

Physical Characterization of a Method for Production of High Stability Suspension

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Abstract

Suspensions/Dispersions are encountered in a wide range of applications, e.g., liquid abrasive cleaners, ceramics, medicines, inks, paints....etc. In most cases it is necessary to keep the suspension stable for the product lifetime. A new modified differential sedimentation measuring system is suggested and used to identify physical parameters affecting the sedimentation in suspensions. The technique is discussed in details. It is found that particle sizes as well as viscosity of continuous phase are the most important factors governing the stability of a suspension. Empirical relations are extracted to quantitatively describe the weight effect of each factor. The modified measuring system shows good accuracy enough to detect fluctuations in concentration of suspended particles due to their Brownian diffusion, as well as the particles concentrations in the stable suspension. This study confirmed the fact that particles diameters measured by Zetasizer are much greater than those measured by the transmission electron microscope. This study presents a proposal for new technique for particle size separation based on the differential sedimentation in viscose fluids. This new method is a differential viscosity column. The proposed size separation technique may help to separate engineered nano-particles with higher resolution.

Introduction

Suspensions/Dispersions are encountered in a wide range of applications, e.g., liquid abrasive cleaners, ceramics, medicines, inks, paints....etc. In most cases it is necessary to keep the suspension stable for the product lifetime [1].

Particle size is one of the most important parameters in materials science and technology as well as many other branches of science and technology, from medicine, pharmacology and biology to ecology, energy technology and the geosciences. Well dispersed stable suspension is one of the most pharmaceutical formats of drugs. Also the stability of suspension is the top feature in paint industry. Therefore this current work aims to describe quantitatively the parameters that are responsible for the stability of a suspension in such a way that one can, initially, right select specifications of the used components before mixing them to produce a high stable suspension at once. Since the particles size (dispersive phase) is the most important parameter, this study will propose a new technique for size separation either in nano scale or micro scale or a mixture of both.

a) Sedimentation of Nano-particles

Most nano particles have been shown to aggregate once they are hydrated, which has a significant effect on their sedimentation rates. Several studies have addressed the aggregation of different nano particles in simpler aqueous

solutions, including the effect of increasing ionic strength (IS) and different pH levels on the size of aggregates [2–5]. It was expressed that nano-fluids would be prepared by suspending solid particles with the size of less than 100 nm inside a base fluid (continuous phase). Terms “synthesis and Characterization” are widely used in literature, describing preparation phase of nano-fluids. In general terms, it could be stated that nano-fluids include nanometer sized solid particles, fibres, rods or tubes suspended in different base-fluids [6]. This means that a nano fluid is a solid nano scaled discrete phase suspended in a continuous liquid phase. Accordingly, the stability and durability of the nano-fluid depends on the stability of suspending the solid phase in the host liquid. Hence, the stability of a suspension should depend on the nature of the nano particles (particle`s material density, particles size, particles shape, particles surface charge, ...etc.), nature of the host liquid (liquid density, viscosity, temperature, pH, particles concentration, ...etc.) and the nature of the mixture (aggregation of particles, quality of dispersion, zeta-potential, sedimentation rate, Brownian motion ...etc.).

Therefore, effects of above factors should be discussed in some details. The equation of motion of a particle in a continuous fluid should clarify the contribution of each of the factors on the stability of the produced suspension. The

equation of motion should represent the forces acting on the particle and cause its motion. Let F_B is the buoyancy force, F_g is the gravitational force, F_R is the frictional Stock's force and D_s is the the Stoke's diameters correspond to equivalent diameters of hypothetical spheres with the same settling behaviour as the irregular, an isometric particles in question. The particle radius is then $R = D_s / 2$ [7]. D_s may be determined from the measured particle size by the Zetasizer, which is shown later. Hence the equation of motion of the particle moves inside the liquid may be written as:

$$m \frac{d^2y}{dt^2} = F_g - F_B - F_R \quad \text{..... (1)}$$

Where dy is the displacement traveled by the particle during time interval dt . Then

$$m \frac{d^2y}{dt^2} = \frac{4}{3} \pi R^3 \rho_s g - \frac{4}{3} \pi R^3 \rho_l g - 6\pi\eta R \frac{dy}{dt} \quad \text{.....(2)}$$

Dividing by the particle mass m and rearranging this equation:

$$\frac{d^2y}{dt^2} + \frac{9\eta}{2R^2\rho_s} \frac{dy}{dt} + \left(\frac{\rho_l}{\rho_s} - 1 \right) g = 0 \quad \text{.....(3)}$$

where, η is the shear viscosity of the liquid, ρ_l and ρ_s are the density of liquid and solid particles, respectively. Equation (3) is a

second order differential equation whose general solution can be in the form :

$$y(t) = -\frac{2}{9} \frac{R^2 \rho_s e^{-\frac{9\eta}{2R^2\rho_s}t}}{\eta} - \frac{C_1}{9\eta} - \frac{2}{9} \frac{R^2 g (\rho_l - \rho_s)t}{\eta} - C_2 \quad \text{.....(4)}$$

where C_1 and C_2 are constants to be determined from the initial conditions, where $y = 0$ at $t = 0$ and $dy/dt = 0$ at $t = 0$. And hence,

$$C_1 = \frac{2}{9} \frac{R^2 g (\rho_l - \rho_s)}{\eta e^{-\frac{9\eta}{2R^2\rho_s}t}} \quad \text{.....(5)}$$

And

$$C_2 = \frac{2}{81} \frac{R^2 g (\rho_l - \rho_s) (2R^2 \rho_s + 9\eta t)}{\eta^2} \quad \text{.....(6)}$$

Introducing equations (5) and (6) into equation (4) one gets the final solution of the differential equation (3).

The solution of equation (3) can be reached using equation solver software like Maple; it gives directly the solution according the given initial conditions in the following form [8]:

$$y(t) = -\frac{4}{81} \frac{R^4 \rho_s g (\rho_l - \rho_s) e^{-\frac{9\eta}{2R^2\rho_s}t}}{\eta^2} - \frac{2}{9} \frac{R^2 g (\rho_l - \rho_s)t}{\eta} + \frac{4}{81} \frac{R^4 g (\rho_l - \rho_s) \rho_s}{\eta^2} \quad \text{.....(7)}$$

This equation shows that:

- In the first term the quantity $\exp(-t)$ reduces the distance traveled by a particle rapidly

specially in high viscose liquids.

- He second term shows a linear increasing of y over long time which means that particles should possess a steady speed after short time from the time of dropping ($t=0$). This steady speed is the limiting velocity (v).
- The third term is a constant independent of time and it describes the depth in the liquid at which the particle is dropped.

The meanings drawn from equation (7) were guide to modify the well known light scattering technique in order to well determine the particle level inside the liquid at $t=0$, from which one may measure the average particle radius R_p after separate measure of η , ρ_l and ρ_s .

b) Static Light Scattering

Light interacts with matter in two ways:

1. Absorption: the photons (the light) disappear.
2. Scattering: the photons change their direction.

Both of the above mentioned interactions will cause a light beam to be attenuated when passing through a solution of particles. It doesn't matter whether light is being attenuated by scattering or absorption: In both cases the transmitted intensity will decrease exponentially with the thickness "x" of the material the light is passing through. If the attenuation is due to

absorption the transmitted intensity I is usually written

$$I = I_0 \cdot 10^{-\alpha x} \dots\dots\dots(8)$$

Here the quantity α is called absorption coefficient. Whereas if the attenuation is due to scattering the intensity is written;

$$I = I_0 \cdot e^{-\tau x} \dots\dots\dots(9)$$

Here the quantity τ is called turbidity [9]. In case of a suspension from nano metallic particles in viscose solution, the turbidity should depend on the concentration of the particles (weight concentration) as well as the distribution of these particles. Stability of the suspension is another factor affecting the turbidity. Thus stability of the turbidity value may refer to the stability of the suspension. Also experimental recording the rate of turbidity variations over a range of time reflects the sedimentation rate of metallic particles through the solution. In these two indications, the intensity of incident light I_0 and solution thickness x should be fixed.

c) Zeta potential

The development of a net charge at the particle surface affects the distribution of ions in the surrounding interfacial region, resulting in an increased concentration of counter ions (ions of opposite charge to that of the particle) close to the surface. Thus an electrical double layer exists around each particle. The liquid

layer surrounding the particle exists as two parts; an inner region, called the Stern layer, where the ions are strongly bound and an outer, diffuse, region where they are less firmly attached. Within the diffuse layer there is a notional boundary inside which the ions and particles form a stable entity. When a particle moves (e.g. due to gravity), ions within the boundary move with it, but any ions beyond the boundary do not travel with the particle. This boundary is called the surface of hydrodynamic shear or slipping plane. The potential that exists at this boundary is known as the *Zeta potential*.

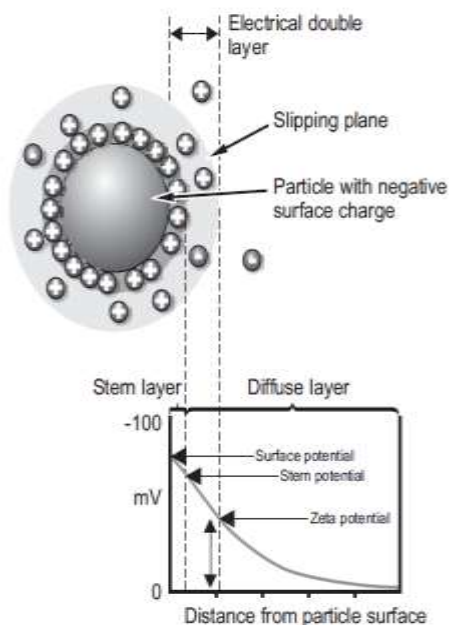


Figure (1): scheme representing the electrical double layer [10].

If all the particles in suspension have a large negative or positive zeta potential then they will tend to repel each other and there is no tendency to flocculate. However, if the particles have low zeta potential values then there is no force to

prevent the particles coming together and flocculating. The Zetasizer Nano series performs size measurements using a process called Dynamic Light Scattering (DLS). Dynamic Light Scattering (also known as PCS - Photon Correlation Spectroscopy) measures Brownian motion and relates this to the size of the particles. It does this by illuminating the particles with a laser and analysing the intensity fluctuations in the scattered light.

In practice, particles suspended in a liquid are never stationary. The particles are constantly moving due to Brownian motion. Brownian motion is the movement of particles due to the random collision with the molecules of the liquid that surrounds the particle. An important feature of Brownian motion for DLS is that small particles move quickly and large particles move more slowly. The relationship between the size of a particle and its speed due to Brownian motion is defined in the Stokes-Einstein equation. As the particles are constantly in motion the speckle pattern will also appear to move. As the particles move around, the constructive and destructive phase addition of the scattered light will cause the intensity appears to fluctuate. The Zetasizer Nano system measures the rate of the intensity fluctuation and then uses this to calculate the size of the particles [10].

Experimental techniques

a) *Materials and preparation*

2 gm of PVA (Poly Vinyl Alcohol) from LOBA CHEMIE dissolved in 198 gm of distilled water, the solution is placed on magnetic stirrer for 12 hours at 75 °C, and the beaker is covered with aluminium foil to minimize the evaporation. This 200 gm solution is of concentration 1% by weight (1 wt.%). Other PVA solutions with concentrations of 1.5, 2, 3, 4, 5, 6 and 8 wt. % are prepared by the same method.

99 gm of each of the above solutions is loaded by 1 gm of copper (Cu) spherical nano particles of 500 nm diameter from Nano-structured & Amorphous Materials, Inc. (NanoAmor, USA). This concentration of Cu is kept constant with all PVA solutions, in order to minimize the effect of Cu concentration on the viscosity of the PVA solutions.

In contrast another set of solutions has been prepared with fixed PVA concentration (8 wt. %) and different Cu concentrations for calibration requirements.

For all Cu-PVA solutions, the Cu nano particles are dispersed for 2 hours by a local made sonicator as shown in figure 2, where a power speaker is excited by power signals from electronic function generator and power amplifier. The dispersion signal is adjusted at 500 Hz.



Figure (2): The used power sonicator for dispersing the Cu particles in PVA solution.

b) **Viscosity measurements**

The viscosity of samples were measured by a rotational digital viscometer model Myr version I, using spindle L2 at the rotation rate 200 rpm through the viscosity range of 150 mPa, at the room temperature 24°C and relative humidity of 75%.

c) **Transmission Electron Microscope (TEM)**

Transmission electron microscope is a fundamental technique for nanotechnology

studies. The prepared suspensions are examined using a JEOL JEM-2100 TEM, the microscope is located in Mansoura University – Egypt.

d) **Density measurements**

The density of solutions either Cu loaded or not loaded by Archimedes rule using 25 ml density bottle and 4 digits electronic balance of resolution 0.0001 gm.

e) **Particle size by Zetasizer**

The average size of particles that are suspended in fluids with different viscosities (stable suspensions) is measured by the Zetasizer model Malvern Zetasize Nano-zs90, from Malvern Co.

f) **Sedimentation measurements**

The classical size range for colloidal dispersions given above (1 nm – 1 mm) assumes dispersed species have a spherical shape. When other shapes are considered, particles with diameters of up to 2 mm can be well described as colloids. Practical suspensions usually have diameters greater than 0.2 mm and often contain particles that exceed the classical size range limits given above, sometimes to 50–100 mm in diameter. The principles of colloid science are still important to the behaviour of such larger particles. The particle sizes can also fall below the classical size limit given above. Nano-particle suspensions are increasingly being developed on the one hand. In some literature, the term “sol” is used to

distinguish suspensions in which the particles are of such very small sizes [11]. As mentioned above there is always a range of particle size defines the colloidal and suspension stability. In the current work a trial to shrink this range which may give more definite properties to the final suspension. This may be done by controlling the sedimentation process by quantitative characterization. This can be done by correlating the sedimentation rate to the main factors; viscosity of the continuous phase, particle size, density difference and particles concentration.

Other associated processes should be taken into account due to their effective contributions. The aggregation and coalescence are among of these processes. Aggregation is when any of Brownian motion, sedimentation, or stirring causes two or more dispersed species to clump together, possibly touching at some points, and with virtually no change in the total surface area. In aggregation the species retain their identity but lose their kinetic independence since the aggregate moves as a single unit.

To carry out this target a modified measuring system is setup and used. This used system is a modified time of flight system, where the time of transportation of a particle or group of similar particles is measured. The operating mechanism of the used system is based on the phenomenon of static

light scattering due to turbidity, the system is shown in figure 3.

A glass cuvette is inserted inside a thick wooden cap to preserve the temperature. The cuvette is filled with the PVA solution which is loaded with Cu particles (suspension under test). The suspension is illuminated by two point sources S_1 and S_2 separated by distance d where $d = 25$ mm. S_1 and S_2 are extra white LED sources excited by DC voltage of 3.5 volts. The transmitted light is received by two light detectors N_1 and N_2 which are LDR (light dependant resistance), each connected in series with a variable resistors R_1 and R_2 . Two storage digital voltmeters V_1 and V_2 are used to measure the voltage drop across R_1 and R_2 and save readings every selected period of time (1 sec, 1 min or other). Thus the change in volts can be recorded and saved for long range of time may exceed several days. Sources S_1 and S_2 as well as detectors N_1 and N_2 are fixed at ends of long channels of 4 mm diameter grooved in a wooden cap to ensure that detectors receive only the un-scattered light rays. Hence as scattering centres increased in the path of rays as the intensity of directly emerged rays is reduced, and accordingly, the recorded voltage is reduced too.

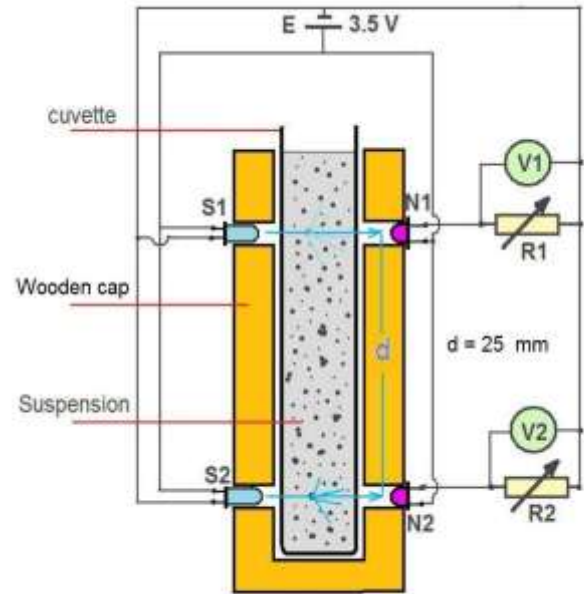


Figure (3): The modified time of flight system

This means that the voltage across R_1 and R_2 measure the transparency of the suspension, while the voltage across the detectors N_1 and N_2 measure the turbidity of the suspension. Then by using mixtures with different known particles concentration, one can calibrate values of recorded voltage to give directly the concentration of particles in the path of light rays. Plotting the particles concentration as function of time should help to calculate the sedimentation rate. It should be noted that either the rate of changing transparency or the rate of changing turbidity should equal the rate of sedimentation. Since the solid phase of the suspension consists of different particle sizes and each size is sediment at specific rate, then it is possible to determine the present number of different sizes by counting the different slopes that are appear in the voltage (V_{1-2})

– time graph. Referring to figure 4, it is clear that sensor at level S1 N1 is recording sedimentation of particles in region A, while sensor at the level S2 N2 is recording sedimentation of particles in regions A and B. Therefore the graph V_1 – time (t) should differ than that for V_2 – time (t). It is expected that the curve V_1 – t should reach stability before the curve V_2 – t, and the time difference between the two stabilities is the flight time of particles across distance d. And hence one can measure the velocity of motion of each group of particles having sizes in very narrow range.

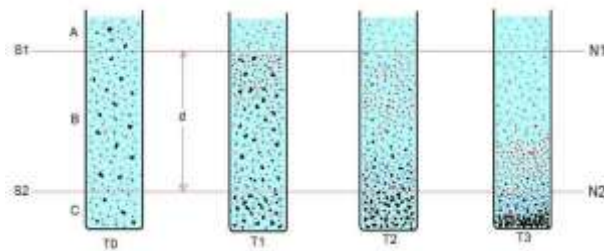


Figure (4): The progress of sedimentation of different particle size at different times as seen at two different levels.

The time of flight across distance d can be measured directly by re-connecting wires in figure 3 so that to use one voltmeter to read the difference $\Delta V = (V_1 - V_2)$. In this case the curve $\Delta V \gg t$ should show a peak value just when V_1 is reached its stability. The part of this curve after the peak represents the motion of particles within the region B (between the two levels whose separation is d), and hence

fluctuations in this part reflect nature of processes occurred during flying across distance d.

It is important to notice that the value of stable V_1 or V_2 is not equals their values for clear liquid of the continuous phase because some particles are suspended in the liquid. The shift from the recorded voltage of clear liquid may represent the concentration of suspended particles. The concentration and size of the suspended particles depends on the viscosity and density of the hosting solution. In order to verify this estimation, the system should be adjusted carefully before starting the measurements.

Initial adjustments of the system:

The adjustment of this sedimentation measuring system should follow the following steps:

- Insert the empty clean cuvette in the wooden cap.
- Adjust the variable resistors R_1 and R_2 until readings of V_1 and V_2 are equal.
- Remove the cuvette and fill it with the liquid of continuous phase only and then insert it in the wooden cap and record the readings V_1 and V_2 , they should show the same values. This value will refer to the zero particle concentration.
- Remove the cuvette and fill it with the suspension under examination.
- Close the cuvette, shake it well and insert it again in the wooden cap, then start

continuous recording of voltages automatically every one minute until steady values for both of V_1 and V_2 .

Results and Discussion

Dealing with nano-powders is a complicated issue and a very difficult one, mainly because of their toxicity and propensity to agglomeration as a result of their high surface area. However, particle dispersion in a liquid media has been found to be a promising approach to result in suspensions with a higher uniformity [12-13]. Shake well before use, it is a sentence usually seen on the drug bottles, now in the age of nano-technology this sentence should be absent. This sentence means that the bottles contain un-stable suspension, or fast settle fluid. As discussed before, the main factors affecting the sedimentation rate are particles sizes of the solid phase, viscosity of the continuous liquid phase, densities of phases and some processes due to surface reaction of particles with liquid like Zeta potential, Brownian diffusion and agglomeration. In this work the effect of some of these factors are measured and results are expressed in form of empirical relations in order to describe, numerically, the initial requirements to produce suspension of high stability. Copper particles (as high density material $\rho_p = 8.6 \text{ g/cm}^3$) is used as the solid phase while solution of polyvinyl alcohol (PVA)

dissolved in water represents the continuous liquid phase. This selection of the continuous phase enables one to change its viscosity easily by changing the concentration of dissolved polymer. Figure 5 shows the relation between viscosity and concentration of PVA.

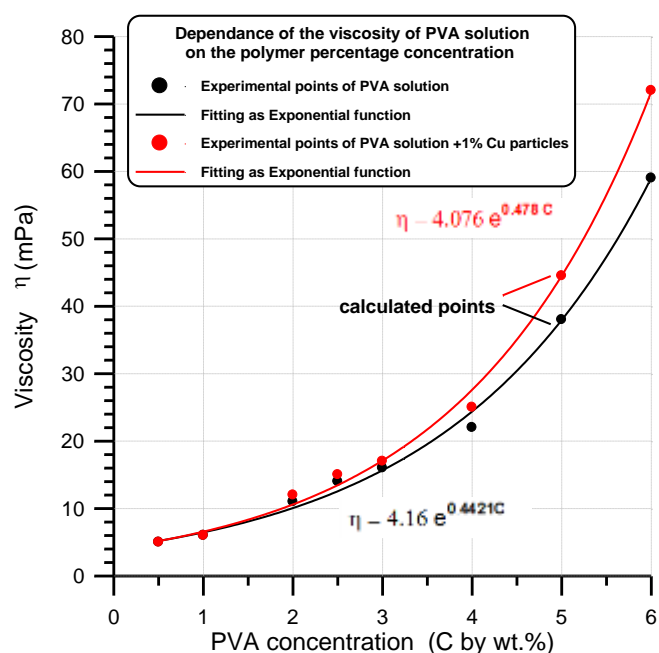


Figure (5): The variation of viscosity of selected continuous phase by changing the polymer concentration in the solution.

The graph shows that addition of solid particles increases viscosity of the liquid according the same mathematical trend as the particles free polymer solution. But it is important to refer to that the viscosity values η in all previous relations are for the particles free solutions. It is found that η is related to the PVA concentration C in wt. % empirically by the equation:

$$\eta = 4.16e^{0.442C} \dots\dots\dots(10)$$

(a point has been calculated using eq. 10 and inserted into the graph to examine the fitting accuracy of eq. 10). This equation helps to get a required viscosity by calculating the PVA concentration that corresponding to this viscosity. But the density of the PVA solution is changed by changing the PVA concentration. Figure 6 shows the dependence of liquid density ρ_1 on the solute concentration. Figure 6 shows that the liquid density is affected by the PVA concentration according to the empirical relation:

$$\rho_1 = 1.02C^{0.0056} \dots\dots\dots(11)$$

The graph shows also that adding solid Cu particles to the solution increases the overall density, but ρ_1 is usable value.

The effect of the continuous phase viscosity on the sedimentation rate is measured by the modified differential sedimentation system. Figure 7 shows the results obtained from this system.

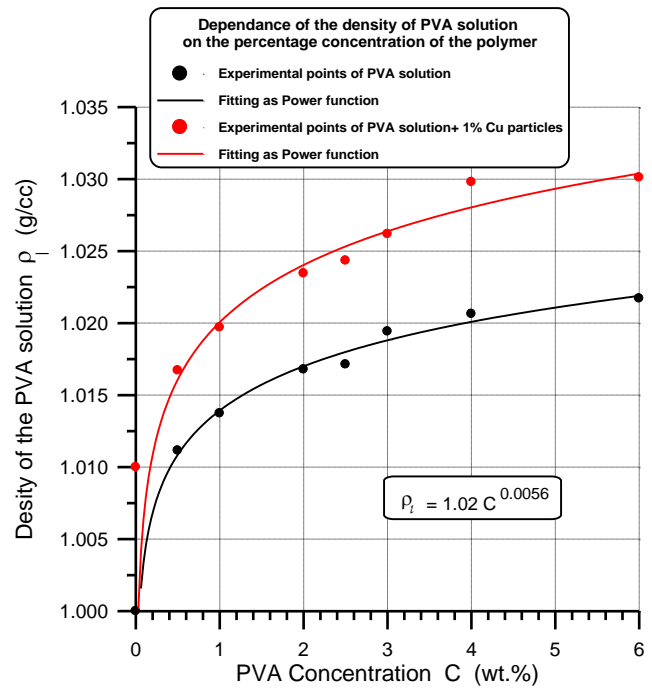


Figure (6): Dependence of the liquid density on the PVA concentration

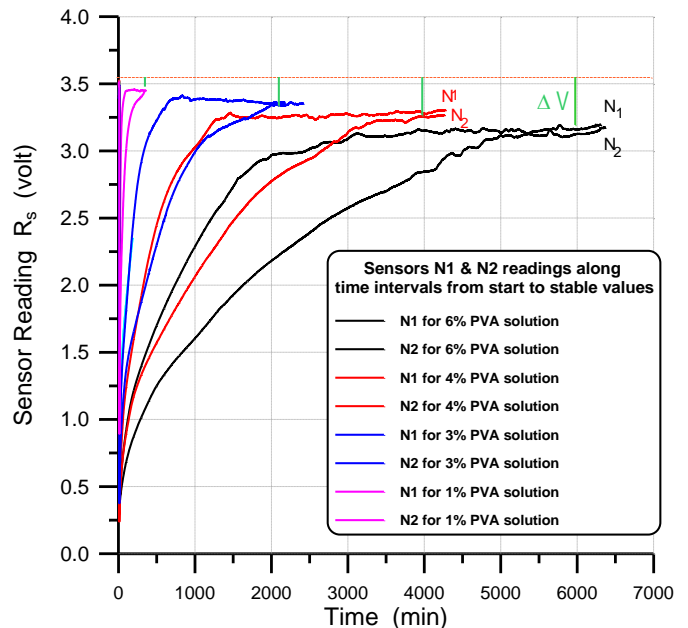


Figure (7): Sensors N1 and N2 readings as recorded by the modified sedimentation system for host liquids with different PVA concentrations.

Note that as the sensor reading increased as the fluid in the cuvette become clearer, i.e. the concentration of scattering centres (suspended particles) is lower. Hence the slope of curves at any point gives the rate of sedimentation at that point. Also one must note that readings of N1 refer to sedimentation of particles above the location of the sensor (region A, fig.4), while readings of sensor N2 refer to sedimentation of particles above the location of the sensor (regions A&B, fig. 4). Therefore readings of N1 reached stability before N2. Also, readings of N2 just after stability of N1 readings to the stability of N2 represent the time of flight of the present particles between N1 and N2 (distance between N1 and N2 is constant and equals 25 mm). So, dividing the distance $d=25$ mm by the time interval between the moments of stability of N1 and N2 gives directly the average speed of sedimentation. But it is difficult to consider the obtained speed represents the velocity of particles through the used viscose solution because of the following factors:

- Particles have different sizes (different weights and shapes) due to aggregation. This can be examined by the TEM.
- Near stability, the Brownian diffusion by the very tiny particles resists the motion of other greater particles. This is appearing clearly from the irregularities that are found in

readings of N2 at stability and near stability.

- It is hard to well determine the moment of stability from figure 7. This problem is dissolved by plotting the difference $N1-N2$ with time; at the moment of stability of N1 the curve should show a peak value, figure 8.

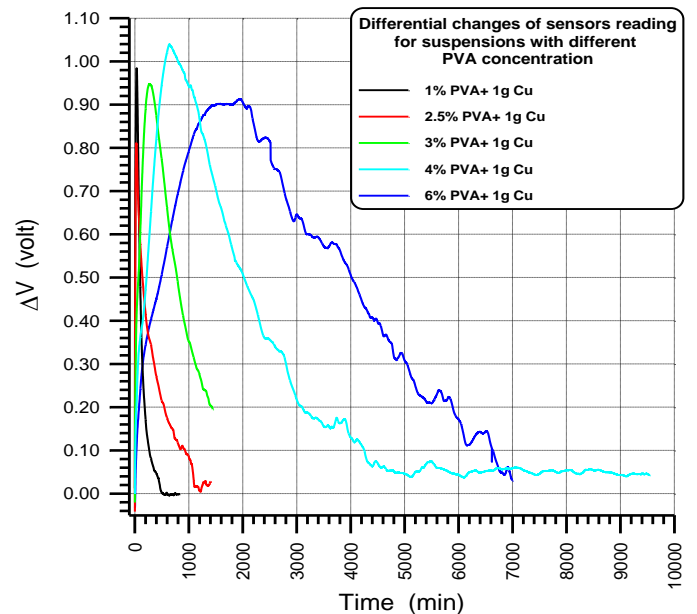


Figure (8): To well determination of the moment of stability of N1

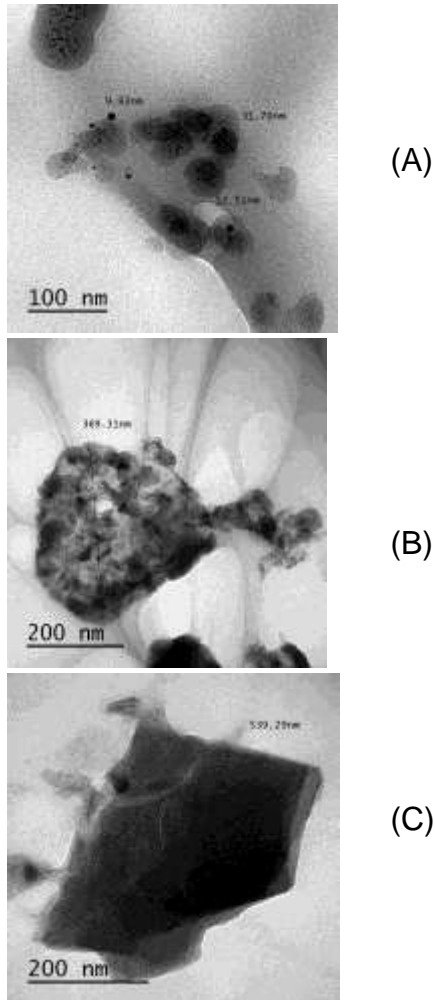


Figure (9): TEM images for the Cu particles suspended in PVA solution of concentrations (A) 1%, (B) 4% and (C) 6%.

Suspensions from 1 gram CU nano-powder in PVA solutions with different concentrations (1% to 6%) are left undisturbed for several weeks and then sample from each solution is drawn from the mid-point and examined by the TEM and Zetasizer to show the size and shapes of the suspended Cu particles. Figure 9 shows shapes and dimensions of the suspended Cu particles in the stable PVA solutions. It is noted that PVA solution of higher viscosity hangs

particles with greater dimensions and vice versa. During the TEM examinations, it is important to ensure that the particles shown are for the Cu particles and not for any contaminations. Electron diffraction by the observed particles is good method for the decision. Figure 10 shows the well known crystal structure of Cu samples.

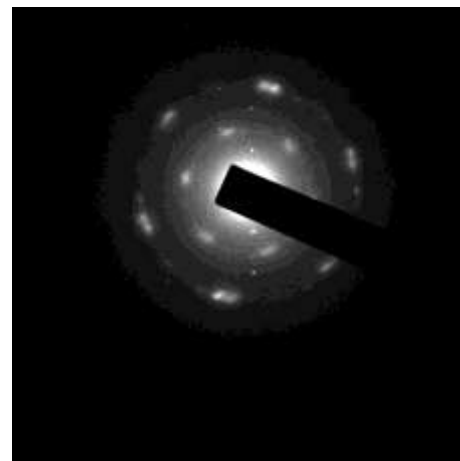


Figure (10): The crystal structure of Cu as seen by the electron diffraction technique that associated with the TEM.

Zetasizer can also be used to estimate the particles size in nanometers. The samples from mid-point from above mentioned stable suspensions are examined by the Zetasizer, figure 11 shows the relation between the particle's diameter as measured by Zetasizer as function of the PVA concentration of the host solution.

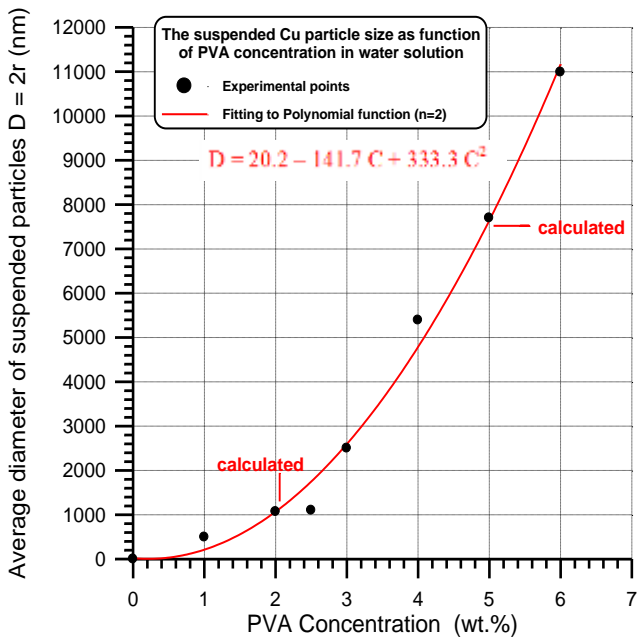


Figure (11): The average diameter of suspended Cu particles in PVA solutions with different concentrations.

The empirical relation represents the curve shown in fig. 11 is

$$D_p = 20.2 - 141.7C + 333.3C^2 \dots\dots\dots(12)$$

Two points on curve in fig. 11 are calculated and inserted on the curve to test the validity of equation 12. Diameters that are measured by Zetasizer are found much greater than those measured from the TEM. This is expected because in Zetasizer the technique considers the particle diameter is extended from the geometrical centre of the particle up to the end of the field of zeta potential including the thickness of the double layer of surface charge (see fig. 1). Equation 12 helps for preparing a stable suspension from Cu particles

in PVA solution. By introducing the particle size of the solid phase to be suspended in the continuous liquid phase into equation 12, one gets the concentration of PVA in the continuous solution that can hang the Cu particles for always. This equation is helpful only for suspensions from Cu and PVA solutions, but what about continuous phases rather than the PVA solutions?

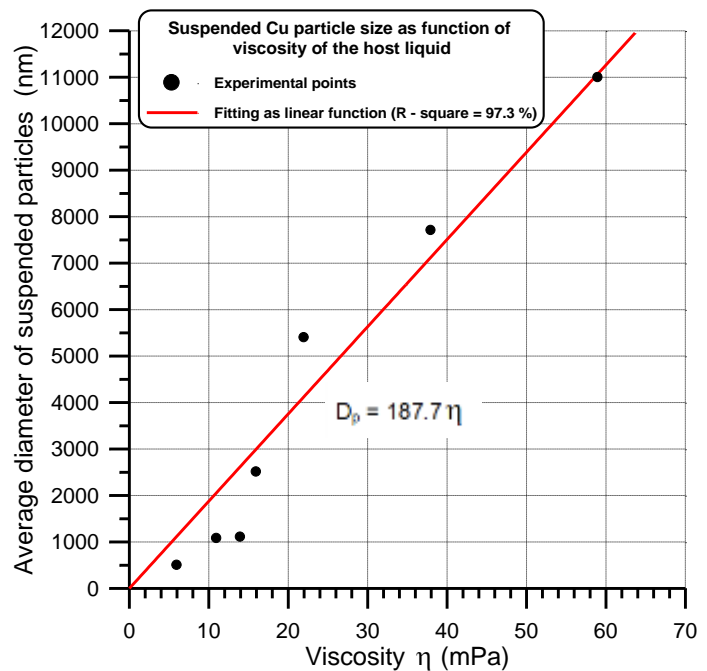


Figure (12): Average diameter of suspended Cu particles in solutions with different viscosities.

Figure 12 represents the relation between the average particle diameters that are suspended in different solutions and viscosities of these solutions. The empirical relation that is describing graph in fig. 12 is:

$$D_p = 187.7\eta \dots\dots\dots(13)$$

This equation confirms that fluids of higher viscosities can hang particles of larger sizes. Also this equation represents a relation between diameter of suspended particles and viscosity of the continuous liquid phase irrespective to the type of this liquid. Hence relation 13 may be applicable for any hosting fluid. But what about the density of this fluid since density of the fluid supports the bouncy force on the particles? Equation 14 is the empirical relation between D_p and ρ which describes the dependence of diameters of suspended particles on the density of hosting fluid.

$$D_p = 1.84\rho^{389.5}$$

..... (14)

Equations 13 and 14 may help to estimate the specifications of the continuous phase fluid that can suspend particles of a given diameter D_p , but how the modified sedimentation system can help to measure the concentration C_p of the suspended particles?

Recalling figure 7, the voltage shift ΔV from the starting voltage should be related to the concentration of the permanently suspended particles C_p . To construct such a relation the change in sensors readings should be calibrated to read the concentration of scattering centres (particles) in the path of light. This calibration is done by testing different fluids with different particles concentration ranging from 0.2 wt. % to 1 wt. % in PVA solution of 8% polymer

concentration. Figure 13 shows that the empirical calibration equation is:

$$C_p = 1.059 + 0.189R_s - 0.318R_s^2 + 0.051R_s^3$$

..... (15)

Introducing the value of R_s (sensor reading) at any moment into the above equation returns the corresponding value of C_p directly. Thus changes of R_s with time can be translated to changes of particles concentration as shown in figure 14.

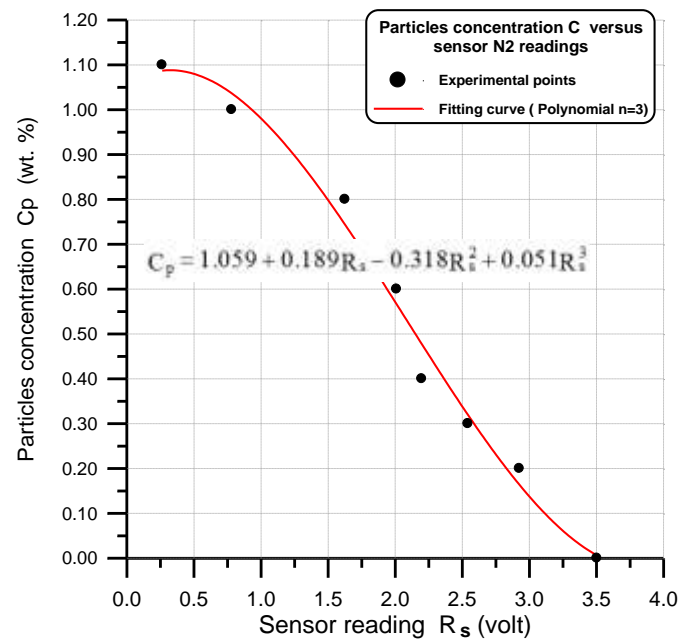


Figure (13): The calibration relation for sensor reading to read C_p

Referring to figure 14, it is easy to deduce the following meanings:

- The upper sensor reaches steady value sooner.
- The function $\Delta C_p(t)$ shows a peak value, where readings of the upper sensor $N1$ is

nearly fixed while readings of N2 is decreasing faster, then the curve inverts its direction.

- When $\Delta C_p = 0$, the whole solution system is reached stability, where both readings by N1= readings by N2 = particle concentration in the final stable suspension.

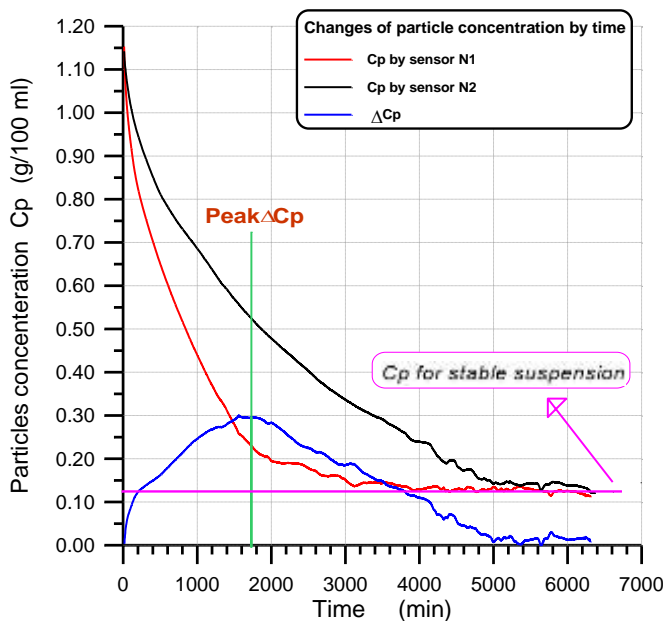


Figure (14): C_p as function of time and graphical determination of C_p for the stable suspension.

It is notable that changes of C_p are smooth at beginning of the sedimentation process where all big particles and particle aggregates are moved down with the fixed limiting velocity. Brownian movements cannot affect the regularity of big particles motion. Also it is notable that at the end of sedimentation process many irregularities in the recorded C_p values are observed well. Near the end of sedimentation, all big particles are settled at bottom of the cuvette and only the tiny particles

are either suspended or move with very slow speed in such a way that Brownian displacements are comparable with the displacements may a very slow particle is doing. Therefore, C_p may show infinitesimal changes up and down randomly.

From the above study one may conclude that determining the particle size is the first step for producing stable suspension. Engineered nano-particles are produced with a wide size spectrum, to increase the size resolution of a considered sample; close sizes should be separated by a way or another. This current work may throw light on a proposal for new technique that may be effective for the target of size separation of nano-particles. Equation 13 shows the linear dependence of particle diameter on the viscosity of the liquid phase, but the viscosity of any liquid is changeable by heating. Thus if a high viscosity liquid is loaded by particles of different sizes and then the mixture is poured in a double wall tube like that shown in figure 15, all particles still suspended in the high viscosity cold liquid.

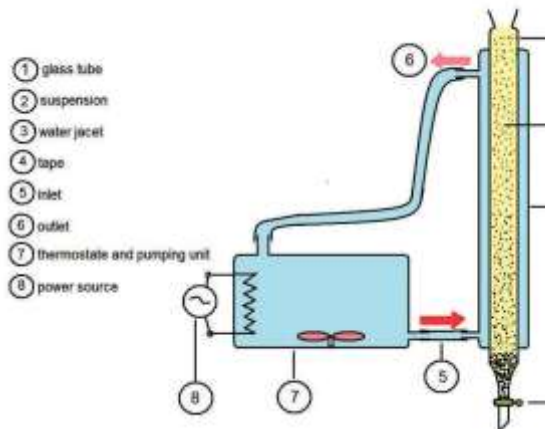


Figure (15): Schematic diagram for the proposed differential viscosity column.

By gradual increasing the temperature of circulating water, the liquid is heated and its viscosity is reduced as shown in figure 16.

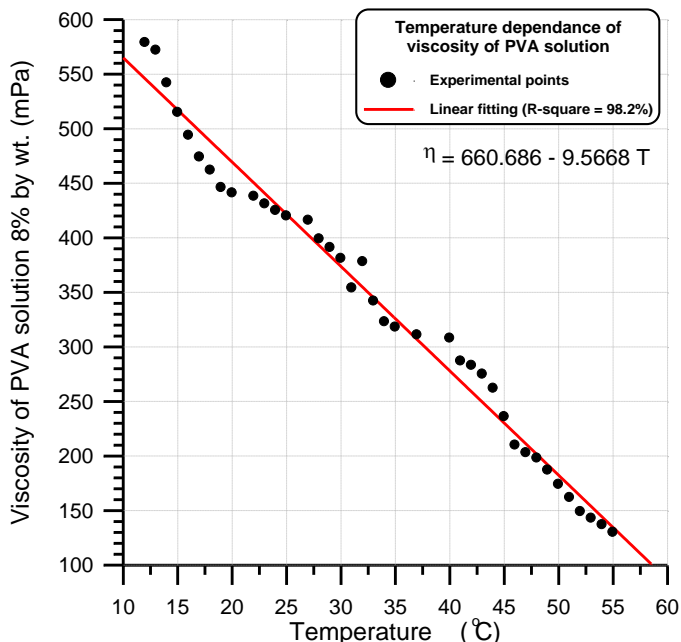


Figure (16): Linear dependence of 8% PVA solution on the temperature

It is notable from fig.16 that viscosity of PVA solution is changed linearly by heating according to the empirical relation:

$$\eta = 660.69 - 9.57T \quad \dots\dots\dots (16)$$

As the temperature of suspension increased, say, 5 °C the viscosity is lowered and great particles move to settle in the bottom of the column. After some time the tap is opened and these great size particle are drawn out. The temperature is now increased further 5°C, and then the next size (smaller than the first) will be released and collected on the bottom to be drawn from the tap. And so on each increase in temperature should reduce the viscosity then a specific size is released. Introducing equation 16 into equation 13 gives a direct empirical relation for particle diameter D_p as function of temperature.

Conclusion

Systems containing colloidal-sized particles, droplets, or bubbles are important because they feature prominently, in both desirable and undesirable contexts, in a wide variety of practical disciplines, products, and industrial processes. The problems associated with colloids are usually interdisciplinary in nature and a broad scientific base is required to understand them completely [11]. Stability of colloid or

suspension is important task in some fields of applications like pharmaceuticals, drug industry, paints,..etc. This study is focused on the modification of a method to measure the stability of suspensions, determination factors affecting the stability of suspensions and prematurely describing properties of components required to produce stable suspensions at once. Practically, it is found that particle size is the most important factor as well as the viscosity of the continuous phase fluid. The suggested modified differential sedimentation system could be calibrated to measure the concentration of suspended solid particles. The modified system showed satisfactorily accuracy enough to detect fluctuations in concentration of tiny suspended particles due to the Brownian diffusions. This study confirmed the fact that particles diameters measured by Zetasizer are much greater than those measured by the transmission electron microscope. Results obtained in this study guided authors to propose new technique for the nano-particles size separation which is deferential viscosity column based on the process of the differential sedimentation.

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