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Minimum agitation speed for solid suspension and mixing time in a torispherical -bottomed pharmaceutical stirred tank under different baffling conditions

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ABSTRACT

MINIMUM AGITATION SPEED FOR SOLID SUSPENSION AND MIXING TIME IN A TORISPHERICAL-BOTTOMED PHARMACEUTICAL STIRRED TANK UNDER DIFFERENT BAFFLING CONDITIONS

by
Dilanji Bhagya Wijayasekara

The minimum agitation speed, N_{js}, required to just suspend solid particles dispersed in water was experimentally determined in this work for a glass-lined type of mixing tank provided with a torispherical bottom and agitated with a retreat-blade impeller under different baffling configurations. N_{js} for the same tank but equipped with a different agitation system, namely an axial impeller, was also experimentally determined for the purpose of comparing of performances of the two systems. The effect of impeller off-bottom clearance and the vessel's liquid level on the minimum agitation speed were also experimentally studied. N_{js} was experimentally determined using Zwietering's method, requiring that the solids do not rest on the tank bottom for more than 1-2 seconds. The value of N_{js} was found to depend strongly on the type of baffling, and was highest in the unbaffled tank and with solid particles 150μm in size and lowest in the partially baffled system.

The mixing time, θ_{95}, required to disperse a tracer in the liquid to achieve a 95% homogeneity level was also experimentally determined in the same system for the partially baffled and fully baffled configurations. A colorimetric method coupled with image processing was used to determine the mixing time.

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APPROVAL PAGE

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To my Love

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CHAPTER 1

INTRODUCTION

1.1 Background

Liquid mixing and other mixing operations involving a dispersed phase, such as finely divided solids, in a liquid are a commons operation encountered in the chemical and pharmaceutical manufacturing industry. A number of such operations, especially in the pharmaceutical industry are carried out in glass-lined, stirred, torispherical-bottomed reactors. Glass lining provides corrosion resistance, ease of cleaning, and reduced product contamination. A typical glass-lined reactor is equipped with a single retreat-blade impeller close to the tank bottom and a single baffle. The reasons for this are mostly practical. The rounded shape of this type of impeller eases glass lining, and, in addition, the customarily torispherical shape of the bottom of industrial vessels allows this impeller to be placed very close to the bottom, making this impeller/tank configuration more efficient for suspending heavy dispersions (Campolo and Soldati, 2002).

In fact, solid suspension and dispersion in a liquid is an important operation carried out in such mixing systems. The primary objective of solid–liquid mixing is to create and maintain a solid-liquid slurry, and to promote and enhance the rate of mass transfer between the solid and liquid phases. Such processes are typically carried out in mechanically agitated vessels and reactors. In agitated vessels, the degree of solids suspension is generally classified into three levels: on-bottom motion, complete off-bottom suspension, and uniform suspension (Paul et al., 2004). For many applications, it is often important just to provide enough agitation to completely suspend the solids off the tank bottom. Below this off-bottom particle suspension state, the total solid-liquid interfacial surface area is not completely or efficiently utilized. Therefore, it is important to be able to determine the impeller agitation speed, N_{js}, at which the just suspended state is achieved by the particles (Armenante and Uehara-Nagamine 1998). Although N_{js} has been obtained for a number of mixing systems, very little information is available in the literature for the solid suspensions in the system of interest here, i.e., a partially baffled, torispherical-bottom, mixing vessel provided with a retreat-blade impeller. Therefore, this study was one of the main components of this work.

In addition, another processing aspect of the mixing system described above was investigated here, namely, mixing time, which is one of the most important factors associated with the mixing performance of an agitation system. Mixing time (also known as blend time) is defined as the time required for a tracer initially added to the system to reach a predefined degree of homogeneity within the system (Paul et al., 2004). The conductivity method and the colorimetric method are two of the proposed methods to determine mixing time, both qualitatively and quantitatively. The conductivity method, one of the quantitative methods, can measure the mixing time by tracing the concentration of an electrolyte added to the liquid in the tank .The colorimetric method, which can be used for both qualitative and quantitative measurements, is an alternative method to determine mixing time non-intrusively. (Nancharos, 2009) The colorimetric method is typically used to measure the approximate mixing time by visual inspection at a definite monitoring point. Currently, image processing of digitized images of the mixing system when a color tracer is added can be used, in combination with imaging processing software, to quantitatively detect a color change at a particular location in the vessel very precisely. Applying image processing to the colorimetric method, one can determine the mixing time at each particular point by monitoring color evolution after the mixing process is initiated. Cabaret et al. have studied this approach and concluded that this method is highly reproducible and can identify unmixed zones. Therefore, in this work the mixing time required to achieve a predefined level of homogenization in the above-mentioned mixing system was experimentally obtained.

1.2 Objectives of This Work

The primary objective of this work was to experimentally determine the minimum agitation speed, N_{js}, required to just suspend the solid particles in a glass-lined type of tank with a torispherical bottom agitated with a retreat-blade impeller for different baffling configurations. N_{js} for the same tank but with a different agitation system, namely an axial impeller, was also experimentally determined for the purpose of comparing of performances of two systems. The effect of impeller off-bottom clearance and the vessels liquid level on the minimum agitation speed are also experimentally studied.

A second objective of this work was to experimentally determine the mixing time, θ_{95}, of the system under investigation for partially baffled and fully baffled configurations using a color metric method. This portion of the work was an extension of the work carried out by Nancharos Chomcharn, a former student also working in this lab, who completed his thesis in May 2009.

CHAPTER 2

EXPERIMENTAL APPARATUS AND METHODS

The equipment and methods described in this section were used to obtain experimental results for the minimum agitation speed for complete solid suspension, N_{js}, as well as for determining the mixing time in a torispherical-bottom mixing vessel provided with a retreat-blade impeller under different baffling configurations.

2.1 Mixing Tank

The mixing tank used in this work was an open cylindrical vessel provided with a torispherical-bottom. This tank was commissioned, and paid for, by Eli Lilly (thanks to Dr. Billy Allen, Eli Lilly, Indianapolis, IN). Fabrication of the tank was completed by the BHR Group in the UK (with Dr. David Brown's assistance) using a thin (0.5 mm) fluorinated ethylene propylene co-polymer (FEP) semi-rigid film. This material has a refractive index of 1.338, i.e., very similar to that of water (1.333), in order to minimize any curvature effect during the image processing steps. The internal tank diameter, T, was 450 mm and its total height was 540 mm. The height of the dish bottom section was 155 mm and the height of the cylindrical section was 385 mm. The tank had a rigid collar and lip at its top, which allowed the tank to be suspended in a larger "host" square tank, as shown in Figure 2.1. The square tank was made of Plexiglas and was used to minimize the optical distortion introduced by the curvature of the cylindrical mixing tank.

During a typical experiment, the mixing tank was placed in the host square tank, and both tanks were filled simultaneously with tap water to eliminate the differential pressure across the thin wall of the mixing tank that could have ruptured it (the reduction of differential pressure across the tank wall is discussed in Section 2.4 in greater detail). Three different water levels were used in the mixing tank depending on the experiment. Specifically, the mixing tank was filled with water so that the liquid height-to-tank diameter ratio, *H/T*, was equal to 0.5, 0.67 or 1, corresponding to volumes equal to 25.71 L, 36.64 L and 60.49 L, respectively.

A mirror was set up under the tanks at a 45°-degree angle with the horizontal plane so that the bottom of the mixing tank could be clearly seen. The set up was illuminated with a 100 W lamp.

2.2 Agitation System

Two types of impellers were used as agitators in this work, i.e., a three-blade retreat-blade curved impeller (RCI), and a four-blade, 45° degree pitched-blade turbine (PBT). The RCI was a scaled down version of a commercial, industrial De Dietrich impeller. It was a kind donation of Dr. San Kiang of Bristol-Myers Squibb, New Brunswick, NJ.

The RCI's dimensions were measured with a caliper and were as follow: impeller diameter, *D* = 219.1 mm; radius of curvature of the blades = 92.08 mm; height of the blade = 25.4 mm; and thickness of the blade = 12.7 mm. The impeller diameter-to-tank diameter ratio, *D/T*, was 0.489 for this impeller. For the PBT, *D/T* was 0.445. The PBT dimensions were measured with a caliper and were as follow: impeller diameter, *D*=190.6 mm; width of the blades=75.7 mm; height of the blades =33.3 mm; and thickness of the blade = 3mm.

Each impeller was mounted on a shaft having a diameter of 12.52 mm and located centrally inside the mixing tank, and was rotated by a 0.25 HP motor (Chemglass, Model

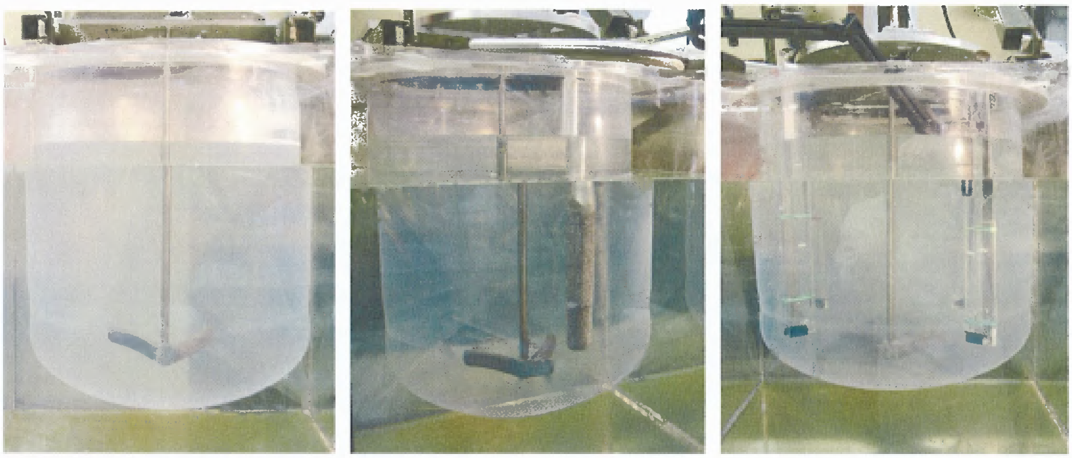
CG-2033-11) controlled by an external controller (Chemglass, Model CG-2033-31). The agitation system was fixed and mounted on a fixed rig. The tank system was mounted on a heavy-duty lift that could be moved both vertically and horizontally.

The agitation speed was measured with a digital tachometer (Model no HT-4100).

2.3 Tank Baffling

The tank was operated under three baffling configurations, i.e., completely unbaffled, partially baffled, or fully baffled. A single beaver-tail baffle, shown in Figure 2.1(b), was used in the partially baffled tank. The dimensions of the baffle were as follows: diameter of the top section, 15.24 mm; length of the top section, 70.64 mm; diameter of the middle section, 22.23 mm; length of the middle section, 199.7 mm; diameter of the bottom section 20.07 mm; length of the bottom section 70.64 mm. The baffle clearance was 90.23 mm from the vessel bottom measured from the lowest point in the tank at the center and 21.38 mm from the top of the impeller to the baffle bottom. The submergence of the baffle was 360mm for the H/T=1 system, 210 mm, for H/T=2/3 system, and 135mm for the H/T=1/2 system respectively, measured from the bottom of the baffle to the free surface. The baffle was placed 70 mm from the wall.

For the fully baffled tank shown in Figure 2.1(c), the baffles were made of four vertical rectangular metal plates mounted from the top. An acrylic rectangular plate was attached to each metal plate in order to increase the width of the baffles, which extended fully along the cylindrical wall of the tank. The total width of each baffle was 45 mm and the length of the submerged part was 300 mm, measured from the liquid surface, for the *H/T*=1 system, 200 mm, for *H/T*=2/3 system, and 75mm for the *H/T*=1/2 system, respectively.



(a) (b) (c)

Figure 2.1 Mixing system used in this work:
(a) Unbaffled tank
(b) Partially baffled tank with beavertail baffle
(c) Fully baffled tank

2.4 Materials

2.4.1 Materials used in the Determination of the Minimum Agitation Speed N_{js}

Tap water at room temperature was always used as the liquid phase in both the mixing tank and the "host" square tank. Superbrite glass particles from Minnesota Mining and Manufacturing Company, having average diameters of 60mm and 150 mm and a density of 2500 kg/m^3 were used as the dispersed phase.

The glass particles used in the solid suspension experiments were processed as follows prior to the experiments. The particles were first sieved using a standard lab sieve stack with screens. Five screens of US standard mesh size 40, 60, 80, 100 and 230 were used. Only about 30 g of particles were placed in the sieve stack at any time and the sieve stack was shaken for five minutes by hand. The particles retained on the size 100 mesh screen (with an average diameter size of 150μm) and size 230 mesh screen (with an average diameter size of 60 μm) were collected separately and labeled accordingly. The procedure was repeated for the total mass of particles to be eventually used (302 g). An electronic scale was used to measure the particle mass. In addition, solid fines were removed from the glass particles by preliminarily placing the particles in the mixing tank with water, mixing them thoroughly, and allowing them to decant for a time appropriate to their desired size. The fines, which settled more slowly and remained in the supernatant, were discarded with the supernatant. The procedure was repeated two times, as described in the experimental section below. Particle samples were placed under a microscope (Microstar, American Optical Model 110) and measured against a micrometric scale as qualitative check, and confirmed that average particle sizes was 60μm and 150μm, respectively. The solid concentration used in all experiments was 0.5% (W/W). Table 2.1 gives the amounts and volumes of solids used for each *H/T* ratios.

Table 2.1 Solids Amounts and Volumes Used in Experiments with Different *H/T* Ratios

H/T	Solid Amounts Corresponding to 0.5% (W/W) (in g)	Volume Percentage
1	302	0.2
2/3	183	0.2
1/2	123	0.2

2.4.2 Materials Used in the Determination of Mixing Time

Distilled water at room temperature was used in mixing tank and tap water at room temperature was used in host tank. NaOH solutions (0.85% w/v) and HCL solutions

(0.85% w/v) were used to change the pH of the distilled water and phenolphthalein was used as an indicator.

2.5 Experimental Procedure for the Determination of Minimum Agitation Speed

The mixing tank was placed in the host tank on a heavy-duty lift platform and this assembly was positioned under the impeller so that the impeller was perfectly centered in the mixing tank. The appropriate baffling system was set up. The mixing tank and the square tank were then filled with tap water (up to 450 mm for H/T=1 system, 300 mm for H/T=2/3 system and 225 mm for H/T= 1/2). The 'host' square tank was simultaneously filled up to the same level to minimize pressure differentials across the mixing tank. The mirror was adjusted so that the bottom of the mixing tank could be clearly seen, and the set up was illuminated. The impeller off-bottom clearance, C_B, was set to the required value by moving the whole tank assembly vertically using a pedal for the lift. C_B was measured from the bottom of the impeller to the bottom of the tank along the centerline of the system. Next, the glass particles were added and were allowed to settle in the mixing tank.

It was noticed, that the suspension contained some fines, which clouded the suspension in preliminary test runs and required a long time (some 10-15 minutes) to settle when the agitation was stopped. To remove the fines, the time for the particles to settle starting from the highest liquid level to the tank bottom was calculated and found to be 52 s for the 150 μm particles, and 150 s for the 60μm particles. The settling times were estimated using Stoke's law assuming Reynolds number (Re) was less than one and drag coefficient as 24/Re. Then, once the particles were all suspended, agitation was stopped, and settling was allowed to take place for only 60 s for the 150μm particles and 180 s for the 60μm particles. At the end of this settling time the supernatant, containing the fines, was removed and it was replaced with fresh water. The procedure was repeated twice. No fines were observed in the subsequent experiments.

A typical experimental run consisted of starting the agitation at low impeller speed and gradually increasing the speed in 5 rpm increments. The movement of the particles near the bottom of the tank as well as the flow pattern throughout the tank was carefully observed at each agitation speed. Solids suspension was observed visually via the mirror set up, and the value of the agitation speed for complete off bottom suspension, N_{js}, was obtained and recorded. The criterion used here to visually determine N_{js} was that defined by Zwietering (1958), as the speed at which no particles were visually observed to be at rest on the tank bottom for more than one or two seconds. This procedure was followed to obtain N_{js} in all experiments. This procedure could only be applied through experience from repetitive experiments in order to acquire a consistent data. In addition, a digital video camera (Canon VIXIA HF 200 HDMS) focused on the mixing vessel's bottom during selected experiment was also used create a permanent record of the observations.

However, a problem was detected when the agitation with the RCI was increased above 130 rpm and the system was unbaffled or partially baffled, in that a deformation of the shape of the bottom of the relatively flexible mixing tank was observed. At higher agitation speeds, the partial or no baffling effect produced a high tangential velocity of the fluid near the tank bottom, which resulted in the formation of a vortex. This, in turn, generated a low pressure zone near the tank bottom. Since the outside pressure head in the square tank was constant, this differential pressure across the thin mixing tank wall produced the deformation inwards and upwards of the tank bottom. The tank bottom was the first to deform since it was the location that experienced the maximum pressure difference. This effect resulted in two major issues. First, the mixing tank could have ruptured because of its very thin wall. Second, the change in the shape of the tank bottom would change the tank geometric configuration, thus changing the hydrodynamics of the system and interfering with the procedure used here to determine N_{js}. To eliminate the problem, the water pressure outside the mixing tank bottom, i.e., the pressure in the square tank was appropriately lowered by decreasing the water level in the square tank. This approach solved this issue. Decreasing the water level in the square tank was done simultaneously with incrementing the system agitation. It was important to restore the water level in the square tank as the agitation speed was lowered since the pressure of the mixing tank was inversely proportional to the agitation speed.

N_{js} was experimentally obtained for an impeller clearance off the tank bottom, C_B, of 40 mm in all the systems studied here. This configuration was used in previous work by this group and was based on the design of a scale-down impeller manufactured by the De Dietrich company, a leading manufacturer of glass-lined equipment and accessories for the pharmaceutical and chemical industries (specifications were kindly provided by Eric Momsen, Process Engineer, for De Dietrich Company, Union, NJ). In addition, experiments were conducted for different values of following operating variables:

- Baffling configuration: (unbaffled, partially baffled, and fully baffled systems)
- Impeller off–bottom clearance (4, 6, 9, 12 and 15 cm)
- H/T ratio (0.5, 0.67, and 1)

- Impeller type (RCI and PBT)
- Solids particle size (60 μm and 150 μm).
- Identical experiments at different times were also conducted to determine the experimental reproducibility. The reproducibility of triplicate experiments was typically found to be ±3%.

2.6 Mixing Time Determination Using Colorimetric Measurements and Image Processing

The mixing time studies were done for *H/T*=1 system with RCI impeller off bottom clearance of 40 mm. Partially baffled and fully baffled systems were studied. Mixing time experiments were conducted using a colorimetric technique based on the change in color of an indicator during an acid-base reaction. Phenolphthalein, which is pink when pH > 10 (base color) and colorless when pH < 8 (acid color), was used as the indicator. The mixing tank and host tanks were set up according to desired configurations and filled with water as mentioned in section 2.4.2. Next, NaOH solution was added to the distilled water in the tank to make its concentration equal to 10M (pH ≅ 11). The presence of phenolphthalein resulted in an initial pink solution. A 10-mL solution of sodium chloride (0.85% w/v) in a syringe provided with a cannula (ID: 1mm) was rapidly injected some 5 cm below the liquid surface inside the vessel, adjacent to the shaft. Each experiment was filmed with a digital video camera (Digital Handycam DCR-TRV740 NTSC, Sony) capturing the color change from pink to colorless at a rate of 30 frames/s. To obtain a homogenous illumination, a white sheet of paper was placed behind the host tank as a light diffuser. The digitized images in .wmp format were analyzed with the MATLAB application software in order to extract the red, green, and blue (RGB) components of the light intensity at selected, fixed, "sampling" locations on each image. Twenty such locations were selected, as shown in Figure 2.2. For each sampling point, the intensity of the green color component was extracted from each image and plotted as a function of time for a given neutralization experiment (data processing and analysis is described below). Each experiment was conducted in triplicate and the resulting values were averaged.

In order to minimize the use of distilled water (60 L per batch), at the end of each neutralization experiment, the batch was not discarded. Instead, the batch was neutralized with a NaOH solution until the pH was 11, as measured with a pH meter (Orion model 410A).

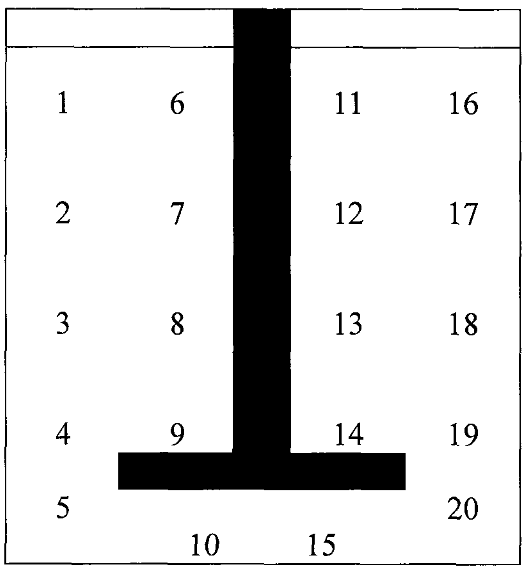


Figure 2.2 Locations of 20 sampling points for mixing time determination analyzed by the colorimetric method.

CHAPTER 3

THEORETICAL BACKGROUND AND DATA ANALYSIS

3.1 Equations for Minimum Agitation Speed for Solid Suspension

There have been many experimental studies and theoretical analyses on this topic, starting with the pioneering work of Zwietering (1958), which is still one of most cited. Zwietering derived the following correlation from dimensional analysis and estimated the exponents by fitting the equation to data for the just suspended solid impeller speed, *Njs* (Paul et al., 2004).

$$N_{js} = s \cdot \frac{v^{0.1} d_p^{0.2} \left(\frac{g \Delta\rho}{\rho_L} \right)^{0.45} X^{0.13}}{D^{0.85}} \qquad (3.1)$$

Several other studies (Zolfagharian, 1990; Choudhury et al., 1995; Choudhury, 1997) indicate that the effect of particle diameter is not as simple as the Zwietering correlation suggests, particularly at solid loading less than about 5 wt%. The exponent reported by Zwietering appears to be an average value for d_p between 0.20 mm and 1 mm. For particles greater than about 1 mm in diameter, N_{js} appears to be unaffected by the particle size. Choudhury reported this critical particle in terms of d_p/D at a value of about 0.01. On the other hand, for particles smaller than 0.20 mm, the average value of the exponent was about 0.5 (Paul et al., 2004).

In this study particles were smaller than 0.200mm and the solid loading was less than 5 wt%. Therefore, the modified expression for *Njs* was also used here:

$$N_{js} = s' \cdot \frac{\nu^{0.1} d_p^{0.5} \left(\frac{g \, \Delta\rho}{\rho_L} \right)^{0.45} X^{0.13}}{D^{0.85}} \qquad (3.2)$$

In this study, the size of the impeller, the characteristics of the liquid and the solids in a given system, and the solids fraction X were not changed. Therefore, the fractional terms in the above equations are constant for any given system. Table 3.1 lists these terms, which were calculated using the above relationships and the values of the operating and physical parameters used here. Using these parameters and by fitting the experimental N_{js} data to equations 3.1 and 3.2, Zwietering parameter s and s' could be calculated for all the systems used here by fitting the experimental data for N_{js} to the equations above.

Table 3.1 List of Fractional Terms in the Zwietering Equation (Equation 3.1) and the Modified Zwietering Equation (Equation 3.2) for Each System Studied Here

System	$\frac{\nu^{0.1} d_p^{0.2} \left(\frac{g \, \Delta\rho}{\rho_L} \right)^{0.45} X^{0.13}}{D^{0.85}}$	$\frac{\nu^{0.1} d_p^{0.5} \left(\frac{g \, \Delta\rho}{\rho_L} \right)^{0.45} X^{0.13}}{D^{0.85}}$
RCI with 150 μm particles	0.485	0.034
RCI with 60 μm particles	0.404	0.022
PBT with 150 μm particles	0.531	0.038

In the literature, the effect of the impeller off bottom clearance on N_{js} is typically presented by plotting N_{js} vs. the C_B/D ratio or the C_B/T ratio. In this work since C_B/D and H/T both are variables N_{js} was plotted using H/T as a parameter and as independent value the impeller non-dimensional submergence S_B, where this variable is defined as:

$$S_B = \frac{H - C_B}{D} \tag{3.3}$$

Hence, the data of this work were presented by plotting Njs vs. S_B for the purpose of comparing the performance of different baffling systems and the two impellers.

3.2 Data Processing and Analysis for the Determination of Mixing Time Using the Colorimetric Method

The raw data captured in .dv format by the digital video camera was converted to windows media video (.wmv) format in order to be compatible with the data processing software (MATLAB). The software used for data conversion was the freeware data converter "clone2go". Data conversion rate was 30 frames per second. Once the converted data were uploaded to MATLAB and processed as describe in Section 2.6, data arrays corresponding to the green light component (GLC) of the images at the respective sampling points were obtained as a function of time. From the plot of the GLC values vs. time, the asymptotic value of GLC was obtained and was used for normalizing the GLC data for that particular sampling location. Then, for each sampling location, the normalized GLC values were plotted against time. The resulting plots typically had a high noise level. Therefore, noise filtering was implemented using the "smoothing spline" algorithm in MATLAB. The use of this algorithm required the selection of a smoothing parameter, which was selected here in such a way so that the oscillations of the asymptotic "tail" of the normalized GLC curve (when the value of the GLC should be theoretically constant) were within ±5% of the average asymptotic value. The 95% mixing time, θ_{95}, at that particular sampling location, was selected as the first time at which the GLC curve entered a pre-defined range (i.e., 0.95–1.05, corresponding to reaching a 95% homogeneity level) and remained within this range. Error curves (±5%) were plotted in the same graph using MATLAB and the time range for 95% mixing of that location was obtained by taking the difference of values of x-axis corresponding to the points of error curves that meets the 0.95 boundary curve.

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Results of Solid Suspension Experiments

A total of 137 solid-liquid experiments were conducted, of which 20 were replicate experiments. As already mentioned, the typical reproducibility of triplicate experiments was ±3%. Detailed results are presented below.

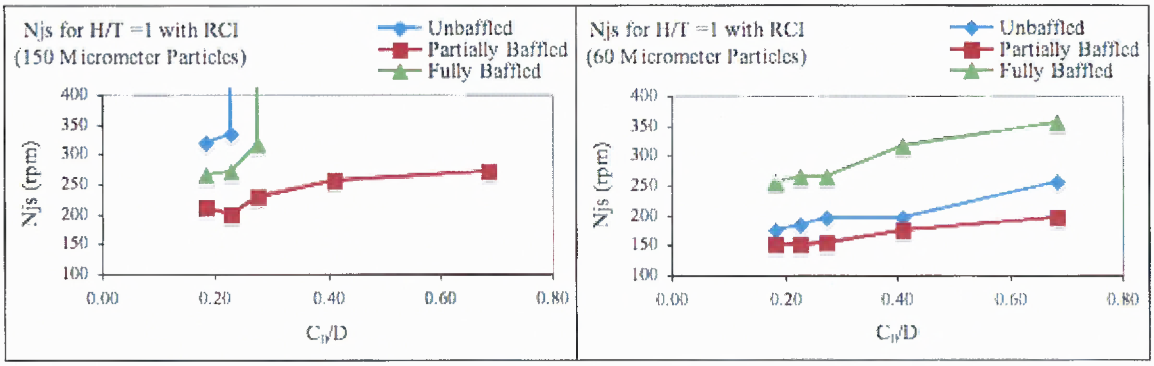
4.1.1 Effect of the Impeller Clearance Ratio C_B/D on the Minimum Agitation Speed, N_{js}, for Solid Suspension Generated by the RCI

The values of N_{js} were experimentally obtained for different C_B/D ratios for a standard system with the retreat blade impeller, H/T=1, and for each of the three baffling configurations examined here, i.e., the unbaffled, partially baffled, and fully baffled tanks. The results obtained using the 150μm solid particles and the 60μm solid particles as the dispersed phase are shown in Figures 4.1(a) and 4.1(b) respectively. Figure 4.1(a) shows that the larger particles could be suspended for any C_B/D ratios, at least in the range examined here, only when the partial baffling configuration was used. The lowest value of N_{js} for this baffling system (200 rpm) was obtained for C_B/D=0.23. This value is close to the value obtained for C_B/D=0.18 (210 rpm). It should be noticed that the standard configuration for this type of agitation system in the pharmaceutical industry is C_B/D=0.18 and partial baffling. This means that industrial systems are operated at a C_B/D value which is very close to the optimum value. When the system was fully baffled, suspension could be achieved but only at a higher agitation speed. However, the particles could not be suspended at all for C_B/D values larger than 0.27, as indicated in Figure

4.1(a) by the line pointing to a vertical asymptote for larger C_B/D values. In such a case, a small swirl was observed near the bottom of the vessel. Finally, when the system was completely unbaffled, the 150μm particles could only be suspended for low values of the C_B/D values. However for C_B/D>0.23 the particles could not be suspended.

The results for similar systems but when small particles (d_p=60μm) were used are somewhat different, as it can be seen in Figure 4.1(b). For the partially baffled system, N_{js} varies with C_B/D similarly to the larger particles, except for the obvious and predictable lower N_{js} value for the same configuration. However, this time the smaller particles could always be suspended, irrespective of the C_B/D value and the baffling configuration. Interestingly, the partially baffled system always produced the smallest N_{js} values. Furthermore, when the smaller particles were used, the fully baffled system required higher N_{js} values than the unbaffled system, although the reverse was true for the larger particles. This phenomenon was reproducible, and was observed in replicate experiments. The main reason for this lies in the generation of a vortex in the unbaffled system. For the larger particle case the vortex was not sufficiently strong to suspend the particles for larger C_B/D values, but it was for the smaller particles. In fact, in the latter case the vortex mechanism was so effective at particle suspension that it required lower agitation speeds to do so than the baffled system. This phenomenon was document by video recording.

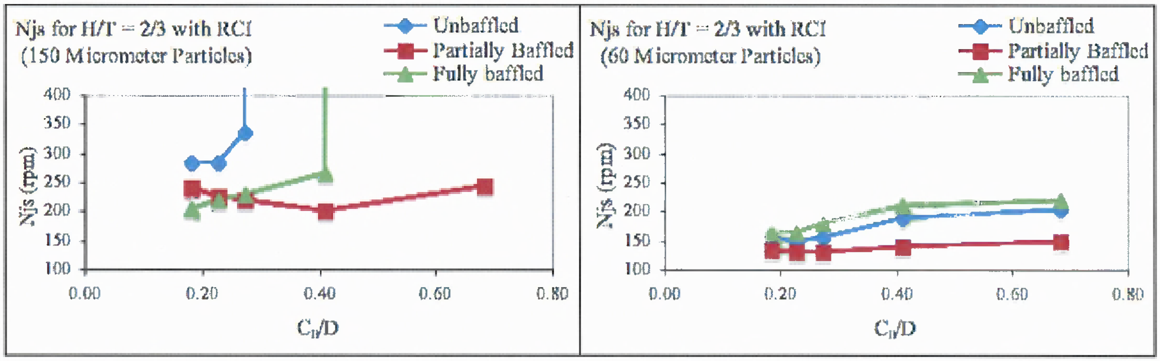
In summary, it appears that the partially baffled configuration is the most flexible and effective at particle suspension of those examined here and that in many cases this configuration is the only one that can produce solid suspension.



(a) (b)

Figure 4.1 Effect of the impeller clearance ratio C_B/D on the minimum agitation speed for solid suspension N_{js} for H/T=1 using an RCI (a) 150μm solid particles (b) 60μm solid particles.

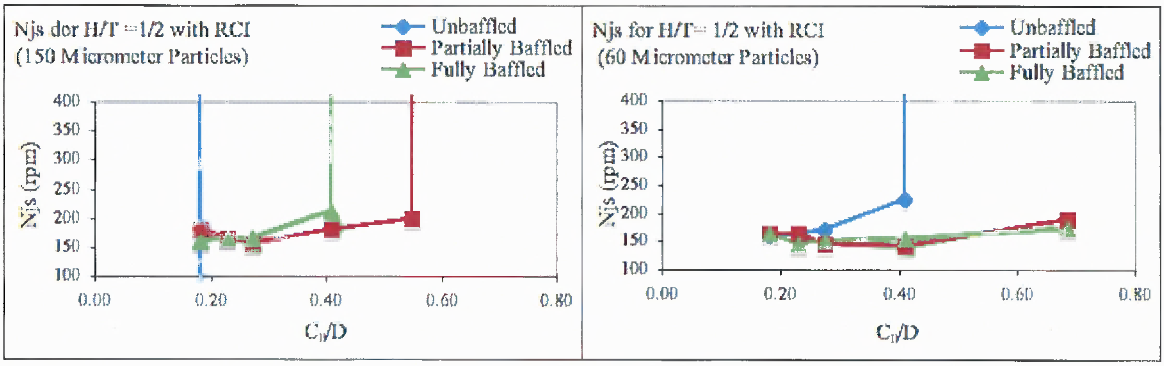
Figure 4.2 show the results for N_{js} vs. C_B/D obtained with the same 150μm solid particles and the 60 μm solid particles, but for the case in which the liquid height was reduced so that *H/T=2/3*. The picture that emerges is similar to that previously seen with *H/T=1* but with some additional complications. For case of the larger particles (Figure 4.2(a)), the partially baffled system is, in general, still the most effective at particle suspension even for *H/T*=2/3, since the other baffling systems are not capable of suspending the particle for the entire C_B/D range. However, the N_{js} curve does not decrease monotonically as in the *H/T*=1 case, but instead has a minimum for C_B/D=0.41. The curves for the other two baffling configurations show that suspension was still not always achievable for all C_B/D cases, but that the C_B/D values at which this occurred was higher than in the *H/T*=1 case. Figure 4.2(b) shows the corresponding plot for the smaller particles. As before, the particles could be suspended under all impeller configurations, although the corresponding N_{js} values to do so was lower than in the *H/T*=1 case, especially for the unbaffled system.



(a) (b)

Figure 4.2 Effect of the impeller clearance ratio C_B/D on the minimum agitation speed for solid suspension N_{js} for H/T=2/3 using an RCI a) For particles of 150μm b) For particles of 60μm.

Finally, the results for the *H/T*=1/2 case are presented in Figure 4.3. For the case of the 150μm particles in the unbaffled system (Figure 4.3(a)), the minimum agitation speed, N_{js}, either could not be achieved at all or could not be determined precisely. In any case, agitation levels above 350 rpm could not be tested because of the vibrations introduced in the system. However, even before reaching this point, it was observed that after a certain speed any further increase of agitation speed did not help suspend the particles off the tank bottom anyway. For lower C_B/D ratios such as 0.18, 0.23 and 0.27, full suspension could not be achieved at any speed. In fact, increasing the agitation speed only resulted in the formation and rapid growth of a vortex around the impeller shaft, drawing air into the system. For C_B/D ratios equal to 0.41 and 0.55 the solids at the center of the tank bottom became fully suspended at the speed in the range 280–310 rpm. However, the vortex did not allow a clear determination of N_{js}. For higher C_B/D ratios like 0.68, increasing the agitation speed resulted in splattering of the liquid, making minimal or no contribution to solid suspension. Again, solid suspension could not be achieved. When the system was partially baffled or fully baffled, the 150μm particles could be suspended, but only below a critical value for C_B/D, equal to 0.68 for the unbaffled case and to 0.41 for the baffled case. This was the only case in which suspension could not be achieved in the partially baffled system. Finally, Figure 4.3(b) shows the results for the 60μm case. For the partially baffled and fully baffled system the solids could be suspended for any C_B/D value (although with higher or lower N_{js} values with respect to the other H/T cases). However, for the unbaffled system case suspension was not achievable for C_B/D values larger than 0.41.



(a) (b)

Figure 4.3 Effect of the impeller clearance ratio C_B/D on the minimum agitation speed for solid suspension N_{js} for H/T=1/2 using an RCI a) For particles of 150μm b) For particles of 60μm.

4.1.2 Effect of the Liquid Level Ratio *H/T* on the Minimum Agitation Speed for Solid Suspension N_{js} Generated by the RCI

The effect of liquid level on N_{js} was studied by re-examining the same results reported above for the RCI and by re-plotting these data in terms of N_{js} vs. the liquid level ratio H/T, for the different baffling configurations. Since solid suspension was more easily achieved a low impeller clearances, the data are presented here for the lowest impeller clearance value only, i.e., C_B=40 mm, corresponding to C_B/D=0.18. The results are shown in Figure 4.4 for the two particle sizes considered here (150 μm and 60 μm).

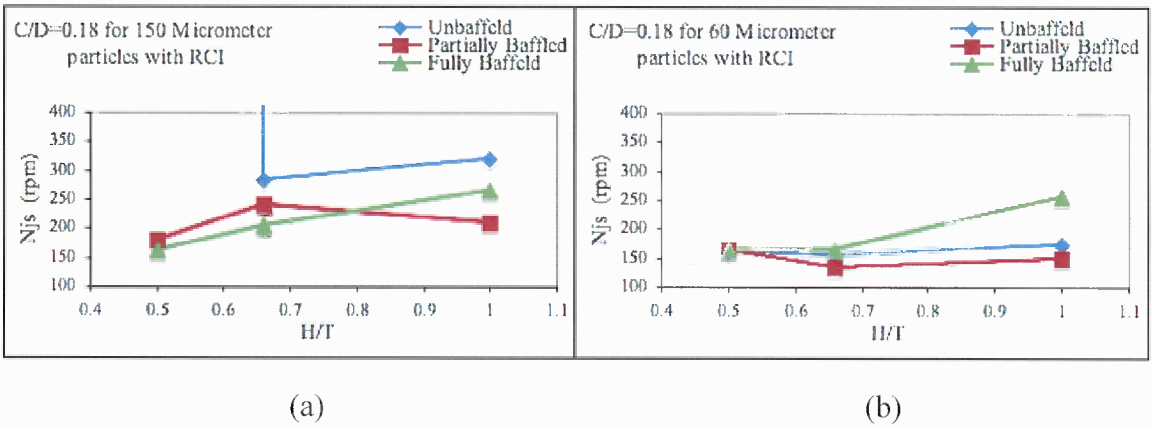


Figure 4.4 Effect of the *H/T* ratio on the minimum agitation speed for solid suspension N_{js} for *H/T*=1 using an RCI (impeller clearance ratio C_B/D=0.18) a) For particles of 150μm b) For particles of 60μm.

This figure shows that no single baffling configuration consistently produced the lowest value for N_{js}. For example, the partial baffling configuration typically resulted in the lowest agitation speed for particle suspension, but not with the larger particles at low *H/T* values. Similarly, the unbaffled tank was the worst in terms of N_{js} when the larger particles were used (suspension could not even be achieved for *H/T*=0.5), but was better than the fully baffled system when smaller particles were used. Clearly the interplay between the complex hydrodynamics for each system and especially near the tank bottom, and the force required to suspend particles of different sizes resulted in a variation of N_{js} which is difficult to predict.

Partially baffled system for RCI gives the lowest N_{js} compared to Fully and Unbaffled system. Both latter systems created a symmetric flow patterns around the Impeller vertical axis, while the single beavertail baffled lead to an asymmetric flow pattern, which appeared to be better at picking up solids from the centre of the vessels bottom. This result is in agreement with the result of Reily et al.,2007 has obtained for the conical bottom vessel agitated with RCI.

4.1.3 Comparison of the Effect of the Impeller Clearance Ratio C_B/D on the Minimum Agitation Speed for Solid Suspension N_{js} for Different Impellers

When the PBT was used as the impeller, the agitation speeds required to suspend the particles were always lower than the corresponding values for the RCI, as shown in Figure 4.5 for the 150μm particles. It should be noticed that particles suspension was always achieved with the PBT, irrespective of the baffling conditions. In addition, the value of N_{js} was relatively constant with C_B/D, especially for the fully baffled and partially baffled cases.

The corresponding data for the H/T=2/3 case is reported in Figure 4.6. This time particle suspension could not be attained when the impeller clearance ratio was larger than 0.3

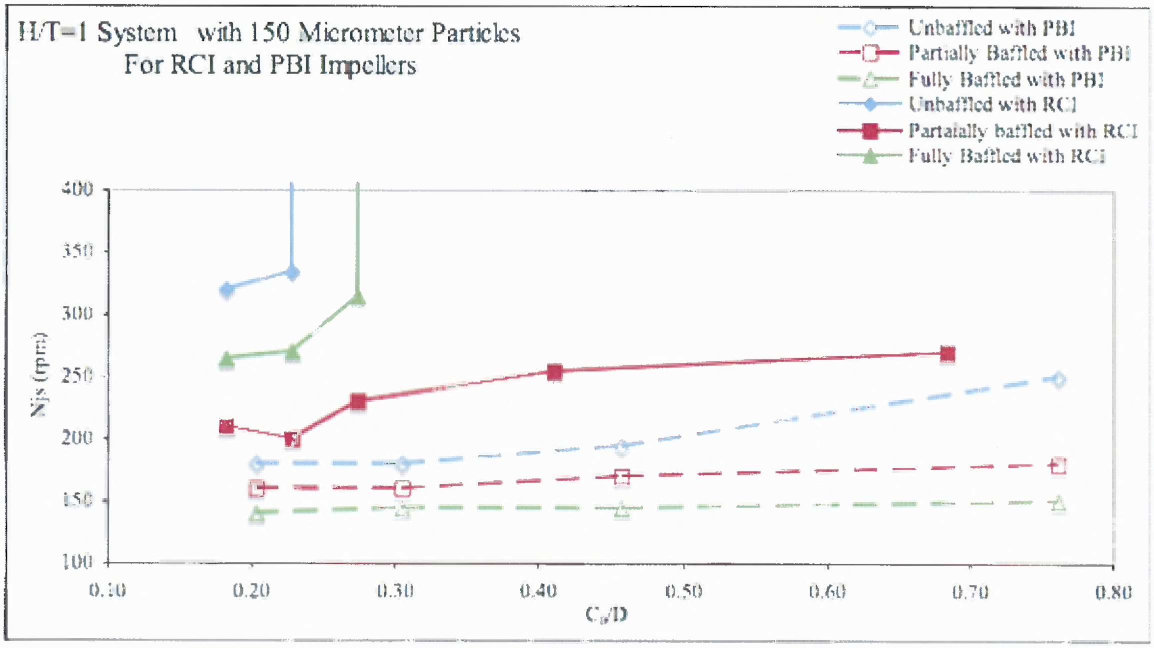


Figure 4.5 Effect of the impeller clearance ratio C_B/D on the minimum agitation speed for solid suspension N_{js} for H/T=1 using an different impellers and baffling configurations.

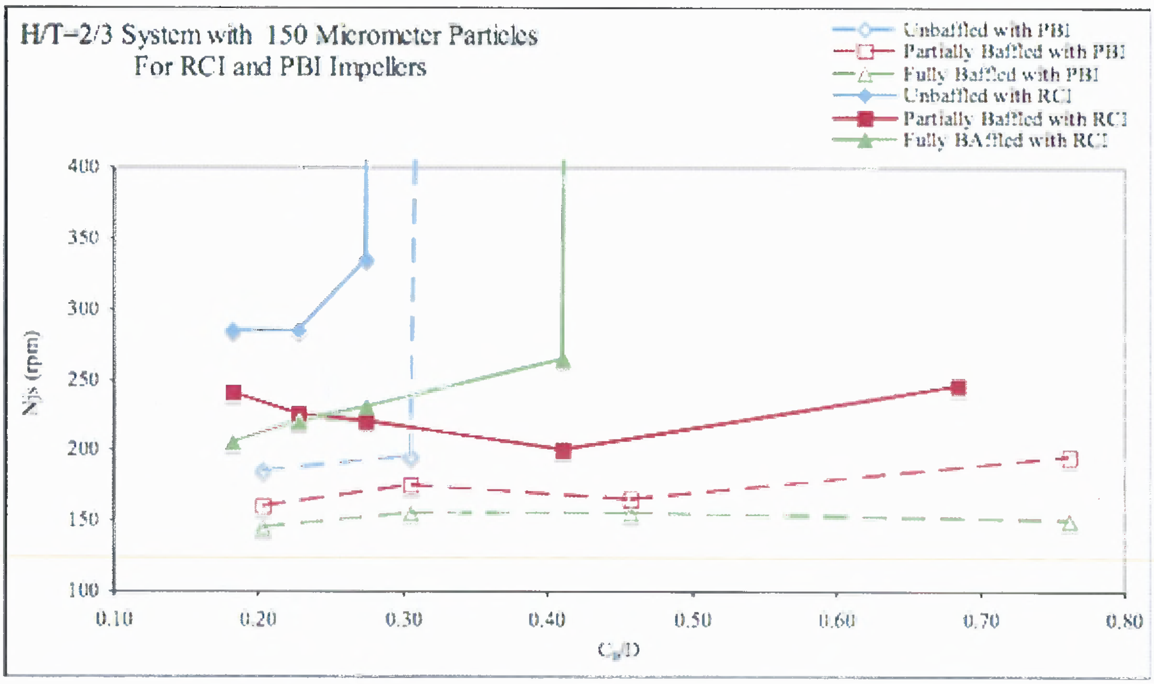


Figure 4.6 Effect of the impeller clearance ratio C_B/D on the minimum agitation speed for solid suspension N_{js} for H/T=2/3 using an different impellers and baffling configurations.

4.1.4 Effect of Submergence S_B on the Minimum Agitation Speed for Solid Suspension N_{js} for Different Impellers

Figure 4.7 shows the variation of N_{js} with the non-dimensional S_B value. Experiments were performed only using the 150μm particles. In all the cases in which the same baffling configuration was used, higher agitation speeds were required to suspend particles with RCI than with the PBT. For the PBT, the lowest values of N_{js} were obtained with the fully baffled system and the highest with the unbaffled system. N_{js} for the partially baffled system fell in between, and the curves did not overlap, as in the RCI case. For the RCI, the partially baffled system was associated, in general, with the lowest N_{js} values, while the unbaffled system produced the highest N_{js}. This figure also shows that suspension was, in general, always attainable with the PBT, except for low submergence in the unbaffled system ($S_B \leq 1$). However, when the RBI was used, this was not always the case. Two cases can be observed in which suspension was not achieved: the first occurred for relatively high values of S_B in the unbaffled and fully baffled systems. The second occurred when submergence was low and the system entrained air that prevented the solids from becoming suspended. It should be emphasized that suspension was always achievable with the partially baffled system, even when the submergence was very low.

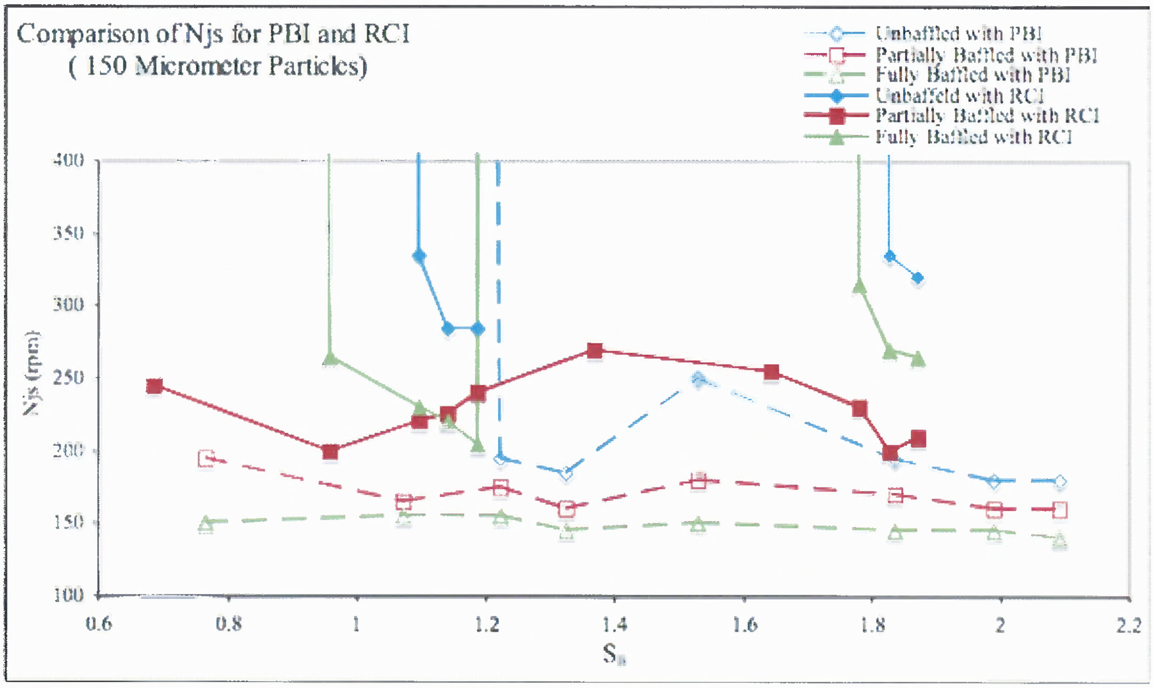


Figure 4.7 Effect of submergence S_B on the minimum agitation speed for solid suspension N_{js} using an different impellers and baffling configurations.

4.1.5 Variation of Zwietering Parameter "*s*" with C_B/D for Different Baffling Conditions

Table 4.1 gives the values of Zwietering "*s*" parameter for the RCI, which were calculated by fitting the experimental data to Equation 3.1. These results were obtained for particles having a size equal to 60μm. Appendix (A.2) reports the fitted *s* values for 150μm particles suspensions with RCI and PBI

Table 4.1 Fitted Zwietering "*s*" Values for RCI for 60 μm Solids

Baffling Condition	C_B/D	Fitted Zwietering "*s*" Values		
		H/T=1	*H/T*=2/3	*H/T*=1/2
Unbaffled	0.18	7.2198	6.3947	6.6010
Unbaffled	0.23	7.6324	6.1884	6.8073
Unbaffled	0.27	8.0450	6.3947	7.0135
Unbaffled	0.41	8.0450	7.8387	9.2826
Unbaffled	0.68	10.5203	8.4575	-
Partially Baffled	0.18	6.1884	5.5696	6.8073
Partially Baffled	0.23	6.1884	5.3633	6.8073
Partially Baffled	0.27	6.3947	5.3633	5.9821
Partially Baffled	0.41	7.2198	5.7759	5.7759
Partially Baffled	0.68	8.0450	6.1884	7.8387
Fully Baffled	0.18	10.5203	6.8073	6.8073
Fully Baffled	0.23	10.9329	6.8073	5.9821
Fully Baffled	0.27	10.9329	7.4261	6.1884
Fully Baffled	0.41	12.9957	8.6638	6.3947
Fully Baffled	0.68	14.6459	9.0764	7.2198

4.2 Results of Mixing Time Experiments Using the Colorimetric Method

4.2.1 Mixing Times in the Fully Baffled System with *H/T*=1

Figure 4.8 shows the normalized GLC output, filtered GLC output and positive and negative error curves as a function of time for a one specific sampling location (Location 20). The output started at a value corresponding to the light intensity when the solution was entirely pink and eventually reached the final level corresponding to a completely colorless solution. In Figure 4.8, the mixing time for this location was obtained by taking the reading of the time at the point where the filtered GLC output reached the 0.95 value and stayed always within the boundaries 0.95-1.05 afterwards, as shown in the figure. For this location, this reading was 4.6 seconds and the possible range of the 95% mixing for that location was 4.4-4.8 seconds, based on the ±5% error curves.

At each sampling point, the mixing time was the average of the results of three experimental replicates. The largest mixing time out of 20 locations was taken as the 95% mixing time for the entire system. The mixing time measured with this method was found to be equal to 14.8 s for the entire system. Figure 4.9 displays the individual mixing times for 20 sampling locations, which were obtained as explained here.

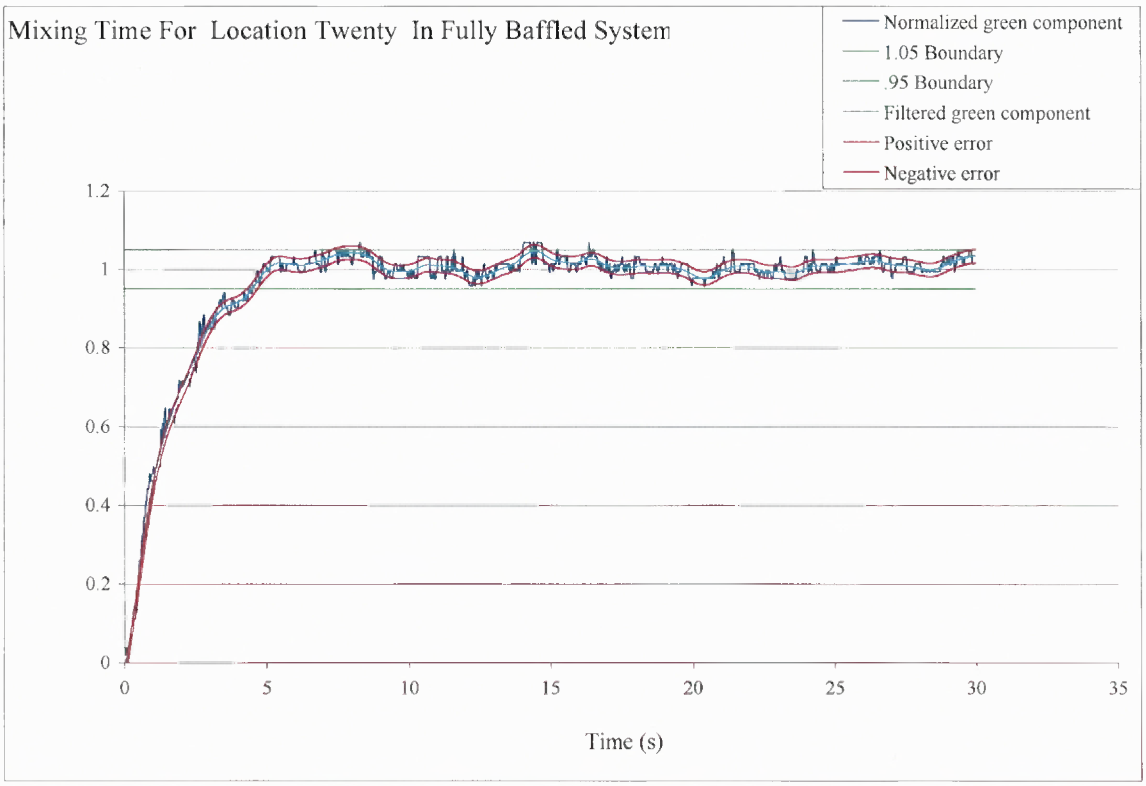


Figure 4.8 Normalized GLC output, filtered GLC output, positive and negative error curve and the 0.95 and 1.05 boundaries for Sampling Location 20.

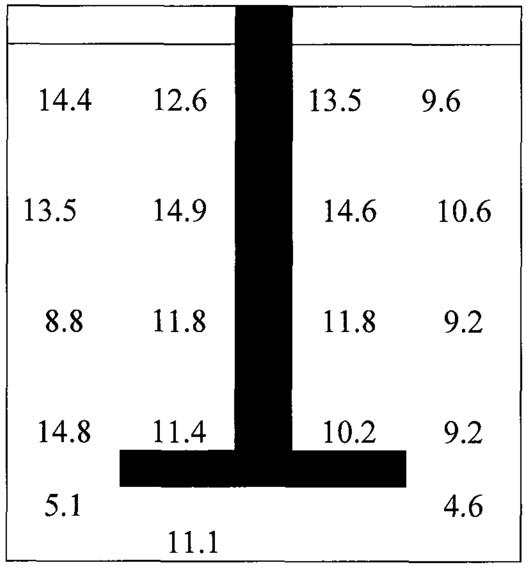


Figure 4.9 Mixing times at each sampling location in the fully baffled system.

4.2.2 Mixing Time in the Partially Baffled System with *H/T*=1

Figure 4.10 summarizes the results for the 95% mixing times (θ_{95}) at individual locations for the partially baffled system. The 95% mixing time for the entire system was 34.6 s, which was the 95% mixing time for Location 15, which gave the longest time.

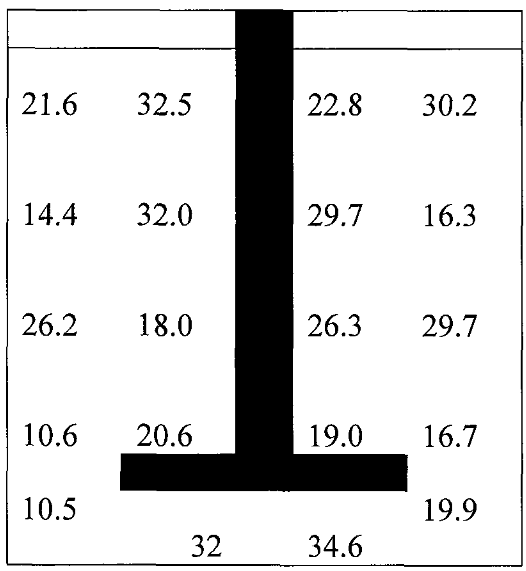


Figure 4.10 Mixing times at each sampling location in partially baffled system.

The mixing times obtained from the colorimetric method in this work for Fully and partially baffled systems were compared with the mixing times obtained from conductive method for the same system. These data was obtained from the thesis work of Nonjaros Chomcharn, a former student in this research group who studied a similar system. Similar to what he found, the 95% mixing time for a partially baffled system agitated at 100 rpm was 31.6 s. The 95% mixing time for the same system agitated at 100 rpm from this work was 34.6 s. The difference may be attributed to the presence of a conductive probe that was used in the conductivity method. For the fully baffled system agitated at 100 rpm, the 95% mixing time found in this work was 14.8 s. This value compared favorably with the result for the conductivity method, which was reported to be 14.33 s.

CHAPTER 5

CONCLUSION

The experimentally obtained values of N_{js} strongly depended on the type of baffling, and were highest in the unbaffled tank when solid particles of 150 μm were suspended and lowest in the partial baffled system.

Solid suspension is unbaffled, partially baffled, and fully baffled tanks using a retreat curve-blade impeller was always achieved in a cylindrical vessel provided with a torispherical bottom and an *H/T* ratio equal to 1 when 60 μm glass particles were dispersed in water, irrespective of the impeller clearance investigated here. However, when 150 μm glass particles were used suspension could be achieved at any impeller clearance, but only in the partially baffled tank. For the other baffling configurations, suspension was achieved only at low impeller clearances.

When the *H/T* was reduced to 2/3 or 1/2 solid suspension for the 150 μm could or could not be achieved, depending on the impeller clearance and the type of baffling.

Experimental results for the minimum agitation speed for off-bottom solid suspension, N_{js} were obtained for different *H/T* values, impeller clearances, impeller type (RBI and PBT), and baffling configurations. The picture that emerges shows a complex interplay of all the independent variables, each one apparently affecting the value of N_{js}.

In general, the partial baffling configuration appears to be the most successful in terms of achievement of the suspension state at the lowest value of the agitation speed with the retreat curve-blade impeller.

Systems stirred by a 45 degree pitch blade turbine could always suspend the solids irrespective of the solid size, and impeller clearance when $H/T=1$, and in most but not all cases, when $H/T=2/3$.

The limited number of experiments conducted to determine the 95% mixing time in the partially baffled configuration produced results that are in close agreement with results obtained previously for the same system using a conductivity method.

APPENDIX A

A.1 Results for Solid Suspension Experiments

Table A.1 Minimum Agitation Speeds for Unbaffled, Partially Baffled and Fully Baffled Systems with Retreat Blade Impeller at H/T =1/2 for 60 μm Solid Particles

C_B/D	Minimum Agitation Speed /(rpm)		
	Unbaffled System	Partially Baffled System	Fully Baffled System
0.18	175	180	180
0.23	180	180	160
0.27	185	160	165
0.41	240	155	170
0.68	Unobtainable	205	190

Table A.2 Minimum Agitation Speeds for Unbaffled, Partially Baffled and Fully Baffled systems with Retreat Blade Impeller at H/T = 2/3 for 60 μm Solid Particles

C_B/D	Minimum Agitation Speed /(rpm)		
	Unbaffled System	Partially Baffled System	Fully Baffled System
0.18	170	150	180
0.23	165	145	180
0.27	170	145	195
0.41	205	155	225
0.68	220	165	235

Table A.3 Minimum Agitation Speeds for Unbaffled, Partially Baffled and Fully Baffled Systems with Retreat Blade Impeller at H/T = 1 for 60 μm Solid Particles

C_B/D	Minimum Agitation Speed /(rpm)		
	Unbaffled System	Partially Baffled System	Fully Baffled System
0.18	190	165	270
0.23	200	165	280
0.27	210	170	280
0.41	210	190	330
0.68	270	210	370

Table A.4 Minimum Agitation Speeds for Unbaffled, Partially Baffled and Fully Baffled Systems with Retreat Blade Impeller at H/T =1/2 for 150μm Solid Particles

C_B/D	Minimum Agitation Speed /(rpm)		
	Unbaffled System	Partially Baffled System	Fully Baffled System
0.18	Unobtainable	195	175
0.23	Unobtainable	180	180
0.27	Unobtainable	170	180
0.41	Unobtainable	195	225
0.55	Unobtainable	215	Unobtainable
0.68	Unobtainable	Unobtainable	Unobtainable

Table A.5 Minimum Agitation Speeds for Unbaffled, Partially Baffled and Fully Baffled Systems with Retreat Blade Impeller at H/T = 2/3 for 150μm Solid Particles

C_B/D	Minimum Agitation Speed /(rpm)		
	Unbaffled System	Partially Baffled System	Fully Baffled System
0.18	300	255	220
0.23	300	240	235
0.27	350	235	245
0.41	Unobtainable	215	280
0.68	Unobtainable	260	Unobtainable

Table A.6 Minimum Agitation Speeds for Unbaffled, Partially Baffled and Fully Baffled Systems with Retreat Blade Impeller at H/T = 1 for 150μm Solid Particles

C_B/D	Minimum Agitation Speed /(rpm)		
	Unbaffled System	Partially Baffled System	Fully Baffled System
0.18	335	225	280
0.23	350	215	285
0.27	Unobtainable	245	330
0.41	Unobtainable	270	Unobtainable
0.68	Unobtainable	285	Unobtainable

Table A.7 Minimum Agitation Speeds for Unbaffled, Partially Baffled and Fully Baffled Systems with Four Blade Axial Impeller at H/T =2/3 for150μm Solid Particles

C_B/D	Minimum Agitation Speed /(rpm)		
	Unbaffled System	Partially Baffled System	Fully Baffled System
0.18	185	160	145
0.27	195	175	155
0.41	Unobtainable	165	155
0.68	Unobtainable	195	150

Table A.8 Minimum Agitation Speeds for Unbaffled, Partially Baffled and Fully Baffled Systems with Four Blade Axial Impeller at H/T = 1 for 150μm Solid Particles

C_B/D	Minimum Agitation Speed /(rpm)		
	Unbaffled System	Partially Baffled System	Fully Baffled System
0.18	180	150	140
0.27	180	160	145
0.41	195	170	150
0.68	250	180	145

A.2 Fitted Zwietering S Values using Experimental Data

Table A.9 Fitted Zwietering s Values for RCI for 150 μm Solids

Baffling Condition	C_B/D	Fitted Zwietering s Values		
		H/T=1	H/T=2/3	H/T=1/2
Unbaffled	0.18	10.9913	9.7892	-
Unbaffled	0.23	11.5066	9.7892	-
Unbaffled	0.27	-	11.5066	-
Unbaffled	0.41	-	-	-
Unbaffled	0.68	-	-	-
Partially Baffled	0.18	7.2131	8.2435	6.1826
Partially Baffled	0.23	6.8696	7.7283	5.6674
Partially Baffled	0.27	7.9000	7.5565	5.3239
Partially Baffled	0.41	8.7587	6.8696	6.1826
Partially Baffled	0.68	9.2739	8.4152	-
Fully Baffled	0.18	9.1022	7.0413	5.4957
Fully Baffled	0.23	9.2739	7.5565	5.6674
Fully Baffled	0.27	10.8196	7.9000	5.6674
Fully Baffled	0.41	-	9.1022	7.2131
Fully Baffled	0.68	-	-	-

Table A.10 Fitted Zwietering s Values for PBI for 150 μm Solids

Baffling Condition	C_B/D	Fitted Zwietering s Values	
		H/T=1	H/T=2/3
Unbaffled	0.20	5.6506	5.8075
Unbaffled	0.30	5.6506	6.1214
Unbaffled	0.46	6.1214	-
Unbaffled	0.76	7.8480	-
Partially Baffled	0.20	5.0227	5.0227
Partially Baffled	0.30	5.0227	5.4936
Partially Baffled	0.46	5.3366	5.1797
Partially Baffled	0.76	5.6506	6.1214
Fully Baffled	0.20	4.3949	4.5518
Fully Baffled	0.30	4.5518	4.8658
Fully Baffled	0.46	4.5518	4.8658
Fully Baffled	0.76	4.7088	4.7088

APPENDIX B

B.1 Matlab Source Code for Detection of RGB Components

MATLAB Source Code to Obtain the Picture from avi File

```
clear all
clc
close all

%
%save the movie as a Matlab file
vidObj=mmreader('directory of avi file');
images = read( vidObj );

%open the first picture and select the 20 points of interest
%for each point clic on Export data to workspace
%name of the point 'P1'

%display initial picture
Size=size(images);
picture=images(:,:,:,1);
image (picture)


%select the points then close the picture and start the second m-file
```

B.2 Matlab Source Code to Extract Green Light Component from Individual Sampling Point

MATLAB Source Code to Extract Green Component (G) from Individual Sampling Point

```
% program to calculate the mixing time from a series of pictures taken with
% a regular camera
% The parameter used for tha analysis is the COLOR Intensity - generally
% the GREEN color intensity is the only one considered since it is less
% sensible to differences in external light

%clc
%clear all
%close all

%INPUT (msec!!!)
InjectionTime=5110;                %msec

RGB_tot=[];

%loop to take each picture from the movie
for i=1:Size(4)

picture=images(:,:,:,i);

%find the RGB component for each picture
%selected points


B=[P1.Position; P2.Position; P3.Position; P4.Position;
P5.Position;P6.Position;P7.Position;P8.Position;P9.Position;P10.Position; P11.Position;
P12.Position;P13.Position;P14.Position;P15.Position;P16.Position;P17.Position;P18.Position;P19.Position;P20.Position;];
RGB_pict=[];

%loop to analize all points
for y=1:20
    RGB=picture(B(y,2),B(y,1),:);
    RGB_pict=[RGB_pict;RGB];
end
RGB_tot=[RGB_tot RGB_pict];
end
%evolution of red component with time (columns) and points (rows)
R_tot=RGB_tot(:,:,1);
G_tot=RGB_tot(:,:,2);
B_tot=RGB_tot(:,:,3);

%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%
%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%
%time vector definition

time (1)=0;
for i=2:Size(4)
  time(i)=time(i-1)+32.987288;
end

%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%
%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%
```

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