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Investigation of the flocculation and sedimentation of TiO_2 nanoparticles in different alcoholic environments through turbidity measurements

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A B S T R A C T

Turbidimeters are low-cost devices, which are widely used for suspended sediment monitoring (SSM). The specific turbidity is typically relative to 1/d (d is the diameter of particles) regarding the suspensions with mono-sized sphere-shaped particles. As the production of the dispersed suspension of TiO_2-NPs is vital for their photocatalysis applications, turbidity provides a test to measure the dispersion of TiO_2-NPs. In the present paper, the performance of a self-manufactured turbidimeter device has been investigated using the suspensions of TiO_2-NPs in different alcoholic media. The results showed that over time, the intensity of light passing through the upper part of the test tube containing TiO_2-NPs suspension increased, suggesting the settlement of TiO_2 nanoparticles. In the middle part of the test tube; however, an almost stable trend was observed, which was more evident in the case of isopropanol with higher viscosity. The results also illustrated that there was no relation between the concentration of suspension and the value of transmitted light for concentration below 0.08 g/l; however, for concentrations above that, the intensity of transmitted light decreased with the increase of suspension concentration.

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1. Introduction

In order to better comprehend and control the sedimentation process of fine particles, field tests, such as particle size distribution (PSD) and suspended sediment mass concentration (SSC) with excellent temporal resolution are crucial. Real-time tests of SSC are indispensable for curbing environmental issues, reducing the abrasion of turbines, and controlling container sedimentation. Several methods are available for suspended sediment monitoring (SSM) and have been widely studied in different standards and literature [1, 2].

Turbidimeters are low-cost devices, which are largely applied for SSM. Using this method, the absorption, transmission, or the back-scattered intensity of samples are tested based on the angle between the receiver and the incident light. Turbidimetry is largely utilized for monitoring suspended solid particles and provides an auxiliary and effective way against the barriers of traditional SSC measurements. Turbidity presents the transparency grade of suspensions and solutions in which light scatters in the media. As a result, by the increase of the intensity of scattered light, the turbidity level increases [3, 4]. The turbidity of suspensions is dependent on the amount of suspended, insoluble, and solid particles with fine sizes in the medium [5]. A variety of turbidimeter types, which are used for submerged or in-line set-ups are accessible. The obtained results of turbidity are transformed to SSC depending on the related sediment mass concentration, evaluated by the gravimetric method (SSC_G) [6-8]. In usual turbidimeters, which employ near-infrared radiation or visible light as an emission source, SSC measures are almost linearly related to turbidity in dilute suspensions. The specific turbidity of suspensions with high turbidity might decline because of the numerous scattering of light. Regarding the suspensions with mono-sized and sphere-shaped particles, the specific turbidity is normally relative to 1/d in which d is the diameter of particles. The specific turbidity of graded particles relies on the width of the PSD, which is considered in the Sauter mean diameter (SMD) [5].

The calibration of turbidimeter with standard samples of suspended sediment is crucial because spectral responses resulted from turbidimeter have not general connectivity with the SSC. The connection between SSC and turbidity is dependent on various properties of suspended sediment including grain size [9, 10], color [11-14], mineralogy of the sediments [15-17], shape, and particle properties such as refractive index, density, and surface roughness [7, 8], as well as the concentration of suspended particles. The turbidity of a suspension with a surfactant is inferior to the one without surfactant [18].

Light scattering techniques, particularly nephelometric turbidimetry, present many benefits such as limited sample-volume requirement, reduced interference, higher detection range, and elevated sensitivity [19]. Consequently, these techniques have been extensively utilized in sediment and colloid transport studies [10], water resource recycling [20], and analysis of flocculation [21]. The turbidimetric facilitates the inves-

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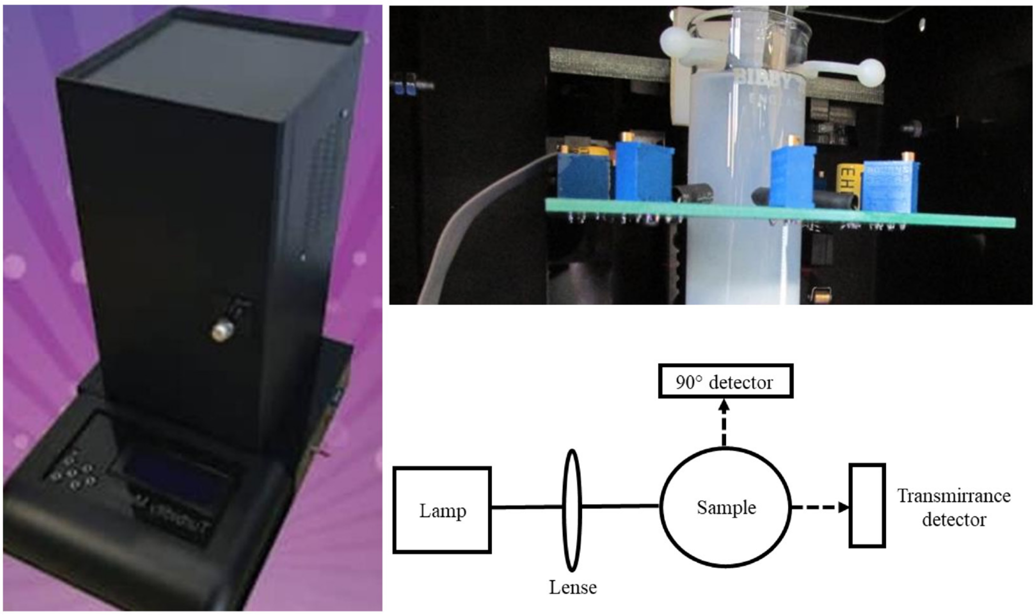

Fig. 1. The self-manufactured set-up functioning with IR light source utilized for turbidity measurements.

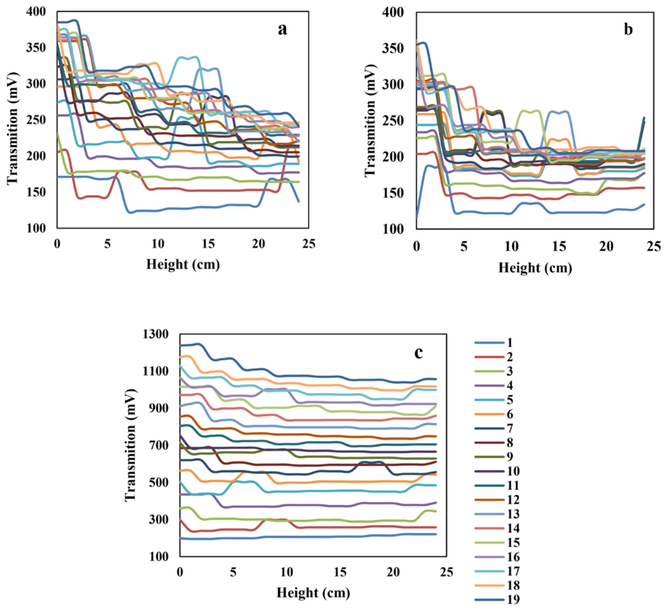

Fig. 2. Changes in light intensity along with the height of the test tube per cycle in methanol- (a), ethanol- (b), and isopropanol-based (c) suspensions.

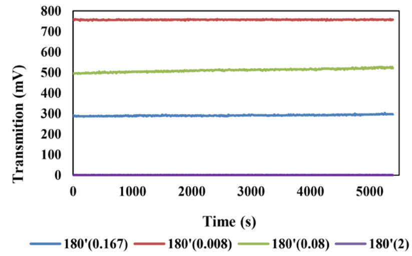

Fig. 3. The changes in light transmission overtime at different concentrations using the isopropanol alcohol.

tigation of the potency of antimicrobials and is a more rapid option than the agar diffusion due to its ability to provide the resulted data in less than 4 hours. Additionally, this technique could be miniaturized [22].

Titanium dioxide (TiO_2) can be used in various industries, such as catalysis [23], photocatalysis [24], nano-paint (self-cleaning) [25, 26], water treatment [27] and materials for air purification [28]. In healthcare, it is used as an antibacterial agents [29] and anti-cancer [30].

TiO_2 nanoparticles (TiO_2-NPs) are widely studied for their photocatalysis applications in water purification because of their excellent catalytic behavior, significant physio-chemical stability, safety, and low cost [31-33]. For their photocatalysis uses, it is crucial to employ appropriate preparation methods of titania colloidal suspensions [34]. Electrophoretic deposition [35] and tape casting [36] methods, which commonly utilize non-aqueous solvents, usually alcohols, as the suspension media, have been employed for preparing titania coatings. The dispersion degree of TiO_2-NPs in the suspension greatly affects the photocatalytic efficacy of the colloidal state [37] and the titania coatings achieved from it [38-40]; thus, the production of the properly dispersed suspension based on TiO_2-NPs is vital. Turbidity provides a test to measure this state and is related to the dispersion level of TiO_2-NPs and their ability to stay dispersed in the suspension [32, 33].

Several studies have investigated the stability of TiO_2-NPs in different solutions using turbidity measurements. Farrokhi-rad et al. [41] have studied the stability of TiO_2-NPs in different alcohols (methanol, ethanol, and butanol). They observed that despite decreasing the zeta potential, the stability of TiO_2-NPs increases with increasing alcohol molecular size (from methanol to butanol). Sakurai et al. [42] have also examined the impact of seawater constituents and dissolved oxygen (DO) on granular Pt/TiO_2 photocatalytic using turbidity measurements.

To the knowledge of the authors, turbidity measurements in combination with several alcoholic media lack in the number. Moreover, none of the related researchers have studied the performance of the self-manufactured turbidimeter in their study. In the present paper, the performance of a self-made turbidimeter device has been assessed using the suspensions of TiO_2-NPs in different alcoholic media. In the following section, an overview of materials and measurement methods is given; and in section three, the results of our study have been discussed in detail.

2. Materials and Methods

Titania nano-powder (Merck, Germany) with an average particles size of 5 nm was used as raw particles. Methanol (99.99%, Merck, Germany), ethanol (99.8%, Merck, Germany), isopropanol (99.9%, Merck, Germany) were used as the solvents. Table 1 presents the properties of the materials used for this study.

To investigate the turbidity and deposition behavior of TiO_2 nanoparticles in different alcoholic environments and the effect of alcohol viscosity on the turbidity level, suspensions of this powder in ethanol,

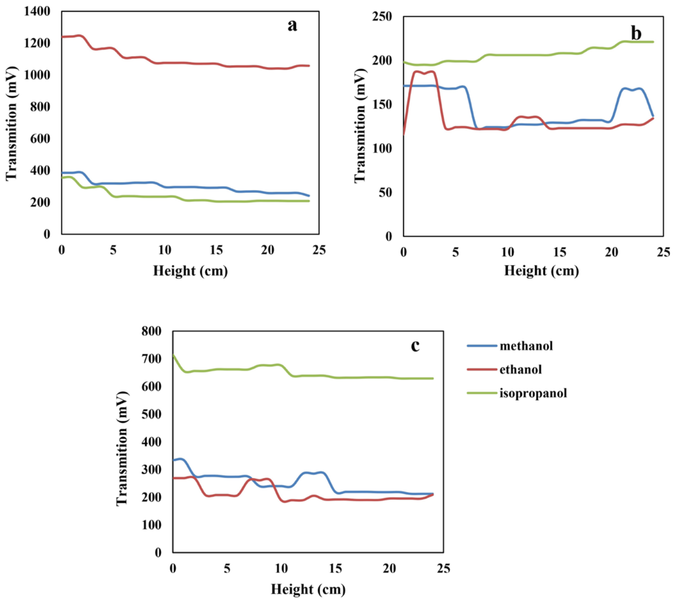

Fig. 4. Changes in light intensity along with the height of the test tube in different alcoholic environments: last cycle (a), first cycle (b), 9th cycle (c).

methanol, and isopropanol with a concentration of 2 g/L were prepared. After ultra-sonication, each sample was tested cyclically for 10 minutes. In this way, once every 15 minutes, the turbidimeter detector moved in the direction of the height of the test tube and sampled it, and for each sample, this cycle was performed 19 times. The suspensions were characterized using a self-manufactured turbidimeter to measure the turbidity of the suspension over time (1 to 24 hours). Fig. 1 presents the self-manufactured set-up functioning with IR light source utilized for turbidity measurements.

In the next step, different slurries containing P25 ceramic powder and isopropanol solution with different concentrations including 0.01 (0.167 g/l), 0.005 (0.08 g/l), and 0.0005g (0.008 g/l) of P25 in 60 cc of isopropanol were prepared to determine the stability, adsorption, and transmittance of a suitable concentration range for working with a turbidimeter. It should be noted that the parameter T indicates the stability of the slurry and is calculated from the following equation:

$$T = I_{90} / (I_{180} + I_{135} + I_{45}) \quad (1)$$

The amount of radiation absorption is also calculated from the following equation:

$$A = - \log (I / I0) \quad (2)$$

Table 1.
The properties of materials

Material	Chemical formula	Viscosity (mPa.s), at room temperature	Grade value	Company
P25 Titania powder	TiO_2	-	Reag. Ph Eur	Merck
Methanol	CH_3OH	0.543	ACS,ISO,Reag. Ph Eur	Merck
Ethanol	C_2H_5OH	1.2	ACS,ISO,Reag. Ph Eur	Merck
Isopropanol	$CH_3CH(OH)CH_3$	2.1	ACS,ISO,Reag. Ph Eur	Merck

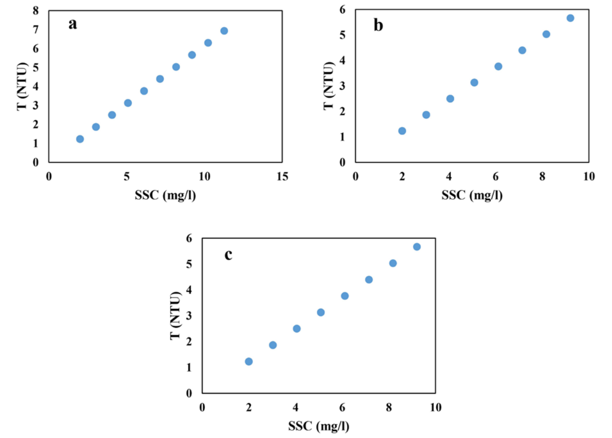

Fig. 5. The turbidity of slurries containing P25 ceramic powder in isopropanol with different concentrations of 0.167 (a), 0.08 (b), and 0.008 g/l (c).

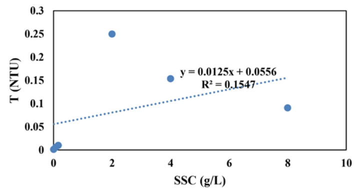

Fig. 6. The turbidity change of slurries vs. P25 concentration.

3. Results and Discussion

In this study, turbidity value is used to determine the ability of the TiO_2-NPs to remain dispersed in the suspension. High turbidity reflects the dispersion of NPs in suspension while low turbidity means most NPs are sediment to the bottom of suspension. To demonstrate the stability of TiO_2 NPs in different alcoholic solutions, a turbidimeter device was used. Fig. 2 represents changes in light intensity along with the height of the test tube during different time cycles in methanol-, ethanol-, and isopropanol-based suspensions. Fig. 3 illustrates the changes in light transmission over time at different concentrations.

Fig. 2 demonstrates that over time, the intensity of light passing through the upper part of the test tube increases, which presents the settlement of TiO_2 nanoparticles. Also, in the middle part of the test tube, an almost stable trend is observed, which is more evident in the case of isopropanol, which has a higher viscosity and can contribute to the higher light transmission by facilitating the settlement of nanoparticles. Fig. 3 also illustrates that there is no relation between the concentration of suspension and the value of transmitted light for concentration below 0.08 g/l; however, for concentrations above that, the intensity of transmitted light decreases with the increase of suspension concentration.

In Fig. 4(a), the last cycle of the samples has been drawn and compared. From the obtained results, it is observed that after the same time, the light intensity (which was initially between 100 and 400 for all three samples) in the isopropanol-based suspension increased much more (to about 1200), which indicates less stability of the suspension where more particles are deposited compared with the other two samples. In addition, the amount of the change of light intensity from the top to the bottom of the test tube is greater for isopropanol. This process is smoother in the case of ethanol and methanol. Fig. 4(b) and Fig. 4(c) also compare the first and ninth cycles, respectively. By comparing these three diagrams, an increase in the intensity of light passing through the upper part of the tube is observed over time. Also, heterogeneity of structure and light transmission in ethanol and methanol environments is seen in the first and ninth cycles compared to the last cycle.

According to the famous Stokes-Einstein equation [43]:

$$D = \frac{K_B T}{6\pi\mu R_0} \quad (3)$$

Where D is diffusion coefficient, k_B is Boltzmann's constant, T is the temperature (K), μ is solvent viscosity, and R_0 is the solute radius.

The Stokes-Einstein equation presents that diffusion is inversely related to viscosity. This means that the higher the viscosity of a material, the harder it is to diffuse. On the other hand, according to the observations, particle settling in the isopropanol medium occurred earlier, which can be related to the higher viscosity of this alcohol compared with the other two samples (however, it was expected that the viscosity would further prevent the particles from diffusing and settling). The viscosity is likely related to the replacement of the precipitated particles with other ones in which high viscosity prevents this phenomenon and the stability of the suspension increases.

Fig. 5 presents the turbidity of slurries containing P25 ceramic powder in isopropanol with different concentrations of 0.167, 0.08, and 0.008 g/l. Fig. 6 illustrates the turbidity change of slurries versus P25 concentration.

Figures 5-6 demonstrate that the turbidity of suspensions having concentrations below 2 g/l, decreases with the decrease of concentration. Conversely, this trend changes for the suspensions with a P25 concentration of more than 2 g/l, in a way that the turbidity of these suspensions decreases by increasing the slurry concentration. As a result, the highest turbidity is associated with the suspension having the P25 concentration of 2 g/l.

Hak et al. [31] have also studied the stability of the TiO_2 NPs to remain dispersed in aqueous media with and without surfactants. Turbidity was measured at 1, 2, 3, and 4 hours from re-dispersion time to get a complete time-dependent measurement. The turbidity of TiO_2 suspensions as a function of time showed that at 2.5 wt.% of surfactant, turbidity of all suspensions is in the range of 35 to 45 at 1-hour measurement. These suspensions were more turbid compared to the control sample (TiO_2 suspension without surfactant) and surfactant solution. This means that in the presence of surfactants most NPs are remained stable to disperse in aqueous media but without surfactants, NPs are easily sedimented to the bottom. After 2 hours, the turbidity decreases to about 10 to 20 for SDS and CTAB and about 10 to 15 for PF-127. These values remain unchanged for the third and fourth hours. This indicates most NPs are settled to the bottom of suspension after 2 hours, leaving the surfactant-stabilized NPs to remain dispersed in the suspension.

Wang et al. [44] have also quantified the settling profiles of nanosized TiO_2 and TiO_2/graphene oxide in glass cylinders in terms of turbidity variations as the function of sedimentation time. According to the turbidity variation, sedimentation of TiO_2/GO could be divided into two domains. In the first domain (before 10 min), the turbidity decreased rapidly from around 5200 NTU to less than 500 NTU, and more than 90% of TSS was removed. In the second domain (after 10 min), the turbidity decreased slowly and took another 5 h to become clear (turbidity<30 NTU). The slow sedimentation might be due to the presence of some small TiO_2/GO clusters. Compared to TiO_2/GO, the sedimentation of TiO_2 was much slower and only less than 40% of the particles were settled in 10 min.

4. Conclusion

In this paper, the performance of a self-manufactured turbidimeter device has been studied using the suspensions of TiO_2-NPs in different alcoholic media (ethanol, methanol, and isopropanol). The obtained results from the manufactured turbidimeter showed that the intensity of light passing through the upper part of the test tube containing TiO_2-NPs suspension increases over time, suggesting that TiO_2-NPs settle as time passes by. In the middle part of the test tube; however, an almost stable trend of light transmittance is observed, which is more obvious in the case of isopropanol with higher viscosity. The results also suggest no relationship between the concentration of suspension and the value of transmitted light for concentration below 0.08 g/l; however, for concentrations above that, the intensity of transmitted light decreases with the increase of suspension concentration.

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Conflict of interest

The authors declare that there is no conflict of interest.

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