Two examples of using physical mechanics approach to evaluate colloidal stability

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Since Mr. Tsien brought up his idea of physical mechanics, as a new field in engineering science, to public attention in the early 50's of the 20th century, innumerable application examples of physical mechanics approach in diverse fields have manifested its strong vitality increasingly. One of important aspects in applications of physical mechanics is to appropriately choose the microscopic quantity for the system in consideration and build a bridge to connect its relevant microscopic information to its desired macroscopic properties. We present two unique cases of using the physical mechanics approach to study colloidal stability. In the first case we measured the outcomes from artificially induced collisions at individual particle levels, by means of directly observing artificially induced collisions with the aid of optical tweezers. In the second case, by using T-matrix method, the microscopic quantity extinction cross section of the doublet can be accurately evaluated and therefore the measurement range and accuracy of the turbidity methodology for determining the CRC are greatly improved.

Tsien [1] had a pioneering landmark article "Physical mechanics, a new field in engineering science" in 1953. In this article Tsien indicated that the purpose of physical mechanics is to predict the engineering behavior of matter in bulk from the microscopic properties of its molecular and atomic constituents. Originally the concept of physical mechanics was mainly aimed at properties of matter in extreme physical conditions, such as ultra-high temperature, ultra-high pressure and ultra-critical condition created by nuclear detonations and rocket technology, where many experimental measurements for engineering parameters are not possible. Physical mechanics opened up an approach to predict the engineering behavior of matter to eliminate practical measurements [2–4]. Along with time passing, however, general

acceptance of the concept of physical mechanics of from-microscopic-to-macroscopic approach has taken root in the scientific community [5], although the term of physical mechanics may not be always actually adopted.

Innumerable application examples of physical mechanics approach in diverse fields have manifested its strong vitality increasingly. In many simple cases, the methods described in statistical mechanics are used to connect the macroscopic properties of matter with the microscopic properties of its molecular and atomic constituents. In this regard, computer simulation, such as molecular dynamics and the Monte Carlo method are typical examples. The molecular dynamics method, for instance, is used to imitate molecular motions and interactions and convert the related microscopic information to desired properties of matter in bulk with the aid of elementary principles in statistical mechanics. How-

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ever, sometimes how to choose proper microscopic quantities is not clear. In this article we present two special examples of using the physical mechanics approach to studying colloidal stability.

In physics, there are four states of matter: solid, liquid, gas and plasma. Colloids, a typical system for so-called soft matter that has attracted a great deal of attention in recent years, are actually systems formed by two different types of matter, usually of different states. In these systems, one type of matter is dispersed in another. In this article, the colloids we discussed mainly means the systems include mesoscopic particles dispersed in a fluid. The distinguishing feature of colloidal systems is their characteristic size, which is much larger than that of their atomic counterparts, and their characteristic energy, which is much smaller. Colloids are much easier to be observed and detected than atoms and molecules, even with microscopes and laser light scattering. The interactions between atoms are fixed, dictated by quantum mechanics, but those in colloids can be very finely tuned. Therefore colloidal systems to serve as a model system for studies of condensed matter have been widely adopted [6–8]. Moreover, because of their ability to assemble themselves into complex structures, they form the major components of biological systems and technological applications. Apparently colloidal systems ought to come into the scope of physical mechanics.

One of the core aspects of colloid science is investigating colloidal stability and coagulation kinetics. It is thus said that the subject matter of colloid science can be organized around the idea of stability [9]. All the characteristics of colloidal systems change remarkably in the transition from the dispersed state to the aggregated state. Even within aggregated systems, the degree of aggregation varies widely. The coagulation rate constant (CRC) is used to indicate the degree of stability of a colloidal system. One may want to know how quickly the aggregation occurs. Therefore CRC is an important parameter for characterizing the stability and coagulation kinetics of colloidal systems, enabling one to efficiently manipulate the states of dispersion for particular applications.

At the very early stage of the coagulation process, only collisions of single particles which form doublets need to be considered. Therefore，the change of particle number concentrations can be approximately expressed as [10,11]:

$$\left(\frac{\mathrm{d}N_1}{\mathrm{d}t}\right)_{t=0} = -k_{11}N_1^2, \tag{1}$$

$$\left(\frac{\mathrm{d}N_2}{\mathrm{d}t}\right)_{t=0} = \frac{k_{11}N_1^2}{2}, \tag{2}$$

where N_1 and N_2 are the number concentration of single particles and doublets, t is time and k_{11} is the CRC. Higher coagulation rate for a colloidal system means less stability. The upper limit of coagulation rates is solely determined by the diffusive properties of the particles, so the most rapid rate is called the diffusion-controlled rapid coagulation rate (k_{rapid}).

In this paper, sect. 1 presents a microscopic approach to evaluate the colloidal stability and sect. 2 describes how to use microscopic quantities characterizing light-scattering features of colloidal particles to improve the turbidity measurement of determining the CRC.

1 A microscopic approach to evaluate the colloidal stability

The methods of determining coagulation rates, such as light scattering and turbidity, are based on information on the long-term, accumulated effects of the motions and interactions of huge numbers of particles during coagulation; thus, they can be called "macroscopic approaches" and their related quantities are macroscopic quantities. In contrast, the "microscopic approach" is meant to assess the colloidal stability at individual particle levels, by means of directly observing artificially induced collision.

A quantity called the stability ratio W is typically used to describe the degree of stability of a colloidal system. The stability ratio W is commonly estimated by the ratio of the rapid coagulation rate (k_{rapid}) to the coagulation rate (k) of the system in consideration [12–15]:

$$W = k_{rapid} / k = R_{rapid} / R. \tag{3}$$

Here $R = (1/\tau_0)(\mathrm{d}\tau/\mathrm{d}t)_0$ is the relative turbidity (τ) change rate at the initial stage of aggregation (the subscript 0 means the beginning time of the aggregation). The reciprocal of the stability ratio $1/W$ is the so-called "sticking probability" or "collision efficiency", the fraction of successful collisions of two particles to form a doublet. We can see that to obtain W, one needs not only a measurement of R for the sample itself, but also must prepare an additional sample characterized by rapid aggregation to acquire R_{rapid}.

The stability ratio W of eq. (3) is physically equal to the ratio of total number of particle collisions (n) to number of collisions leading to permanent doublets (n_c) [14,15]:

$$W = n/n_c. \tag{4}$$

The reciprocal of W is the "sticking probability", $p = n_c/n$ [16–18]. For slow coagulation or reaction-limited cluster aggregation (RLCA), the energy barrier prevents every encounter from becoming an effective aggregation. Only a fraction, $1/W$, of collisions are successful. The difficulty of using eq. (4) is that Brownian motions make particle collisions take place at unpredictable locations; therefore, one has no way to observe particle collisions and their outcomes to get a true n_c. To solve this problem, optical tweezers (or optical trap) [19,20] have been used [21] to catch two particles, and bring them together for a collision in an area observable by a microscope, see Figure 1. By observing this "artificially induced collision", it is possible to check what

occurs when they are released from the trap: namely, whether they stick together or continue to separate after their collisions. After sampling n pairs of particles for such artificial collisions, a sticking probability can be estimated by the ratio of the number of sticking pairs n_c to sampling pairs n.

When two particles are caught in the optical trap at time t =0 and released at the time $t= t_{dur}$, the total (or accumulated) sticking probability $P(t_{dur})$ for the particle pair to stick together can be deduced to have the following expression [21]:

$$P = 1 - e^{-pft_{dur}}, \tag{5}$$

where p is the sticking probability after a single collision of a particle pair in the optical trap, and f is the collision frequency in the trap.

Figure 2 shows a typical curve of the sticking probability P vs. t_{dur}, obtained from experiment [21]. This curve, showing exponential growth of P with t_{dur}, is basically consistent with prediction of eq. (5). The major difference is that there is a great jump at the beginning of the induced collision, instead of starting from zero at t=0, as indicated by eq. (5).

A possible explanation for this difference is that, at the beginning of trapping, the trapping speed brings the particle pair into the "compact status" with higher collision frequency, and then quickly falls into a relaxed status with lower collision frequency [22,23]. This change in collision frequency causes an abnormally increasing rate of total sticking probability P appeared at the beginning, and then diminishes to a smaller, normally-increasing rate corresponding to the relaxed status.

Based on the feature of the P-t_{dur} curve in Figure 2, the approach for achieving the sticking probability p (or the stability ratio W) from the experiments of artificially induced collisions has been proposed, and the results for the
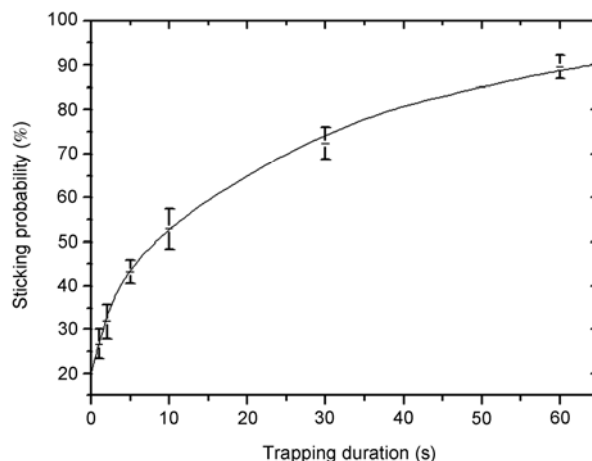
Figure 2 The dependency of the total sticking probability P on the trapping duration t_{dur} [21].

stability ratios W are consistent with those from turbidity measurements [21,24]. Although f seems to be an important parameter for particle collision, the proposed approaches have bypassed the necessity of determining the value of the collision frequency f [21,24].

One of the superiorities of the microscopic approach as compared to the turbidity method is that the former needs only performance of measurement on the suspension sample itself; the latter needs not only the sample itself for k evaluation, but one must also prepare an additional sample to acquire k_{rapid}. For an unknown suspension, this is not always possible. For instance, the methods using eq. (3) cannot, in general, be applied to sterically stabilized systems [21]. An additional advantage of this procedure is that only a small amount of dispersion solution is needed for sampling tests (about 0.1 mL).

The reason for existence of two statuses of the artificially induced collisions—compact and relaxed states—in an optical trap is explained by a Brownian dynamics simulation on the collision-sticking dynamics of two colloidal particles in an optical trap [25]. In this simulation, various contributing factors—including the DLVO (Derjaguin-Landau-Verwey-Oberbeek) interaction of particles, hydrodynamic interactions, optical trapping forces on the two particles, and the Brownian motion—were all considered. The simulation results have reproduced the relevant features of the $\ln(1-P)$-t_{dur} curve found in the experiments.

In this study the microscopic quantities we are interested in are the collision frequency and collision efficiency, and the macroscopically measurable quantity is the total (or accumulated) sticking probability $P(t_{dur})$. The connection between these two quantities is shown in eq. (5).

2   Determination of coagulation rate by the turbidity method at microscopic level

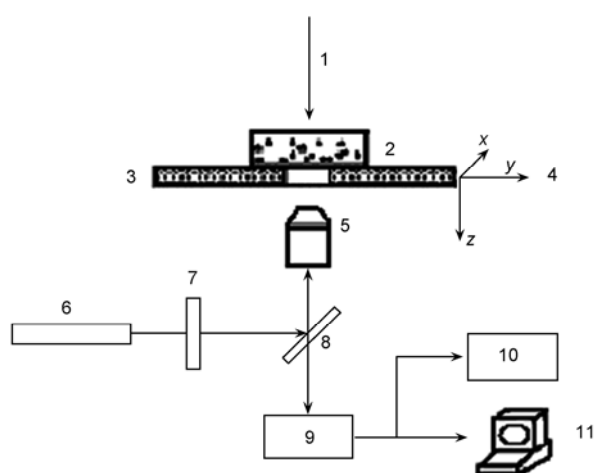Turbidity is a measure of light loss of the transmitted beam.

Figure 1 Experimental setup: 1=illumination light; 2=sample cell; 3= sample stage; 4=3D motion; 5=objective; 6=light source of optical tweezers; 7=electronic shutter; 8=dichromatic mirror; 9=CCD camera; 10= video recorder; 11=computer [21].

Therefore, the information on particle aggregation can be extracted from the change in turbidity, which is associated with the change in characteristics of the light scattered and absorbed by particles due to coagulation.

Turbidity measurement of the coagulation rate has been extensively adopted [26,27] because of its simplicity and ease of implementation. The purpose of the turbidity measurements here is to determine the rate constant. The turbidity measurement becomes possible only when the turbidity change of the system has some connection with aggregation process. If suspensions are stable, their turbidity will keep a constant value (here other affecting factors, such as sedimentation, are excluded) and the rate constant will be zero. In the initial stage, most of particles are singlets and each of them has the same extinction (or attenuation) cross section. However, for stable suspensions, these singlets have zero contribution to the turbidity change and therefore have no influence on the measured rate constant. Microscopically, if the suspensions are not stable, two primary particles are combined to form a doublet during coagulation in the early stage of the aggregation. In this case there may be a corresponding change in their extinction sections resulting in the turbidity change of the system. Thus the turbidity measurement becomes possible only when extinction sections of two singlets are not equal to the extinction section of one doublet.

If a monochromatic incident light beam of intensity I_0 traverses a suspension of length (l), and its transmitted intensity becomes I_{trans}, then the turbidity of the suspension (τ) can be expressed as [11]:

$$\tau = (1/l)\ln(I_0 / I_{trans}). \qquad (6)$$

At the early stage of the aggregation, only single particles and doublets exist and the turbidity can be expressed as $\tau = N_1 C_1 + N_2 C_2$, where N_1 and N_2 are the number concentration of single particles and doublets, respectively; C_1 and C_2 are the extinction (or attenuation) cross sections for single particle and doublet, respectively, and τ is the turbidity [11,27]. What we are concerned with is the time-dependent change of the turbidity, because it is this change which reflects how quickly particles of the system are aggregating. For unstable suspensions, in the early stage of the aggregation only one process basically exists: two primary particles are combined to form a doublet. In this case, if $2C_1 \neq C_2$, there will be a corresponding change in extinction sections for the suspension resulting in the turbidity change of the system. The change rate of turbidity due to the aggregation of single particles can be written as [11,27]:

$$\frac{d\tau}{dt} = C_1 \frac{dN_1}{dt} + C_2 \frac{dN_2}{dt}. \qquad (7)$$

Combining eqs. (1), (2) and (7), the CRC can be connected with the rate of turbidity change in the turbidity measurement by the following equation [27]:

$$k_{11} = \frac{\left[d(\tau / \tau_0)/dt \right]_0}{\left[(C_2 / 2C_1) - 1 \right] N_1}, \qquad (8)$$

where τ_0 is the turbidity at the coagulation starting time $t=0$. Assuming the denominator in eq. (8) can be taken to be a constant, the coagulation rate will be proportional to the quantity of $R = \left[d(\tau / \tau_0)/dt \right]_0$, the relative rate of the turbidity change, which is the slope of the (τ / τ_0-t) curve at $t=0$ and is used above (in eq. (3)). Thus, $|R|$ is usually used to represent the relative coagulation rate. It has been shown [11] that even for the same dispersions, the slope of the line of τ versus t may be positive or negative, depending on the wavelength λ used.

The plot in Figure 3 shows the dependence of R on λ (as well as the dimensionless size parameter $\alpha = 2\pi a/\lambda$) for suspensions consisting of PS (polystyrene) particles ($a=250$ nm).

From Figure 3, we can see that R varies dramatically with λ: not only is its magnitude very different at different λ, but its signs may also change from negative, through zero, to positive. When two primary particles are combined to form a doublet during the coagulation, there will be a corresponding change in their extinction sections. If $(2C_1) < (C_2)$, $(d\tau/dt) > 0$ (namely $R > 0$); if $(2C_1) < (C_2)$, $R < 0$. That the case of $R = 0$ corresponds to the extinction section of two singlets is, by coincidence, exactly equal to that of one doublet $(2C_1 = C_2)$. If the measurement is performed at the λ with $R = 0$, then R will have no response to the actual coagulation process (the "blind point" for measurement). This means that in the zone of the λ with $R \approx 0$, the change in the turbidity during coagulation completely loses its sensitivity to the change in particle number N of the suspensions. That is, there is a zero-sensitivity zone (or the blind zone) for the turbidity measurement around $\lambda = 320$ nm, where R, the magnitude of the slope of τ-t curve at $t = 0$, is too small to distinguish (the signal is covered by noise) [11]. Thus, to enhance the signal-to-noise ratio and reduce the uncertainties of the measurement, one should perform the turbidity measurement at a proper incident light wavelength λ, where the magnitude of R is sufficiently large [11].

To determine the absolute CRC from turbidity measurements, one of the most difficult problems to overcome the dimensionless parameter $F = [(C_2/2C_1)-1]$, which is referred to as the optical factor, in the denominator of eq. (8). R can be obtained from the turbidity measurement. This optical factor F has to be quantitatively calculated by means of light scattering theory.

The calculation of the optical factor is involved in the evaluation of extinction cross sections for single particle and doublet. It has been shown that all theories used in the
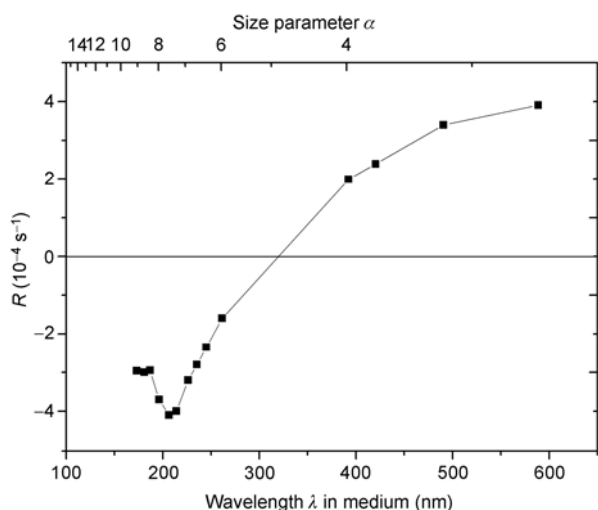
Figure 3 The relative rate of the turbidity change at $t \rightarrow 0$, $R = (1/\tau_0)$ $(\mathrm{d}\tau/\mathrm{d}t)_0$, versus wavelength λ and the size parameter α [11].

past, including Mie theory with coalescence approximation, RGD theory, RGD with coalescence approximation and the modified RGD theory [27], have limitations because it cannot address the issue of large particles [11].

Thus the measurement of CRC by using the turbidity measurement with the extinction cross section of doublets, calculated based on the above theories, can yield a reasonable approximation only for small particles.

Sun et al. [11] have shown that there are some inherent problems when small particle suspension is used in the turbidity measurement. For small particles, a high-number concentration of particles is usually required in order to enhance the signal-to-noise ratio in the measurement. This is because scattering intensity decreases towards smaller particles with the sixth power of radius, and suspensions of small particles have very low turbidity. In this case, dispersions of smaller particles at low-number concentration become an almost transparent medium, and therefore the change in its turbidity becomes very difficult to detect accurately. The turbidity of smaller particles can be enhanced by increasing the number-concentration of particles. However, a high-number concentration of particles will make the coagulation occur too quickly, thus causing the linear portion of the turbidity-time curve to become very short before it curves. In this case, the measurement usually needs to be done within seconds [28]. This causes large errors associated with the data fluctuations and the insufficient data collection time. In contrast to small particles, when large particles with a low-number concentration are used, the linear portion of the curve of τ/τ_0-t can be quite long, as shown for particles of a=250 nm thus allowing the measurement of R to be more accurate [11].

To solve the problem with a quickly-curved line of $(\tau/\tau_0$-$t)$ when high number-concentration of particles is used, Puertas et al. [29] applied the RGD approximation to calcu-

lating the total light scattering cross section of the aggregate, taking into account the contributions from pairs of particles with zero, one, and two particles between them, and developed a technique for getting the CRC from the turbidity measurement by fitting the curve of turbidity change for a longer time.

An additional disadvantage of using small particles is that, if the methodology is only for small particles and small α, one will greatly restrict the range of λ which can be selected. As mentioned above, R varies with λ, so to reduce experimental errors one should choose a λ at which R is large [11].

Using the T-matrix technique, the extinction cross sections of single particles and doublets, and thus the optical factor F, can be calculated without additional assumptions. More comprehensive discussion of the technical details and data calculations of the optical factor for different particle sizes and wavelengths is given by Xu et al. [30].

To assess the quality of various theoretical treatments (calculation of the optical factor F) dealing with the turbidity measurement for determining the rate constants, a useful criterion was proposed [11]. This criterion is based on a reasonable prediction that optical factor F should ensure that eq. (8) yields an identical CRC, no matter what wavelength is used in the turbidity measurement.

Comparisons for the absolute CRCs determined by eq. (8), with the optical factor derived from the T-matrix method and other theoretical approaches, are presented in Figures 4–6 [11]. All points of the rate constants in each figure are based on the same original experimental data (namely, the slopes of the linear portion of the $(\tau/\tau_0$-$t)$ curves at different incident light wavelengths). In all of these Figures, two different scales are used for each abscissa, and are marked by both the wavelength λ and the size parameter α. The particle sizes of a=170, 250 and 500 nm are used, respectively, in Figures 4–6 [11]. These plots show that except for the T-matrix method, none of the previous theories can give an unaltered quantity for the whole range of the wavelength λ, or the size parameter α, used in the experiments. There are some theories which, in many cases, even yield negative values for the rate constants. These values seem to be trivial.

Figure 4 (Suspensions of particles with radii a = 170 nm) shows that the data from the RGD (real) and T-matrix are actually overlapping when $\alpha \lesssim 2$, meaning that, for small particles and small α, the T-matrix treatment coincides with RGD (real) method. This fact indirectly proves that use of the T-matrix solution is correct, because it is known that the RGD (real) is a good approximation when treating doublets for small α and small particles. In addition, for these-sized particles, all the data of the rate constants from the four previous theories are basically within the same order of magnitude when $\alpha < 3$, although still showing significant deviations from a constant value.

Figure 5 (suspensions for particles of $a = 250$ nm) shows that among the four previously discussed theories, the RGD (corrected) has the best performance and the RGD (real) follows closely when $\alpha < 4$. There is no data available in the zone ($4 < \alpha < 6$) because of the zero sensitivity zone, as previously mentioned. When $\alpha > 6$, RGD (coalescence) and RGD (real) yield negative values for the rate constants, negating the results.

Figure 6 (suspensions for particles of $a = 500$ nm) shows that all previous theories failed to give reasonable data for the rate constant for such-sized suspensions when $\alpha > 6$. Therefore, the T-matrix solution is the only surviving method with a good performance throughout the whole α range.

It is readily apparent that this new approach is effective in significantly extending the applicability range of the turbidity methodology and increasing measurement accuracy [11,31]. From this study we note that:
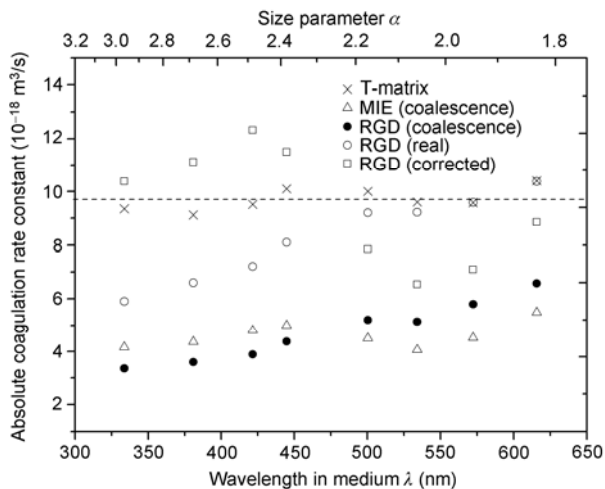

Figure 4 Absolute CRC derived from different theoretical treatments for suspensions of particles with radii a=170 nm [11].
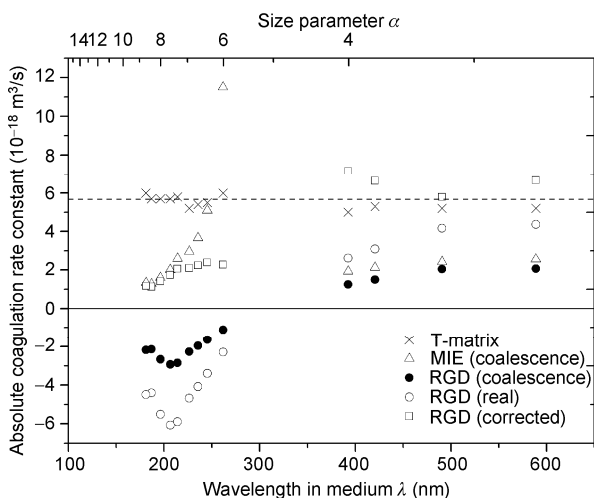

Figure 5 Absolute CRC derived from different theoretical treatments for suspensions of particles with a=250 nm [11].
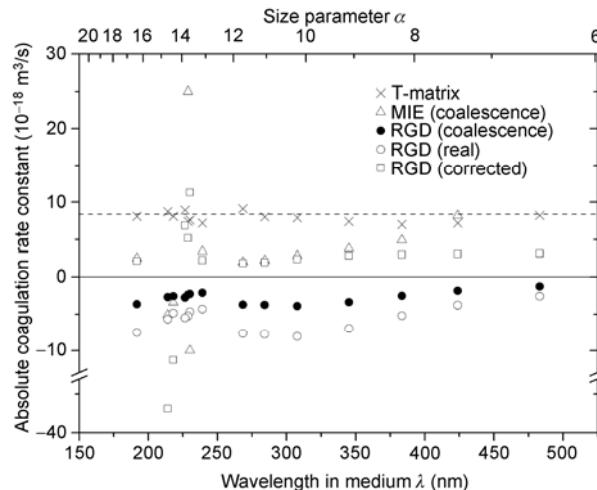

Figure 6 Absolute CRC derived from different theoretical treatments for suspensions of particles with a=500 nm [11].

(1) To achieve a more accurate absolute rate constant, using larger particles is more preferable.

(2) The degree of response of the turbidity change to the coagulation varies significantly with particle size and operating wavelength; at a certain wavelength, the change in turbidity completely loses its sensitivity to the coagulation process (the so-called blind point) that makes the measurement impossible, suggesting the measurement should be performed at a wavelength away from the blind point.

(3) The key microscopic quantity for achieving the CRC from the turbidity measurement in this study is the extinction cross section of the doublet formed during the coagulation process. We have showed the T-matrix method is accurate to evaluate this quantity.

3 Summary

Physical mechanics has shown its exuberant vitality by innumerable application examples in diverse fields in the past. One of important aspects in applications of physical mechanics is to intelligently choose the microscopic quantity for the system in consideration and connect it to the desired macroscopic properties.

In this paper we describe two examples of using the physical mechanics approach to study the colloidal stability.

In the first one, in contrast with traditional macroscopic measurement methods, a "microscopic approach" to assess the colloidal stability is suggested. This approach checks the outcomes from artificially induced collisions at individual particle levels, by means of directly observing artificially induced collisions with the aid of optical tweezers.

In the second case, by using T-matrix method, the microscopic quantity extinction cross section of the doublet can be accurately evaluated and therefore the measurement range and accuracy of the turbidity methodology for deter-

mining the CRC are greatly improved.

One of the advantages to solve scientific problems at the microscopic level is that we can have a good chance to gain insight into the physical nature of the problems. For instance, for the turbidity method, it is shown for the first time that when extinction sections of two singlets are equal to the extinction section of one doublet, turbidity method will be invalidated (blind point), suggesting the measurement should be performed at a wavelength away from the blind spot.

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