result of laser ablation of the tantalum target in the liquid medium.

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The colloidal suspension obtained after 30 minutes of ablation appears relatively clearer, which suggests a lower concentration of nanoparticles due to the smaller number of laser pulses delivered to the target. In contrast, the suspension synthesized for 60 minutes shows a noticeably higher turbidity, indicating an increased nanoparticle concentration with longer ablation time. In general, increasing the ablation time in the PLAL process leads to a higher ablation rate and a greater number of nanoparticles dispersed in the liquid medium, which is reflected by the increased turbidity of the colloidal solution [15].

Furthermore, no visible sedimentation or large aggregates are observed in either sample, demonstrating that PEG effectively functions not only as a liquid medium but also as a stabilizing agent for the nanoparticles. The hydroxyl (–OH) functional groups along the PEG polymer chains can interact with the surface of tantalum oxide nanoparticles, thereby suppressing agglomeration and maintaining colloidal stability. This behavior is consistent with previous studies reporting the role of PEG in enhancing the stability of nanoparticles synthesized via laser ablation in liquids [17].

In contrast, tantalum oxide nanoparticles synthesized without the addition of PEG tend to form visible sediments at the bottom of the vial tube, indicating significant particle agglomeration. This comparison clearly highlights the crucial role of PEG in providing colloidal stability to tantalum oxide nanoparticles through its functional properties.

3.2 Microstructural characterization of tantalum oxide nanoparticles in a polyethylene glycol (PEG) medium

3.2.1 UV-Visible Spectroscopy

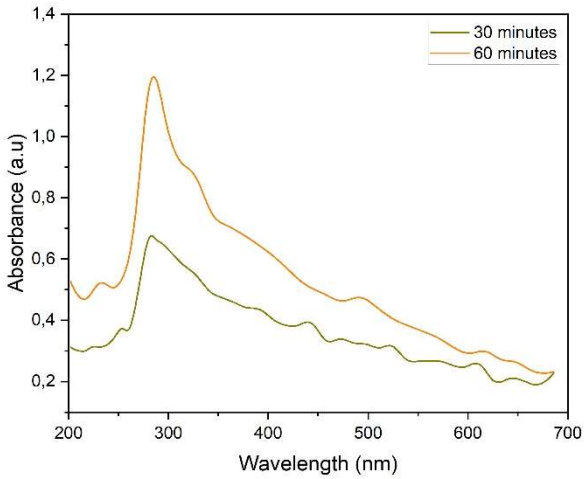

Figure 3. Absorption spectra of tantalum oxide nanoparticles in a polyethylene glycol (PEG) medium for different synthesis times.

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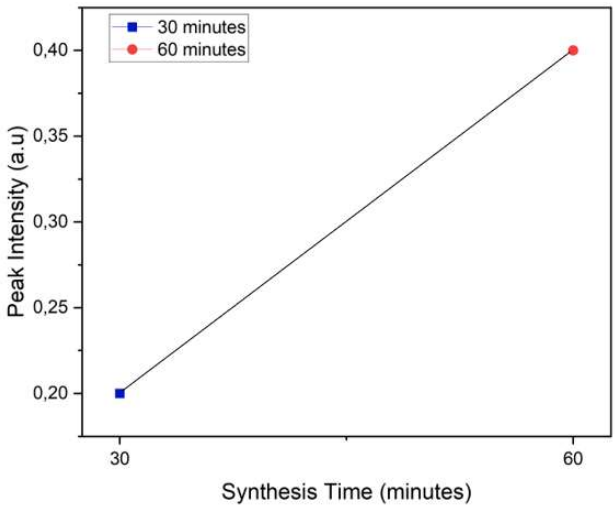

Figure 4. UV–Vis absorbance peak intensity of tantalum oxide nanoparticles in a polyethylene glycol (PEG) medium for each synthesis time.

The UV–Vis spectroscopic characterization results indicate that synthesis time has a significant influence on the formation of tantalum oxide nanoparticles in a polyethylene glycol (PEG) medium. The absorption spectra exhibit a characteristic absorption peak in the ultraviolet region, specifically in the wavelength range of approximately 285–290 nm, confirming the successful formation of tantalum oxide nanoparticles. An analysis of synthesis time variation shows that the sample synthesized for 60 minutes exhibits a higher absorbance intensity compared to the sample synthesized for 30 minutes. In general, extending the synthesis time in the pulsed laser ablation in liquid (PLAL) method enhances the ablation rate of the target material, resulting in a higher nanoparticle concentration, which is reflected by the increased absorbance peak intensity [15].

Although this study is limited to ablation durations of 30 and 60 minutes, a linear trend in nanoparticle concentration enhancement can still be observed. However, for longer ablation durations, it is expected that this trend would deviate from linearity due to a saturation effect caused by self-absorption [14]. In this process, suspended nanoparticles can obstruct the laser beam, thereby reducing the ablation rate over time.

Although direct particle size measurements such as Transmission Electron Microscopy (TEM) were not performed, the sharp absorption peak in the 285–290 nm range without a significant peak shift indicates a relatively narrow size distribution, estimated below 50 nm [16]. This observation is consistent with previous reports showing that metal oxide nanoparticles with sizes below 50 nm synthesized via the PLAL technique display sharp ultraviolet absorption bands in the short-UV region, where the absence of peak shifting reflects particle size stability and minimal further growth during the synthesis process [14] [15].

Based on the Tauc plot method, the absorption peak at 285–290 nm corresponds to a photon energy of approximately 4.27–4.35 eV. This places the absorption edge within the wide band gap region of 3.9–4.0 eV characteristic of high-purity Ta_2O_5 [9]. This correlation confirms that the synthesized nanoparticles are high-purity Ta_2O_5 with stable electronic properties, consistent with the reported wide band gap characteristics of tantalum oxide. Furthermore, the use of PEG as the polymeric medium plays an important role in stabilizing the nanoparticle size through a steric stabilization mechanism, which effectively limits excessive crystal growth and prevents significant agglomeration. This stabilization effect allows the nanoparticles to maintain a small nanometer-scale size even with prolonged ablation time, as evidenced by the stable UV–Vis absorption peak position within the 285–290 nm range [9].

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3.2.2 Functional Group Analysis by FTIR Spectroscopy

The FTIR spectroscopic analysis confirms the successful formation of tantalum oxide nanoparticles and their interaction with the polyethylene glycol (PEG) medium, as evidenced by the presence of several characteristic absorption bands in the FTIR spectra. Functional group assignments were performed based on the wavenumber positions and were further supported by a quantitative analysis of peak intensities to provide a more rigorous interpretation beyond qualitative observations.

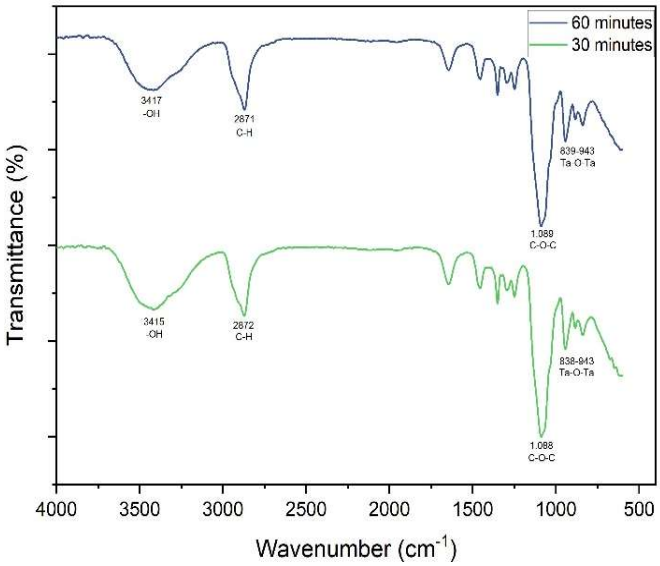

Figure 5. FTIR spectra showing the functional groups of tantalum oxide nanoparticles synthesized in a PEG medium

As shown in Figure 5, the formation of tantalum oxide is confirmed by the appearance of an absorption band at approximately 615 cm^{-1}, which is attributed to the stretching vibration of the tantalum–oxygen (Ta–O) bond. This peak is observed for both synthesis times (30 and 60 minutes), indicating that the basic oxide structure is formed under both conditions. In addition, absorption bands corresponding to ether (C–O–C), alkyl C–H, and hydroxyl (–OH) groups are observed, confirming the presence of PEG and its interaction with the nanoparticle surface.

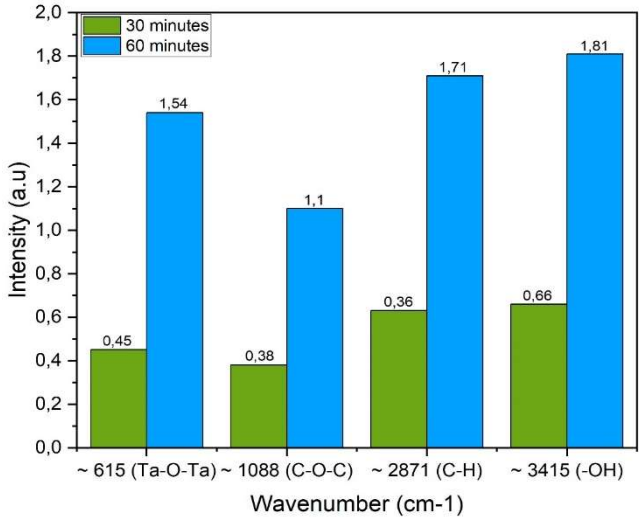

Figure 6. Quantitative FTIR peak intensities of Ta–O, C–O–C, C–H, and –OH functional groups for tantalum oxide nanoparticles synthesized in PEG at different synthesis times

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The quantitative FTIR analysis presented in Figure 6 reveals a systematic increase in peak intensities for all major functional groups as the synthesis time increases from 30 to 60 minutes. The Ta–O peak intensity shows a pronounced increase, indicating enhanced oxide formation with prolonged ablation time. Similarly, the increasing intensities of the C–O–C and C–H bands suggest stronger adsorption of PEG chains onto the nanoparticle surface, forming a more effective capping layer that suppresses particle agglomeration and enhances colloidal stability. The –OH band also exhibits a notable increase in intensity, reflecting enhanced surface hydroxylation and hydrogen-bonding interactions within the PEG–tantalum oxide system.

Table 1. ***Quantitative FTIR peak intensities and functional group assignments of tantalum oxide nanoparticles synthesized in polyethylene glycol (PEG) at different synthesis times***

Wavenumber (cm-1)	Functional Group	Intensity (30 minutes)	Intensity (60 minutes)
615	Ta-O	0,45	1,54
1088-1089	C-O-C	0,38	1,10
2871-2872	C-H	0,63	1,71
3415-3417	OH	0,66	1,81

As summarized in Table 1, the Ta–O peak intensity increases significantly from 0.45 at 30 minutes to 1.54 at 60 minutes, indicating a higher density of Ta–O bonds or an increased concentration of tantalum oxide nanoparticles with prolonged synthesis time. The intensity of the C–O–C band increases from 0.38 to 1.10, suggesting enhanced interaction between PEG ether groups and the tantalum oxide surface. Furthermore, the C–H peak intensity rises from 0.63 to 1.71, reflecting increased PEG chain adsorption, while the –OH peak intensity increases from 0.66 to 1.81, indicating stronger hydrogen-bonding interactions and improved surface stabilization of the nanoparticles.

Overall, the quantitative FTIR analysis presented in Figure 6 and Table 1 demonstrates that increasing the synthesis time from 30 to 60 minutes leads to a systematic enhancement in the intensities of all major FTIR bands associated with Ta–O, C–O–C, C–H, and –OH functional groups. These results confirm that a longer synthesis duration not only promotes the formation of tantalum oxide nanoparticles but also strengthens the interactions between the nanoparticle surface and the polyethylene glycol (PEG) medium, as reflected by the increased intensities of surface-related functional groups. Prolonged synthesis time facilitates more extensive surface functionalization, thereby enhancing nanoparticle–polymer interfacial interactions, improving dispersion, and increasing the overall colloidal stability of the system. This behavior is consistent with previous studies reporting that surface functionalization and modification of metal oxide nanoparticles in polymeric media significantly improve interfacial interactions and dispersion stability within polymer matrices [1] [6] [15].

Quantitative FTIR analysis shows a systematic increase in peak intensity for all major functional groups as the synthesis time increases from 30 to 60 minutes. The Ta–O peak exhibits a significant increase, indicating more intense oxide formation with prolonged ablation time. The increased intensity of the C–O–C and C–H bands suggests stronger adsorption of PEG chains onto the nanoparticle surface, forming a more effective coating layer to suppress particle agglomeration and enhance colloidal stability. Longer synthesis duration facilitates more extensive surface functionalization, thereby enhancing the nanoparticle-polymer interface interaction and improving the dispersion of the colloidal system.

While this study focuses on the Ta_2O_5 system within a PEG 4000 medium, the choice of this medium is based on its superior ability to provide steric stabilization compared to pure water, where initial observations without stabilizers showed rapid sedimentation. The validation of the Ta_2O_5 phase formation was performed by correlating the experimental absorption peaks (285–290 nm) and the FTIR vibration bands (615 cm^{-1}) with established literature values for high-purity tantalum oxide; thus, the use of additional bulk reference materials is represented by credible secondary comparative data. Furthermore, the characteristics of PEG functional groups within the colloid serve as an internal baseline, where intensity changes in the ether and hydroxyl bands provide

direct evidence of polymer adsorption interactions on the nanoparticle surface, which is the primary objective of the surface functionalization analysis in this study.

The authors acknowledge certain limitations in the experimental design of this study. The current investigation focuses on two ablation time points (30 and 60 minutes) to provide an initial proof-of-concept for this synthesis method. Therefore, the observed increase in absorbance and nanoparticle concentration is described as a localized trend within that specific duration. Given the limited number of data points and replicates at this stage, further research involving a broader range of ablation times and statistical triplicate measurements is required to establish a more comprehensive kinetic model and assess the long-term reproducibility of the process. Nevertheless, these preliminary findings serve as a fundamental basis for further optimization in the production of tantalum oxide nanoparticles via the PLAL technique.

While this study successfully demonstrates the formation of Ta_2O_5 nanoparticles in a PEG medium, the authors acknowledge certain limitations in the experimental design. The current investigation focuses on two ablation time points (30 and 60 minutes) to provide a preliminary proof-of-concept for this synthesis method. Consequently, the observed increase in absorbance and nanoparticle concentration is described as a localized trend within this specific duration. Due to the limited number of data points and replicates at this stage, further research involving a broader range of ablation times and statistical triplicate measurements is necessary to establish a more comprehensive kinetic model and assess the long-term reproducibility of the process. These initial findings, however, serve as a fundamental basis for further optimization of tantalum oxide nanoparticle production via the PLAL technique.

IV. CONCLUSION

This study demonstrates the preliminary synthesis of tantalum oxide nanoparticles (Ta_2O_5) in a PEG medium using the PLAL technique. The results indicate that increasing the ablation time from 30 to 60 minutes enhances the nanoparticle concentration, as evidenced by the rise in UV–Vis absorbance intensity from 0.20 a.u. to 0.40 a.u. FTIR analysis confirms the formation of the Ta–O–Ta network at 615 cm^{-1}, where the quantitative increase in peak intensity suggests a higher density of oxide bond formation with prolonged ablation duration. PEG effectively functions as a dispersion stabilizing agent through steric hindrance, evidenced by the molecular interactions between PEG functional groups and the nanoparticle surface. While limited to two time points, these findings provide a proof-of-concept regarding the influence of ablation duration on particle density and surface interactions in the Ta_2O_5–PEG colloidal system.

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