ABSTRACT

EXPERIMENTAL DETERMINATION OF SOLIDS SUSPENSION WITH ANGLED IMPELLER IN A PHARMACEUTICAL MIXING VESSEL

by
Yingxi Tang

Angled-mounted impellers are commonly used in a number of pharmaceutical industry applications, from laboratory reactors to full-scale tanks. In these systems, the impeller is not centrally and vertically mounted, as in most stirred vessels, but it enters the vessel, and is therefore immersed in the fluid, at an angle from the vertical. This arrangement provides some baffling effects that would not otherwise exist if the impeller was centrally mounted.

One common requirement for such systems is that they are capable of suspending small solids dispersed in the liquid. Therefore, it is critical to know the value of the minimum agitation speed at which solids become just suspended, N_{js}. Despite their common use in industry, and especially the pharmaceutical industry, these systems have received little attention in the literature in general and as far as their ability to solid suspension is concerned in particular.

Therefore, the focus of this work was to determine under what conditions fine divided solids become suspended in a liquid-filled, unbaffled stirred vessel provided with angled-mounted impellers. A small laboratory vessel with a hemispherical bottom was used here. Different types of axial and radial impellers mounted at different angles (0°, 5° and 10°) at different off-bottom impeller clearances were tested. The value of N_{js} was experimentally obtained by visually inspecting the tank bottom and determining the impeller agitation speed at which the solids were observed to rest on the tank bottom for

no more than 1-2 seconds before being swept away. In addition, a novel observer-independent criterion was developed, relying on the experimental determination of the fraction of solids remaining on the vessel bottom at increasing agitation speeds. This approach results in values of N_{js} that are close agreement with the visually determined N_{js} values.

This approach was used to determine N_{js} for different impellers under a number of operating conditions. In general, mounting the impeller at an angle resulted in N_{js} values lower than those obtained with centrally mounted impellers. In addition, it was found that lower impeller clearances resulted in lower N_{js} values, although the minimum for N_{js} was obtained when the impeller clearance was about 20% of the vessel diameter.

The results obtained in this work are expected to be applicable to a number of systems currently used in the pharmaceutical industry.

EXPERIMENTAL DETERMINATION OF SOLIDS SUSPENSION WITH ANGLED IMPELLER IN A PHARMACEUTICAL MIXING VESSEL

by

Yingxi Tang

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APPROVAL PAGE

EXPERIMENTAL DETERMINATION OF SOLIDS SUSPENSION WITH ANGLED IMPELLER IN A PHARMACEUTICAMIXING VESSEL

Yingxi Tang

Dr. Piero M. Armenante, Thesis Advisor Date
Distinguished Professor of Chemical, Biological and Pharmaceutical Engineering, NJIT

Dr. Robert R Barat, Committee Member Date
Professor of Chemical, Biological and Pharmaceutical Engineering, NJIT

Dr. Laurent Simon, Committee Member Date
Associate Professor of Chemical, Biological and Pharmaceutical Engineering, NJIT

BIOGRAPHICAL SKETCH

Author: Yingxi Tang

Degree: Master of Science

Date: May 2015

Undergraduate and Graduate Education:

- Master of Science in Pharmaceutical Engineering,
  New Jersey Institute of Technology, Newark, NJ, 2015

- Bachelor of Science in Biotechnology,
  Beijing Union University, People Republic of China, 2013

Major: Pharmaceutical Engineering

To my family

With the great support during my life

To my friends

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NOMENCLATURE

D	Impeller diameter (mm)
T	Tank diameter (mm)
H	Liquid height (mm)
C_b	Bottom clearance (mm)
N	Rotational speed (rpm)
N_{js}	Minimum agitation speed for solid suspension
x	Coordinate axis
y	Coordinate axis

Greek letter

ρ	liquid density (kg/m^3)
υ	kinematic viscosity, m^2/s

Subscripts

js	refers to just-suspended conditions
l	refers to liquid
s	refers to solid

CHAPTER 1

INTRODUCTION

1.1 Background

Stirred vessels and reactors are often encountered in pharmaceutical manufacturing facilities where they are used for a variety of applications. In the pharmaceutical industry, these reactors are routinely employed to synthesize Active Pharmaceutical Ingredients and their intermediates. These types of reactor are very versatile, and there are many different operations that are performed in these vessels, ranging from mixing, to solid suspension, crystallization, precipitation, multi-phase and chemical reactions. Many such systems are provided with centrally mounted impellers and four baffles to ensure adequate mixing. In the pharmaceutical industry, baffled tanks are commonly used because baffles can eliminate swirling flow. In these vessels, four flat-plate vertical baffles mounted at the vessel wall are commonly used. The literature on this type of systems is very substantial.

However, angled-mounted impellers are also commonly used in a number of pharmaceutical industry applications, from laboratory reactors to full-scale tanks. In these systems, the impeller is not centrally and vertically mounted, as in most stirred vessels, but it enters the vessel, and is therefore immersed in the fluid, at an angle from the vertical. This arrangement provides some baffling effects that would not otherwise exist if the impeller was centrally mounted.

A typical requirement for such systems is that they are capable of suspending small solids dispersed in the liquid. Therefore, it is critical to know the value of the minimum agitation speed at which solids become just suspended, N_{js}. Despite their common use in industry, and especially the pharmaceutical industry, these systems have received little attention in the literature in general and as far as their ability to solid suspension is concerned in particular.

In the study by Myers et al. (2011), these authors worked with an unbaffled tank provided with an angled impeller to test the power number of angle-mounted impellers under different conditions. When an axial-flow impeller was used in this configuration, the suspension was achieved at agitation speed lower than in centrally mounted systems.

There are three levels to classify the degree of solid suspension, 1. On-bottom motion; 2. Completed off-bottom suspension; 3. Uniform suspension (Paul et al., 2004); According to many research papers, they most used complete off-bottom suspension to determine solid suspension. Below this off-bottom particle suspension state, the complete interfacial surface area is very difficult to utilized. In 1998, Armenante and Uehara-Nagamine determined the impeller agitation speed N_{js}, at which N_{rpm} the just suspended state was achieved by the particles for a number of standard systems. Although studies on N_{js} can be found in the literature for centrally mounted impellers, the information on N_{js} for angle-mounted impellers is very limited.

In Dr. Piero Armenante's laboratory, Anqi Zhou found a novel approach to determine N_{js} of solid suspension in a glass-lined stirred tank. In that work, she used a torispherical-bottomed tank and a hemispherical-bottomed tank. A novel method to determine minimum agitation speed N_{js} was obtained ($N_{js\text{-}As\text{-}Method}$ and $N_{js\text{-}Ds\text{-}Method}$). This method turned out to be very effective and was extended to the work carried out in the present work.

1.2 Objectives of This Work

The typical method to measure experimentally N_{js} is that of Zwietering's (1958). Accordingly, N_{js} is obtained by visually inspecting the tank bottom and visually determining the impeller agitation speed at which the solids are observed to rest on the tank bottom for no more than 1-2 seconds before being swept away. Currently, this is the primary way to obtain N_{js} experimentally. Although this method is quite reliable, there is clearly a need to develop a new method that is observed-independent.

Therefore, the first objective of this work was to develop a new experimental method to determine the minimum agitation speed, N_{js} in unbaffled vessels with angled impellers. This was achieved here by developing a novel observer-independent criterion relying on the experimental determination of the fraction of solids remaining on the vessel bottom at increasing agitation speeds.

The second of objective of this work was to determine N_{js} for different axial and radial impellers under a number of operating conditions. This was achieved by using the newly developed criterion to determine N_{js} in a small laboratory vessel in which impellers were mounted either in a central position or at different angled positions were used in order to compare the performance of these two systems.

The configuration of the system used here is based on the previous work of Nonjaros Chomcharn, Dilanji Bhagya Wijayasekara and Anqi Zhou, who completed their thesis research work in this laboratory.

CHAPTER 2

EXPERIMENTAL APPARATUS, MATERIALS AND METHODS

The approach described in this section was used to obtain the minimum agitation speed for off-bottom solids suspension N_{js}, and obtain the diameter and area of solids in the bottoms of vessels as a function of N.

2.1 Apparatus

2.1.1 Hemispherical-Bottomed Mixing Vessel and Impellers

The vessel used here was made by Chemglass (Chemglass Life Sciences, Vineland, NJ). The vessel volume was 4000mL.The basic dimensions of this vessel, shown in Figure 2.1, were as follows:

- Internal diameter (T): 160.94 mm
- Overall height: 310 mm
- Height of dish bottom: 80.47 mm
- Height of cylindrical section: 229.53 mm

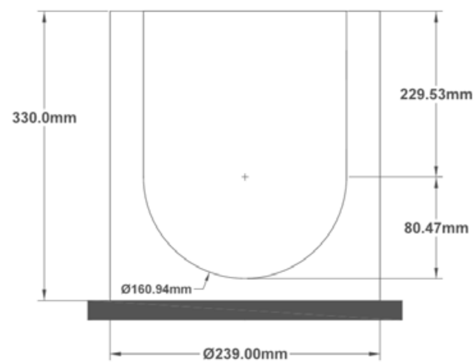
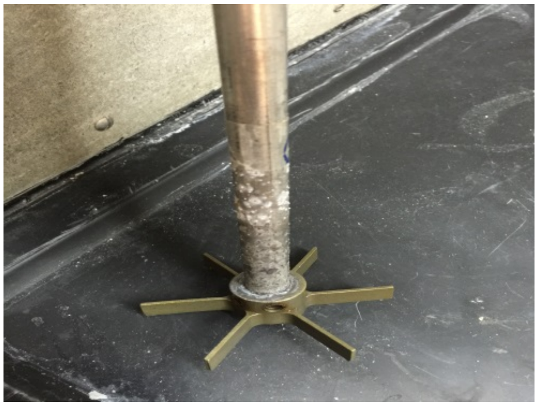


Figure 2.1 Dimensions of Hemispherical-Bottomed were used in the agitator mixing system.

A black mark pen was used to draw a circle with the diameter d=10mm at the bottom of the vessel in order to have the reference scale distance for future used in data acquisition with a camera. The mixing vessel was placed in a larger “host” cylindrical tank (H=330mm D= 239mm) and this whole assembly was placed on laboratory jacks in order to change easily the position of the vessel with respect to the motor (and especially the impeller clearance) and to better observe the vessel bottom for easy determination of $N_{js\text{-}visual}$ and to take pictures. A mirror is placed at a 45-degree angle aligned with the horizontal plane in order to see the bottom of the mixing vessel. This set was illuminated with a 100W lamp, during the agitator speed while recording the picture.

In this experiment, four types of impellers were used, as shown in Figures 2.2 (a), (b), (c) and (d), i.e., a 6-blade flat-blade turbine (6-FBT), an Lightnin A-310, a disk turbine (DT), and a 6-blade curved-blade turbine (6-CBT).The following are the impeller dimensions measured with a caliper, for the 6-FBT (6-flat-blade turbine impeller): impeller diameter (D) =71.4 mm; blade length = 14.8 mm; blade thickness = 1.5 mm; and an impeller diameter-to-tank diameter ratio *D/T*, of 0.443. The dimensions of the A310 were as follows (Figure 2.3(b)): impeller diameter D = 89.60 mm; blade length = 32 mm; blade width = 15.1 mm; blade thickness = 1.18 mm. and *D/T*, of 0.557. The dimensions of the DT were as follows Figure 2.4(c)): impeller diameter D = 71.4 mm; blade length = 14.8 mm; blade thickness = 1.5mm, and *D/T* of 0.443. The dimensions of the 6-CBT were as follows (Figure 2.3(d)): impeller diameter D = 85.1 mm; blade length = 22 mm; and blade thickness= 1.5mm *D/T*, of 0.404. Two types of impeller, 6-PBT and DT, have the same diameter.

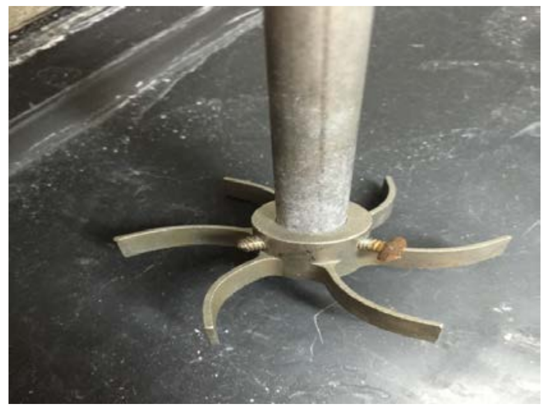
The selected impellers were attached to a shaft (diameter 12.52 mm), rotated by a 0.25 HP motor (Chemglass, Model CG-2033-11), and controlled by an external controller(Chemglass,ModelCG-2033-31).



(a)

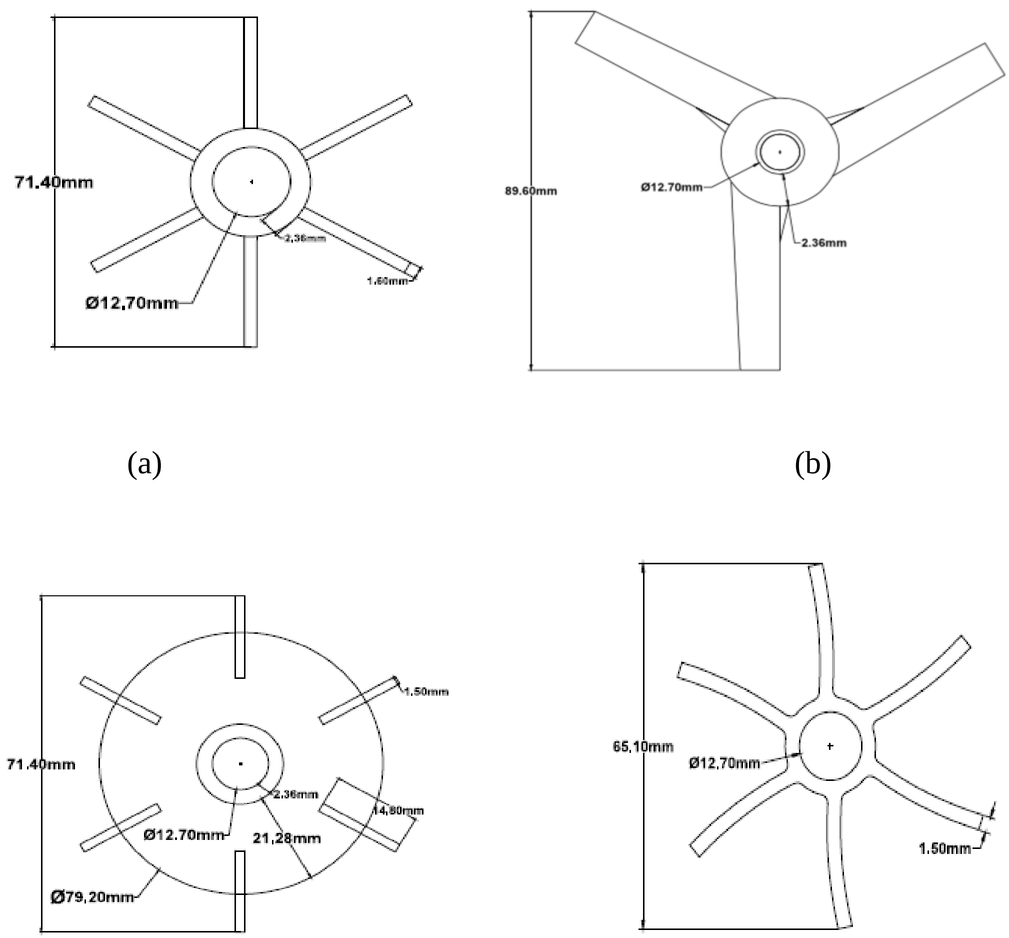
(b)

(c)



(d)

Figure 2.2 Impeller were used in the hemispherical-bottom mixing system
(a) 6-FBT impeller (b) Lighting A310 impeller; (c) DT impeller; and (d) 6-CBT impeller.



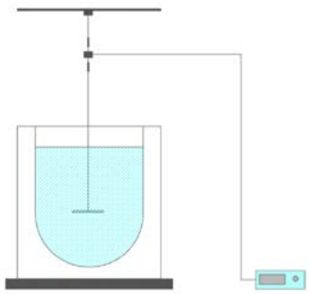
(a) (b)

(c) (d)

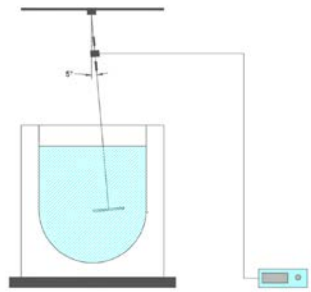
Figure 2.3 Dimensions of impeller size were used in the hemispherical-bottom mixing system plane image (a) 6-FBT impeller (b) A310 impeller; (c) DT impeller; and (d) 6-CBT impeller.

2.1.2 Angle-Mounted Agitator Geometry

In an experiment, an impeller was mounted on a shaft having a diameter of 12.52 mm and connected to the motor. The motor motor-shaft-impeller assembly was then placed on its own support separate from the vessel so that the impeller was inside the vessel. Depending on the experiment, the motor-shaft-impeller assembly was mounted a three different angles: 0º, 5º, and 10º (Figure 2.4(a), (b), (c); Figure 2.5). In all cases, the impeller was first placed centrally in the vessel. When the 0º position was investigated, the impeller was left in this position. When the angled impeller positions were investigated, the motor-shaft-impeller assembly was rotated as needed and the angle was measured with digital angle ruler (iGaging). When placed in the 5º position and 10º position, the shaft centerline was, respectively, 9 mm and 18 mm away from the vessel centerline measured at the liquid surface ($H=T$) (Figure 2.6). The impeller clearance was varied depending on the experiments. The whole agitator is shown in Figure 2.5.



(a)



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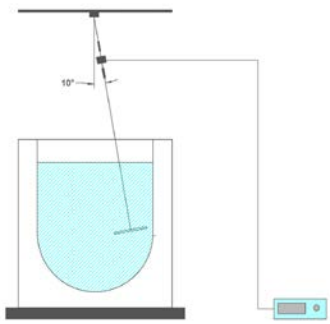


(c)

Figure 2.4 Agitator system used in the experiment (a)centered impeller mounted system; (b) 5-degree angled impeller mounted system; (c) 10-degree angled impeller mounted system.

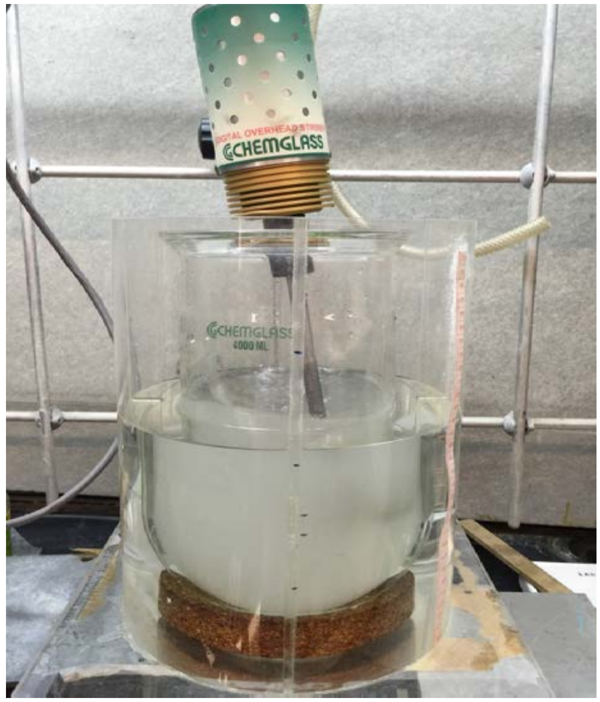


Figure 2.5 Hemispherical-Bottomed glass vessel system with angle-mounted impeller.

Figure 2.6 presents the top view of the angle-mounted agitator. The coordinate system to describe the impeller is also shown in this figure. In this work, the impeller was mounted in the x (+) direction. The impellers were rotates clockwise, i.e., pumping downwards for the case of the A310 impeller.

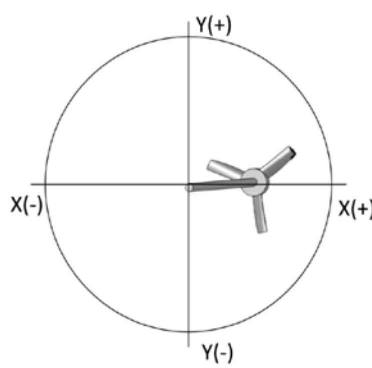


Figure 2.6 Top view illustrating coordinate system.

An impeller was placed at the different (C_b/T) locations with respect to the vessel. In this work, five different clearance ratios were used i.e., C_b/T = 0.5, 0.4, 0.3, 0.25 and 0.2, corresponding to clearances equal to 74mm, 64.376mm, 48.282mm, 40.235mm, and 32.186mm. In previous work (Myers et al., 2011), the clearance ration was set at C_b/T =0.25. In this type of experiments, the clearance is important since different C_b/T may cause the flow pattern to be changed. If the impeller was placed at an angle, the clearance was measure from the lowest point on the impeller to the bottom of the vessel (Figure 2.7).

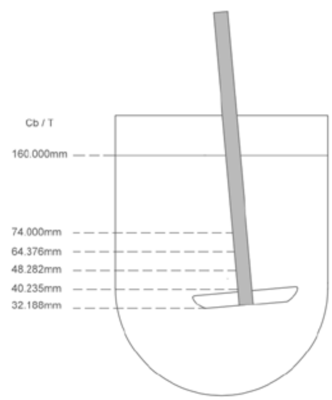


Figure 2.7 C_b/T dimension in the Hemispherical-Bottomed vessel.

2.1.3 Agitation System and Data Acquisition

The agitation speed was measured with a digital tachometer. Photographs of the tank bottom were taken with a Sony-5T camera to determine the area of the vessel bottom covered by solids, as explained below. Images were then processed with ImageJ software 1.48 (http://imagej.nih.gov/ij/download.html) to quantify this area.

2.2 Materials

Distilled water at room temperature was used in all experiments and the liquid height was always equal to the tank diameter in all experiments ($H=T$).

Glass beads having average of diameters of 200 μm were used as the disperse phase. Prior to their use, prescreened glass beads were sieved. Four US standard screens of mesh size 40, 60, 80 and 100 were selected. 20g Glass beads were used in the system. Process glass beads in the mesh, shaking for 5 minutes. The particles retained on the size of 80 mesh (with an average diameter size of 200 μm) screen were collected. The solid concentration used in all the experiment was 0.5% (W/W).

2.3 Experimental Method and Approach to N_{js} Determination

It was noticed that the particles some fines, which made the suspension cloudy and required almost 15 minutes to settle down. Therefore, the fines were removed as follows. Stokes' law was used to calculate the setting time for particle size of interest.

$$V_{pt} = g\frac{(\rho_p - \rho)}{18\mu} D_p^2 \tag{2.1}$$

$$\mathrm{t} = \frac{H}{V_{pt}} \tag{2.2}$$

The setting time was calculated to be about 5 s for 200μm particles settling in a 430 mm-tall cylinder, assuming that the particle Reynolds number (Re) was less than one and drag coefficient was 24/Re. The particles were place in the cylinder with water, the system was shaken, and after 5s, the supernatant (and the fines) was discarded. Fresh water was added and the process was repeated replaced two more times. No fines could be observed at this point.

In an actual experiment, the mixing tank was inserted in the host tank and the whole assembly was placed on two laboratory jacks. The vessel was filled with water in the level H (160.94mm) was equal to the tank diameter (H/T=1), corresponding to a liquid volume V of 2.25 liters. This assembly was positioned under the impeller so that the impeller was in the vessel. The host tank was filled up with the same level of water as the hemispherical-bottomed vessel (up to 160.97mm for H/T=1 system). The host'square vessel helped to support the vessel which made it more stable when changing the impeller clearance. The solid particles (20g) were added to the vessel where they settled. A mirror was placed 45-degree under the bottom of the tank. A light (100W lamp) illuminating the bottom portion of the vessel was turned on and a video camera (Sony 5-T) was used to take the picture of the vessel bottom (to calculate the D_s, A_s and A~D).

In this work, a typical experiment consisted of setting the agitation speed at a given value, N. When the stable speed was reached a digital picture of the vessel bottom was taken. This procedure was repeated at different increasing speeds. Each picture showed the area A_s on the vessel bottom covered with solids at that speed. In experiments with centered impellers, this area nearly perfectly circular and was to measure. However, with the angle-mounted impeller system, the area was not a perfect circle. Therefore, A_s was calculated using the ImageJ software applied to these images. The A_s values at different N values were further used as explained below. Typically, images were taken at five N values each spaced 20 rpm apart.

In addition, the visual value of the N_{js} was obtained using the *Zweitering's* criterion (*Zweitering* 1958). This is defined as the agitation speed at which no particles were visually observed to be at rest on the tank bottom for more than one to two seconds.

All experiments were repeated in triplicates. Experiments were conducted in different angles and different impellers, the impeller clearances ratio and summarized in Table 2.1

Table 2.1 Summary of Experimental Conditions and Variable Ranges Tested in This Work.

System Variable	Centered		5-Degree Angle	10-Degree Angle
Impeller Type	6-FBT	A310	DT	6-CBT
C_b/T	0.2, 0.25, 0.3, 0.4, 0.05			
Diameter D	71.4mm	89.6mm	71.4mm	65.1mm
D/T	0.443	0.557	0.443	0.404
Particle Size	200 µm			

2.4 Data Processing and Analysis for the Determination of $N_{js\text{-}method}$.

The raw image data captured in .jpg format by the camera ware transfer to a computer. The image was processed with software Image J. The circle originally drawn on the vessel bottom (10mm in diameter) was used as a scale (Figure 2.8 (a)). The software automatically adjusted the color threshold. The color was chosen, and the resulting surface area of portion of the image occupied by the solids (darker area) was calculated by the software.

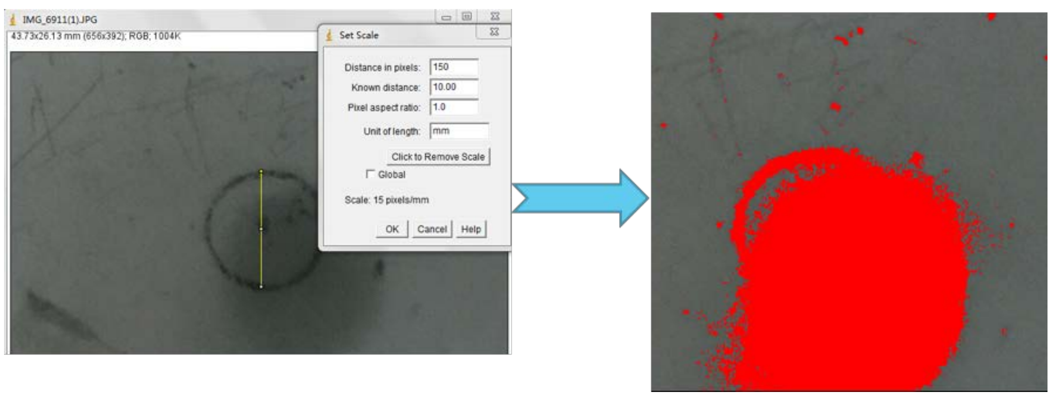


Figure 2.8 Image J processing picture

In order to determine how the fraction of unsuspended solids at the vessel bottom shrank with increasing agitation speeds and extrapolate the data to obtain N_{js}, three different criteria were used:

1. Three diameters were measured from each image (longest, shortest, and in between). An average diameter was the calculated. Plots of D_s vs. N were then constructed, a regression line was passed through these points, and the value of N for $D_s \rightarrow 0$ was taken as the estimated value of N_{js}, called here $N_{js\text{-}Ds}$.

2. The value of A_s was obtained from each image. Plots of A_s vs. N were then constructed, a regression line was passed through these points, and the value of N for $A_s \rightarrow 0$ was taken as the estimated value of N_{js}, called here $N_{js\text{-}As}$.
3. The diameter D_{As} of an equivalent circle with area A_s was calculated from:

$$\mathrm{D} = \sqrt{\frac{4A}{\pi}} \tag{2.3}$$

Plots of D_{As} vs. N were then constructed, a regression line was passed through these points, and the value of N for $D_{As} \rightarrow 0$ was taken as the estimated value of N_{js}, called here $N_{js\text{-}As}$.

CHAPTER 3

RESULTS AND DISCUSSION

3.1 Result of Solid Suspension Experiment

3.1.1 Comparison of the N_{js} Values Obtained with the Proposed Methods with Those Obtained with the Conventional Zwietering's Visual Approach

In this section, the results of the experiment analyzed using the first two of the three different methods described above to obtain N_{js} are compared with the conventional value of N_{js} obtained with Zwietering's approach. The third method was less successful in correlating the data and it is described separately and later in this chapter.

Accordingly for each experimental, the values of D_s and A_s were obtained for increasing values of the agitation speed, N. Figures 3.1-3.12 show the N-vs-D_s and N-vs-A_s plots for different condition and the resulting N_{js} values in different situation.

The results are obtained from the experiment with the 6-FBT impeller, A310, DT and 6-CBT impellers. The first observation from Figure 3.1 is that the points align themselves on straight lines(R=0.99). From a regression of each line is possible to predict the value of N_{js} at the intersection of each line with the y-axis, thus identifying the values of N_{js} using the D_s data ($N_{js\text{-}Ds\text{-}Method}$) or using the A_s data ($N_{js\text{-}As\text{-}Method}$). For a 6-FBT impeller mounted in the center, Figure 3.1(a) and Figure 3.1(b) show that the $N_{js\text{-}Visual}$ is 389*rpm*, while $N_{js\text{-}Ds\text{-}Method}$ is equal to 393.58 *rpm* and $N_{js\text{-}As\text{-}Method}$ is equal to 370 *rpm*. (although, as described later, the difference between $N_{js\text{-}Visual}$ and $N_{js\text{-}A\sim D\text{-}Method}$ is significant (Section 3.1.3)). This implies that the D_S method is a valid alternative to the conventional method to determine N_{js}. For the 5-degree angled system shown in Figure

3.1 (c) and Figure 3.1(d), an N_{js} value of 260 rpm is close to $N_{js\text{-}Ds\text{-}Method}$ (266.40 rpm) whereas $N_{js\text{-}As\text{-}Method}$ was 283.26 rpm. The same result for the 10-degree angled impeller mounted case shown in Figure 3.1 (e) and Figure 3.1 (f) visual observation resulted in $N_{js\text{-}Visual}$ equal to 228 *rpm* which is very similar to N_{js} measured by $N_{js\text{-}Ds\text{-}Method}$ (226rpm) but still has a big different from the $N_{\text{js-As-Method}}$ (269.95 rpm).

Similar results were obtained for the A310 impeller as shown in Figure 3.2. In the unbaffled system, the value of N_{js}-$_{\text{visual}}$ is 411 rpm and the N_{js} by *As-Method* is 468 rpm. The value of N_{js} by *Ds-Method* is 368.87 rpm.

The same results were obtained with the DT impeller. In the 5-degree angled system, the value of N_{js} by visual observation is 344 rpm and the N_{js} by $A_{s\text{-}Method}$ is 319 rpm. The value of N_{js} by $D_{s\text{-}Method}$ is 340 rpm. It is also identified that the N_{js} using $D_{s\text{-}method}$ is better than $A_{s\text{-}method}$. The result can be also concluded by Figure 3.3(a), (b), (c),(d).

Additionally, the Hemispherical-bottomed tank equipped with and 6-CBT impeller was tested. Results of $A_{s\text{-}Method}$ and $D_{s\text{-}Method}$ applied to this system are shown in Figure 3.4. The results of these experiments agreed with those of the previous experiments: in unbaffled systems visually determined the N_{js} values were in close agreement with the $N_{js\text{-}Ds\text{-}Method}$, the same results in angled 5-degree angled impeller mounted system and 10-degree angled impeller mounted system.

It can be concluded that the proposed approach to N_{js} determination based on the D_s approach is valid, at least for the systems tested here, and that this method can be extended to other systems under different operating conditions.

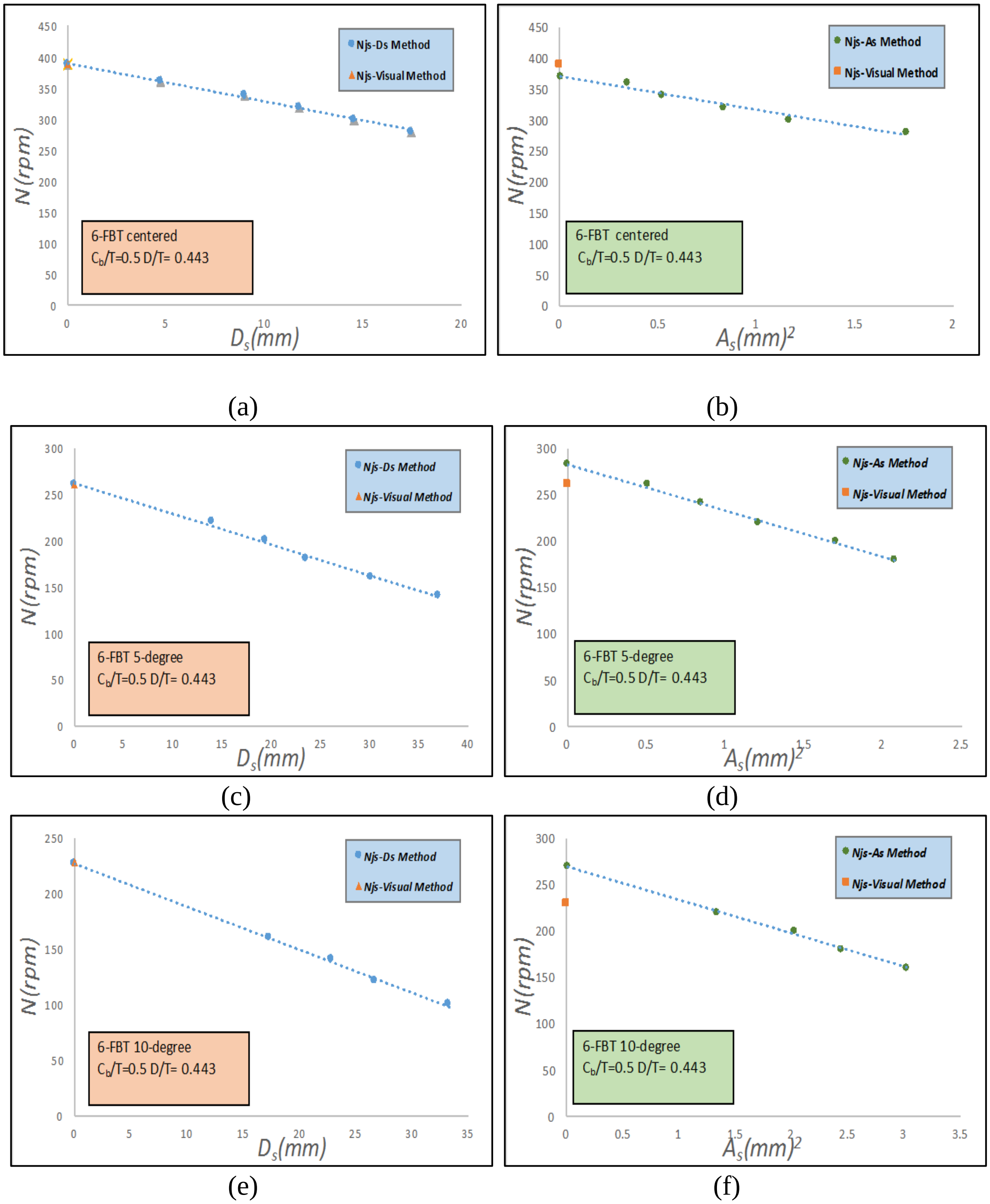


Figure 3.1 N_{js} measured the Hemispherical-bottomed tank with the 6-FBT using for 200 µm particles: (a) $N_{js\text{-}Ds\text{-}Method}$ in centerline system;(b) $N_{js\text{-}As\text{-}Method}$ in centered system;(c) $N_{js\text{-}Ds\text{-}Method}$ in 5-degree system; (d) $N_{js\text{-}As\text{-}Method}$ *in* 5-degree system; *(e)* $N_{js\text{-}Ds\text{-}Method}$ in 10-degree system ;(f) $N_{js\text{-}As\text{-}Method}$ in 10-degree system.

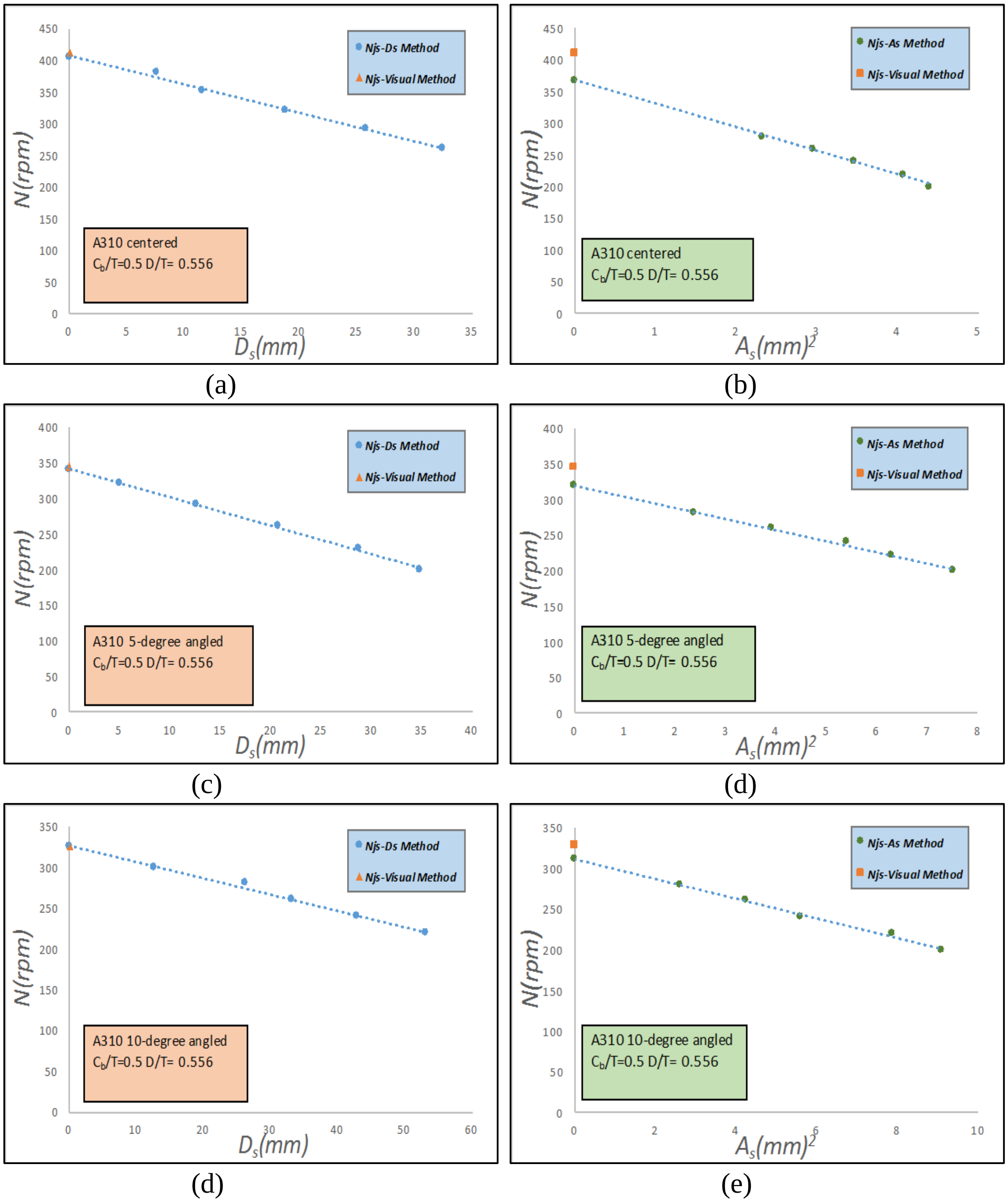


Figure 3.2 N_{js} measured the Hemispherical-bottomed vessel with the A310 using for 200 μm particles: (a) $N_{js\text{-}Ds\text{-}Method}$ in centered system;(b) $N_{js\text{-}As\text{-}Method}$ in centered system;(c) $N_{js\text{-}Ds\text{-}Method}$ in 5-degree system; (d) $N_{js\text{-}As\text{-}Method}$ in 5-degree system; (e) $N_{js\text{-}Ds\text{-}Method}$ *in* 10-degree system; (f) $N_{js\text{-}As\text{-}Method}$ in 10-degree system.

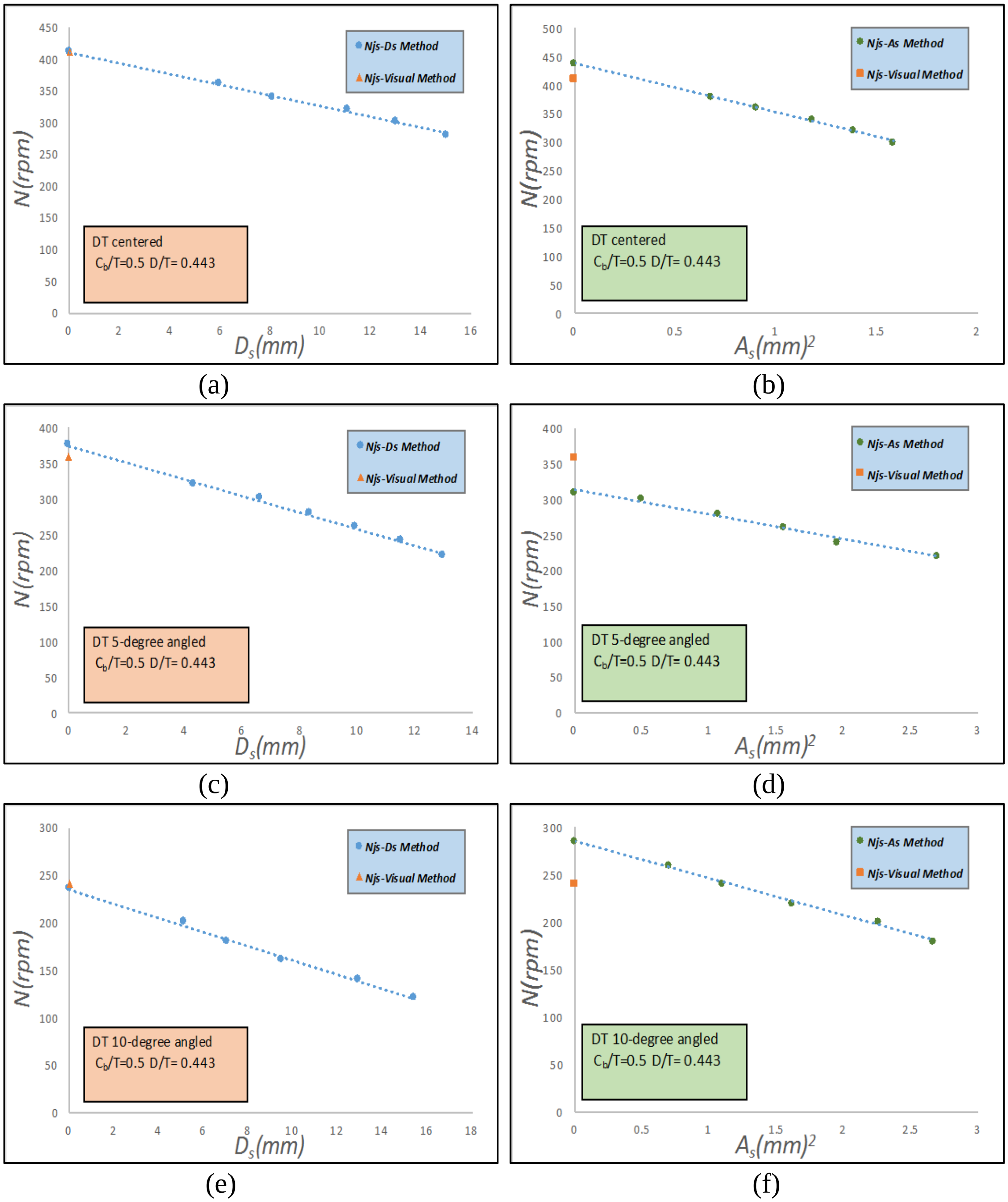


Figure 3.3 N_{js} measured the Hemispherical-bottomed tank kwith the DT impeller using for 200 µm particles: (a) $N_{js\text{-}Ds\text{-}Method}$ in centered system; (b) $N_{js\text{-}As\text{-}Method}$ in centered system; (c) $N_{js\text{-}Ds\text{-}Method}$ in 5-degree system; (d) $N_{js\text{-}As\text{-}Method}$ in 5-degree system; (e) $N_{js\text{-}Ds\text{-}Method}$ in 10-degree system; (f) $N_{js\text{-}As\text{-}Method}$ in 10-degree system.

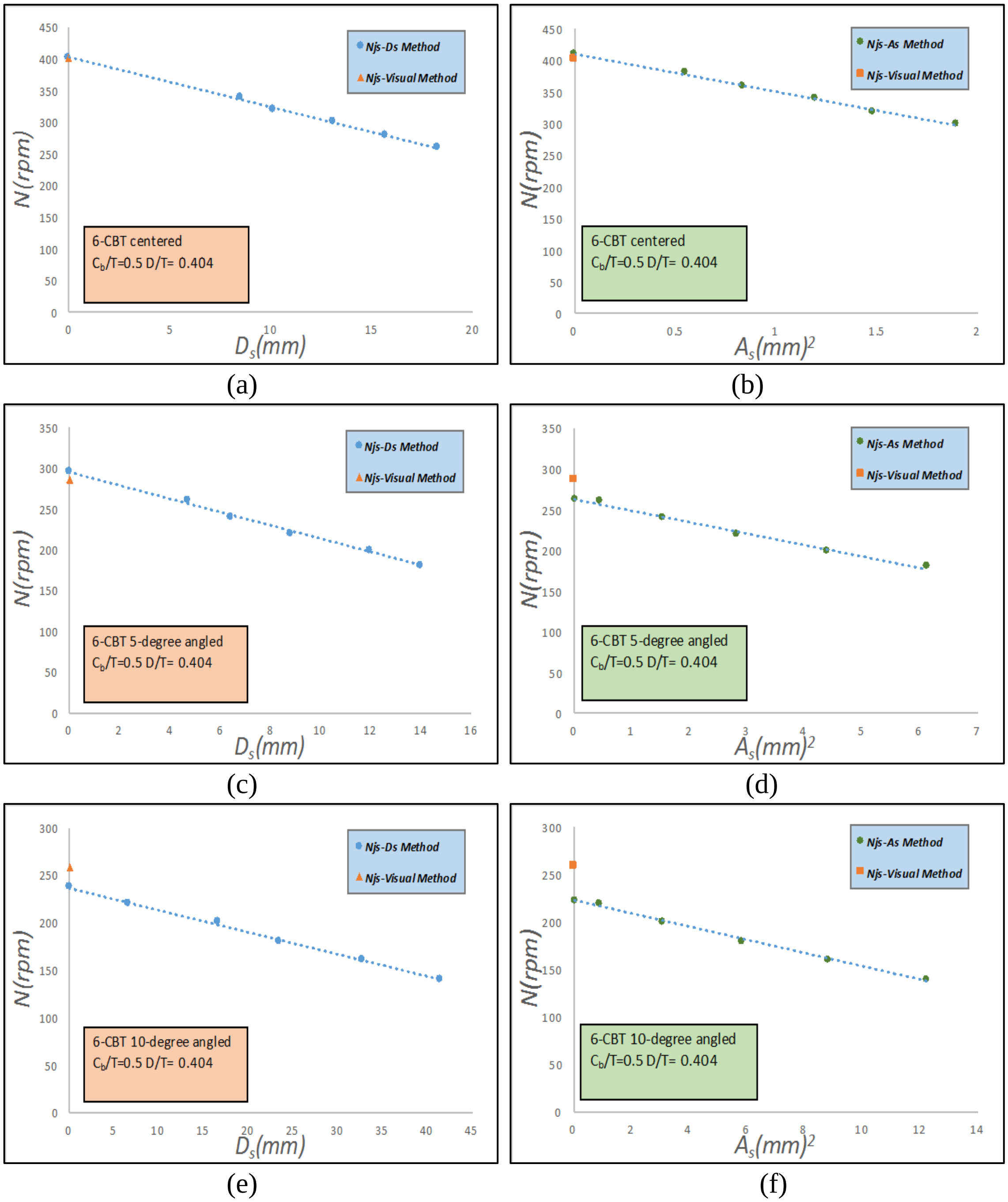


Figure 3.4 N_{js} measured the Hemispherical-bottomed tank with the 6-CBT impeller using for 200 µm particles: (a) $N_{js\text{-}Ds\text{-}Method}$ in centered system; (b) $N_{js\text{-}As\text{-}Method}$ in centered system; (c) $N_{js\text{-}Ds\text{-}Method}$ in 5-degree system; (d) $N_{js\text{-}As\text{-}Method}$ in 5-degree system; (e) $N_{js\text{-}Ds\text{-}Method}$ in 10-degree system; (f) $N_{js\text{-}As\text{-}Method}$ in 10-degree system.

3.1.2 N_{js} Results for Different Systems and Operation Conditions

The results for N_{js} which were obtained from the different angle-mounted impeller when the off-bottom ration (C_b/T) was varied from 0.2 (impeller off-bottom distance=32.1mm) to 0.5 (impeller off bottom distance=74mm) are presented in Table3.1 for 6-FBT impeller. This results shown that the $N_{js\text{-}Ds\text{-}Method}$ is much more appropriate than $N_{js\text{-}As\text{-}Method}$ to determine N_{js}. In the same impeller system, the impeller mounted in the centered N_{js} is much higher than impeller mounted with the angle. In addition, the 10-degree angled impeller mounted has a lower N_{js} than the 5-degree angle mounted impeller.

Additionally, the results for the other three types of impellers are shown in Table 3.2, Table 3.3 and Table 3.4. For different types of impeller, the the $N_{js\text{-}Ds\text{-}Method}$ appears to be appropriate while the $N_{js\text{-}As\text{-}Method}$ dose not work well, at least in this work.

Table 3.1 Result for N_{js} with 6-FBT, 200μm

Cb/T	Particle size(μm)	Impeller type	Angle(º)	$N_{js\text{-}Visual}$	$N_{js\text{-}Ds\text{-}Method}$	$N_{js\text{-}As\text{-}Method}$
0.5	200	6-FBT	0	389	393.58	370
0.4	200	6-FBT	0	322	326.08	323.24
0.3	200	6-FBT	0	318	318.73	318.73
0.25	200	6-FBT	0	312	280.6	277.72
0.2	200	6-FBT	0	307	295.52	273.93
0.5	200	6-FBT	5	260	266.49	283.26
0.4	200	6-FBT	5	248	257.96	268.33
0.3	200	6-FBT	5	230	242.71	261.07
0.25	200	6-FBT	5	224	223.41	249.06
0.2	200	6-FBT	5	242	306.47	283.17
0.5	200	6-FBT	10	228	226.08	269.95
0.4	200	6-FBT	10	216	217.96	236.65
0.3	200	6-FBT	10	211	223.56	234.5
0.25	200	6-FBT	10	252	258.41	236.46
0.2	200	6-FBT	10	260	264.39	242.17

Table 3.2 Result for N_{js} s with Lighting A310, 200μm

Cb/T	Particle size(μm)	Impeller type	Angle(°)	$N_{js\text{-}Visual}$	$N_{js\text{-}Ds\text{-}Method}$	$N_{js\text{-}As\text{-}Method}$
0.5	200	A310	0	411	403.64	368.87
0.4	200	A310	0	368	334.39	320.51
0.3	200	A310	0	340	311.79	310.15
0.25	200	A310	0	310	308	312.53
0.2	200	A310	0	302	305.89	298.05
0.5	200	A310	5	344	340.63	319.59
0.4	200	A310	5	329	326.92	333.16
0.3	200	A310	5	313	309	310.15
0.25	200	A310	5	302	306.74	285.86
0.2	200	A310	5	333	320.44	295.37
0.5	200	A310	10	326	328.58	310.17
0.4	200	A310	10	316	326.92	295.97
0.3	200	A310	10	300	298.98	279.68
0.25	200	A310	10	311	306.32	284.47
0.2	200	A310	10	318	308.67	309.08

Table 3.3 Result for N_{js} with DT Impeller, 200μm

Cb/T	Particle size(μm)	Impeller type	Angle(º)	$N_{js\text{-}Visual}$	$N_{js\text{-}Ds\text{-}Method}$	$N_{js\text{-}As\text{-}Method}$
0.5	200	DT	0	411	411.69	439.29
0.4	200	DT	0	409	411.06	387.27
0.3	200	DT	0	402	409.6	377.54
0.25	200	DT	0	381	374.86	392.24
0.2	200	DT	0	359	357.86	365.84
0.5	200	DT	5	358	374.85	310.21
0.4	200	DT	5	345	326.16	304.97
0.3	200	DT	5	335	317.99	296.48
0.25	200	DT	5	320	307.45	281.2
0.2	200	DT	5	338	339.77	366.15
0.5	200	DT	10	240	234.84	284.98
0.4	200	DT	10	215	200.91	252.36
0.3	200	DT	10	209	200.88	248.65
0.25	200	DT	10	237	260.97	240.76
0.2	200	DT	10	236	224	264.51

Table 3.4 Result for N_{js} with 6-CBT Impeller, 200μm

Cb/T	Particle size(μm)	Impeller type	Angle(°)	$N_{js\text{-}Visual}$	$N_{js\text{-}Ds\text{-}Method}$	$N_{js\text{-}As\text{-}Method}$
0.5	200	6-CBT	0	402	404.3	410.89
0.4	200	6-CBT	0	398	402.72	396.59
0.3	200	6-CBT	0	387	381.82	392.93
0.25	200	6-CBT	0	378	372.79	381.92
0.2	200	6-CBT	0	368	361.7	361.25
0.5	200	6-CBT	5	286	295.84	262.455
0.4	200	6-CBT	5	276	247.36	261.69
0.3	200	6-CBT	5	272	272.56	251.73
0.25	200	6-CBT	5	268	263.35	241.71
0.2	200	6-CBT	5	281	306.82	243.99
0.5	200	6-CBT	10	258	236.97	222.92
0.4	200	6-CBT	10	242	246.57	219
0.3	200	6-CBT	10	234	245.28	247
0.25	200	6-CBT	10	263	263.49	243.03
0.2	200	6-CBT	10	260	286.91	243.54

In order to better visualize these results, parity plots of $N_{js\text{-}visual}$ vs. $N_{js\text{-}Ds\text{-}Method}$. were obtained (Figure 3.5 for different impeller and Figure 3.6 (a)-(c)). An overall parity plot including all data is presented in the Figure 3.7. In all cases, one can see that the values of $N_{js\text{-}Ds\text{-}Method}$ agree well with those for $N_{js\text{-}Visual.}$ The R-value for all the points is 0.985

above. From this Figures, it can been seen that $N_{js\text{-}Ds\text{-}Method}$ is very close to N_{js}.

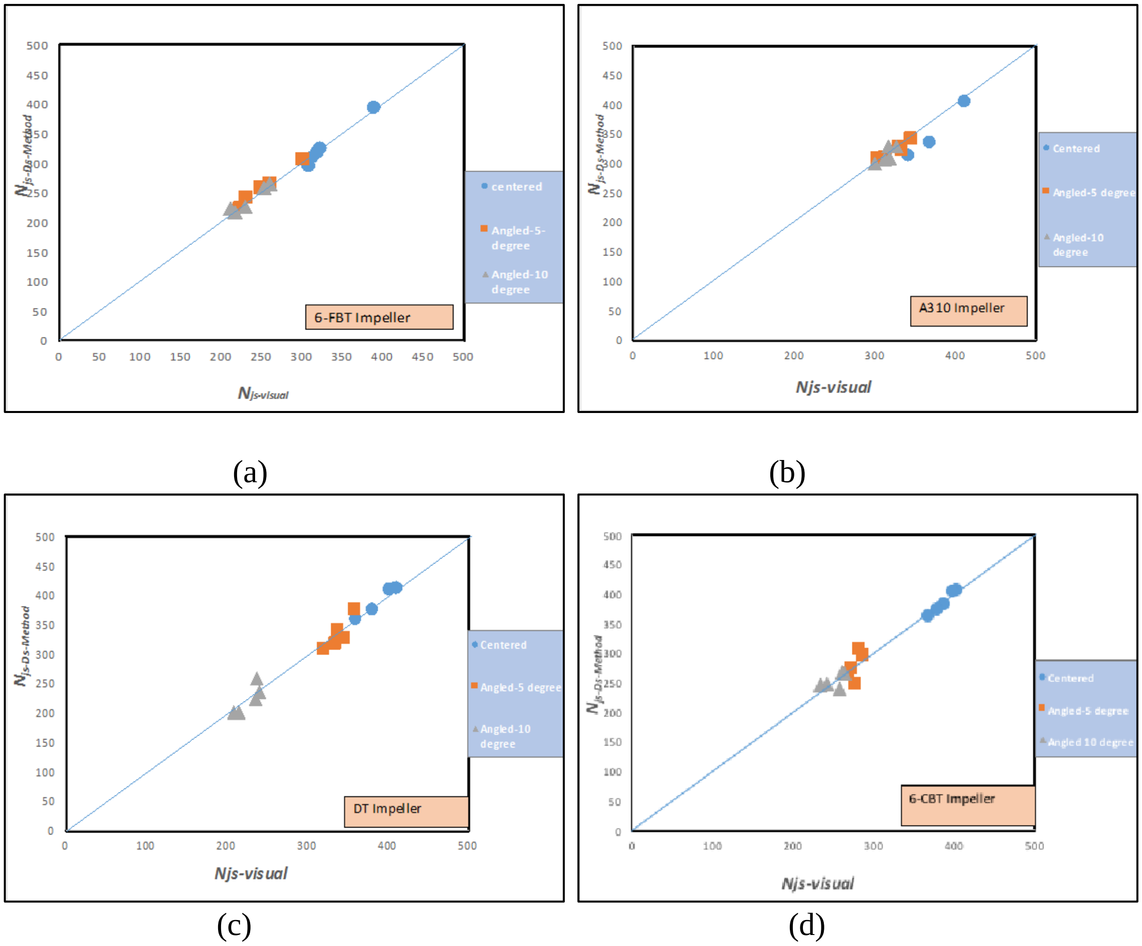


Figure 3.5 Parity plots of $N_{js\text{-}Ds\text{-}Method}$ (for C_b/T at 0.5 0.4 0.3 0.25 0.2) vs. $N_{js\text{-}Visual}$. (a) Parity plot for all 6-FBT impeller; (b) Parity plot for all A310 impeller mounted; (c) Parity plot for all DT impeller mounted; (d) Parity plot for all 6-CBT impeller mounted.

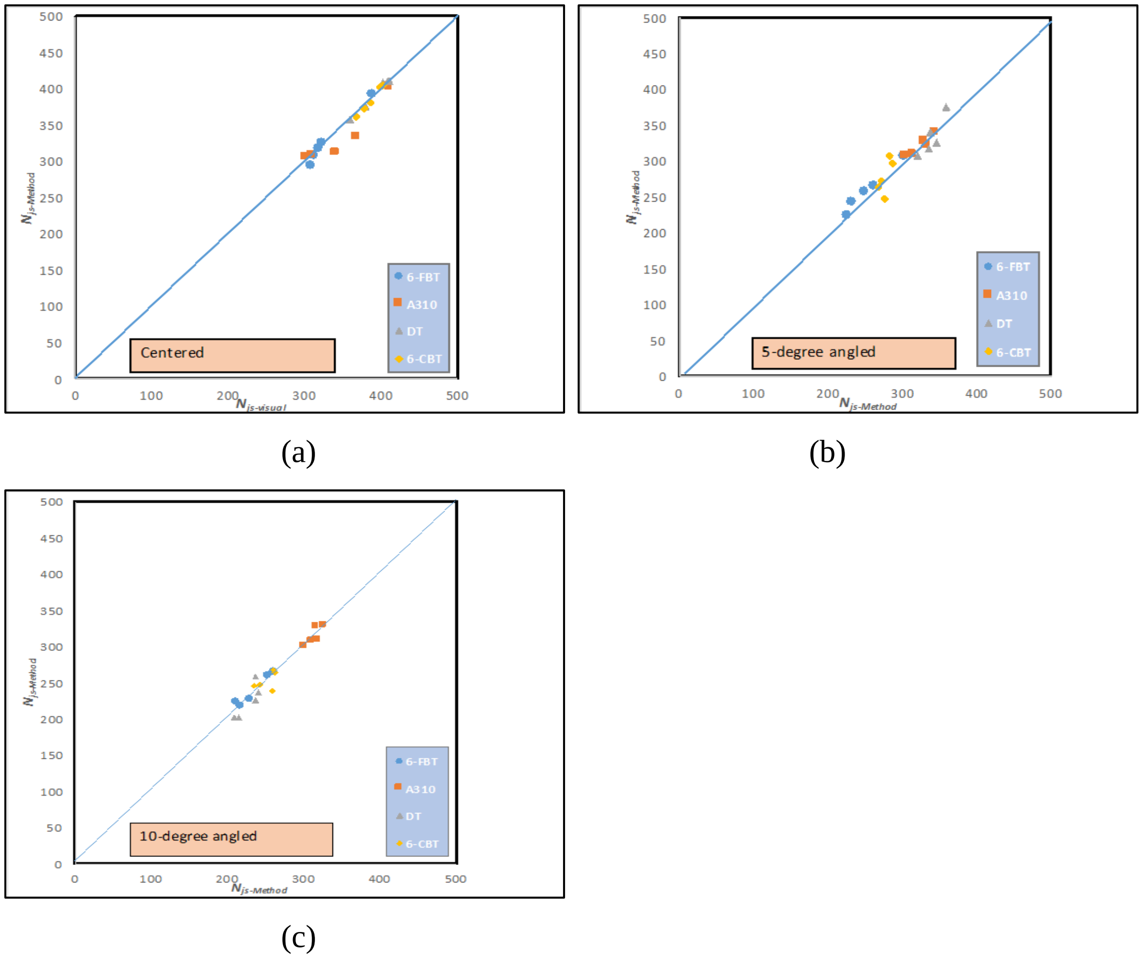


Figure 3.6 Parity plots of $N_{js\text{-}Ds\text{-}Method}$ (for C_b/T at 0.5 0.4 0.3 0.25 0.2) vs. $N_{js\text{-}Visual}$. (a) Parity plot for all centerline impeller mounted; (b) Parity plot for all 5-degree impeller mounted; (c) Parity plot for all 10-degree impeller mounted.

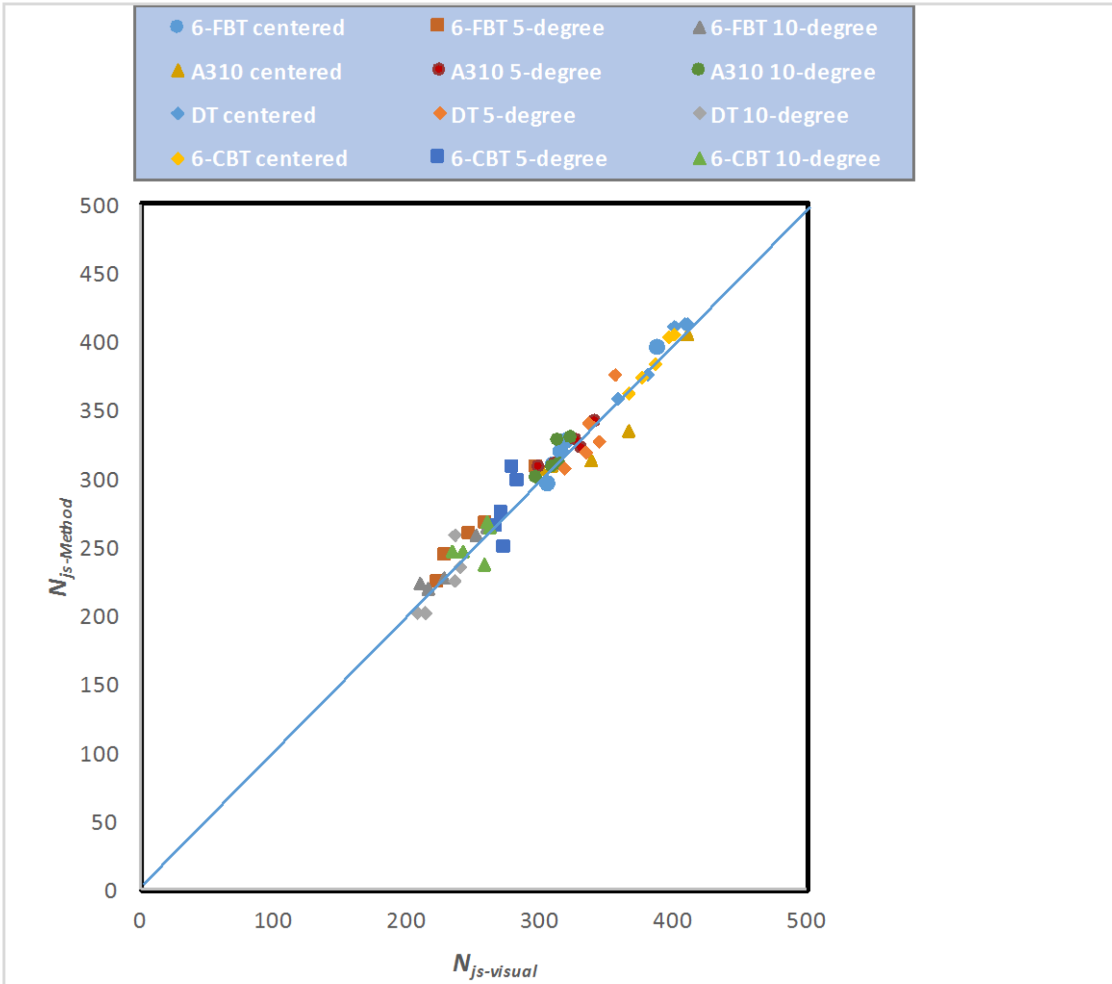
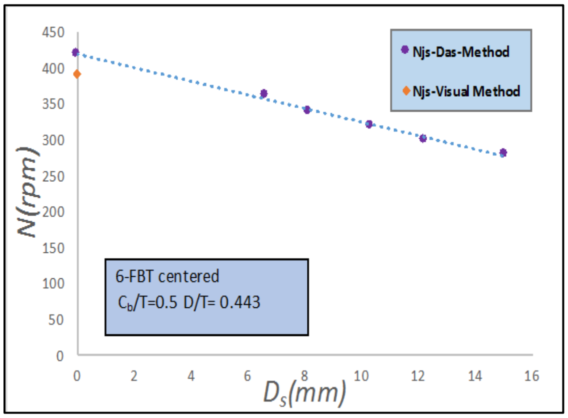


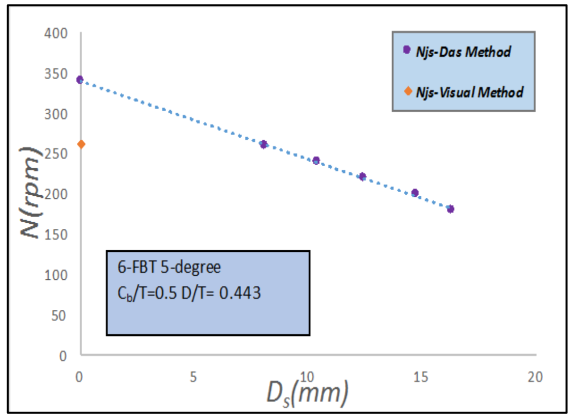
Figure 3.7 Parity plot for all data.

3.1.3 Results using $N_{js\text{-}DAS\text{-}Method}$

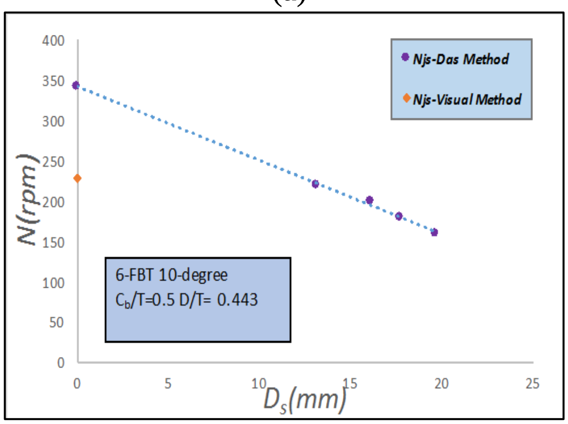
The results obtained using the $N_{js\text{-}DAS\text{-}Method}$ are shown in Figure 3.8. From this figure one can see that the results of this method are not very correct. Therefore, this approach was discarded for the determination of N_{js}. The other results are shown in APPENDIX.



(a)



(b)



(c)

Figure 3.8 $N_{js\text{-}DAS\text{-}Method}$ measured the Hemispherical-bottomed tank with the 6-FBT using for 200 m particles: (a) in centered system; (b) in 5-degree angled system; (c) in 10-degree angled system.

3.1.4 Comparison of the Effect of the Impeller Off-bottom Clearance Ration C_b/T on the Minimum Agitation Speed for Solid Suspension N_{js} for Different Impeller types

The values of N_{js} were experimentally obtained for different C_b/T ratios for different systems. The results are shown in Figure 3.8. For the 6-FBT impeller at different angles, in the centered system N_{js} is corresponding increased with C_b/T increased. A different phenomenon in 5-degree angled system, when C_b/T is < 0.2, the N_{js} increased with C_b/T decreased. Also, it is evident that N_{js} in the centered system is much higher than in the angled system. Figures (b) in Figure 3.9 showed that the when A310 impeller tested in C_b/T =0.25, N_{js} almost the same in different angled system. Figures (c) in Figure 3.9 is shown that the effect of C_b/T to N_{js} when the DT impeller were used. N_{js} of the 6-CBT impeller in 5-degree angled system and 10-degree angled system is almost the same when C_b/T =0.25 (Figures (d)). Moreover, in the 5-degree angled system N_{js} increased at C_b/T < 0.25 while in 10-degree angled system N_{js} decreased.

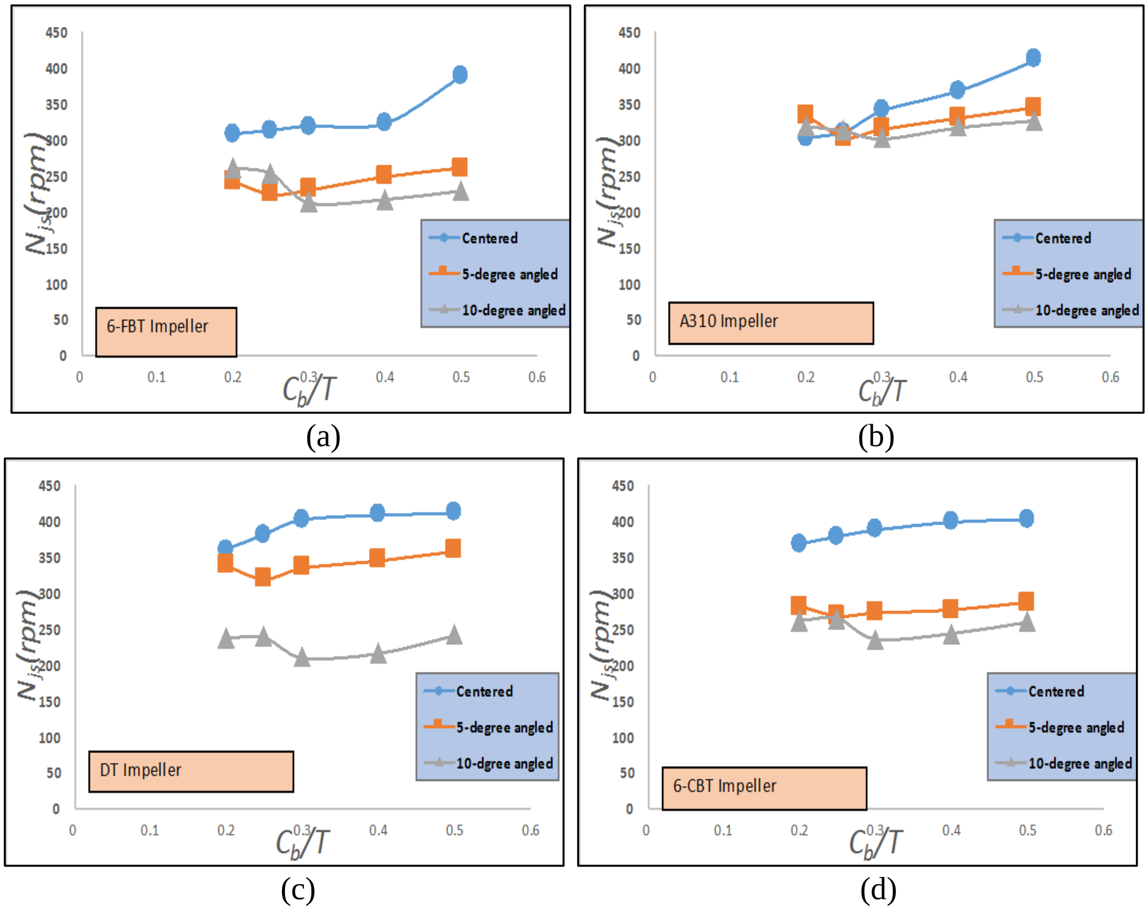
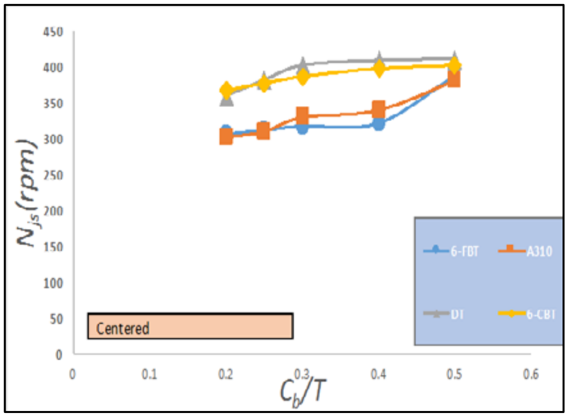


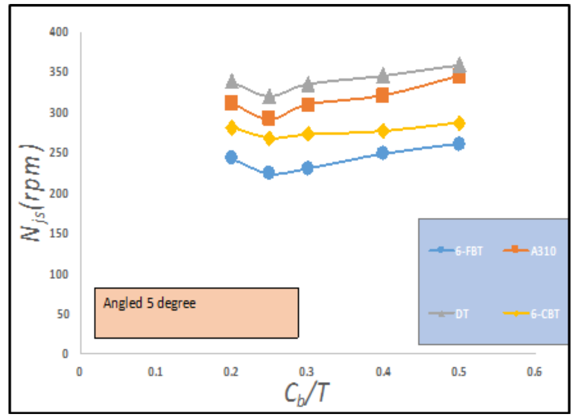
Figure 3.9 Effect of the Impeller Off-bottom Clearance Ratio C_b/T on N_{js} for different impeller types: (a) 6-FBT; (b) A310;(c) DT; (d) 6-CBT.

3.1.5 Comparison of the Effect of the Impeller Off-bottom Clearance Ration C_b/T on the Minimum Agitation Speed for Solid Suspension N_{js} for Different angled systems

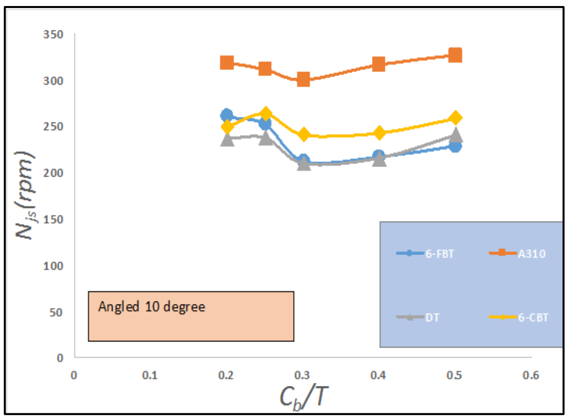
In this experiment, 4 types of impellers were used: 6-FBT, A310, DT and 6-CBT impeller. 6-FBT and DT impeller has same diameter, because the vessel did not change so they have same D/T=0.443. In the centered system (Figure 3.10(a)), the N_{js} of the DT impeller is higher than the other types of impellers. The same situation happened in A310 impeller and 6-PBT impeller when $C_b/T < 0.25$, the N_{js} of the 6-PBT impeller is higher than the A310 impeller. The different results came from 5-degree impeller mounted system (Figure 3.10(a),(b)). In the 5-degree angled system, N_{js} decreased when C_b/T <0.25. In pharmaceutical industry, DT impeller it is not commonly used. In 10-degree angled impeller mounted system which is shown in Figure3.10(c), the 6-CBT impeller and the 6-FBT impeller have a similar N_{js}. The change rate is not much.



(a)



(b)



(c)

Figure 3.10 Effect of the Impeller Off-bottom Clearance Ratio C_b/T on N_{js} for different angle-mounted impeller system: (a) Centered impeller mounted system; (b) 5-degree angled mounted system; (c) 10-degree angled mounted system.

3.2 S-Value for Zwietering Equation

The Zwietering Equation is:

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

The *S-value* was obtained here for all systems. In this equation, N_{js} was obtained from experiments. The S parameter was obtained by fitting the experimental N_{js} data to the Zwietering Equation for different systems.

$$s = \frac{D^{0.85}N_{js}}{\vartheta^{0.1}d_p^{0.5}\left(\frac{g\Delta\rho}{\rho L}\right)^{0.45}X^{0.13}}$$

From the literature, the effect of impeller clearance on N_{js} is typically presented by plotting N_{js} vs C_b/T or C_b/D. In this work, C_b/T was varied. Therefore, the data of this work is plotting by C_b/T vs *S-value* for the purpose of comparing different impellers mounted at different angles.

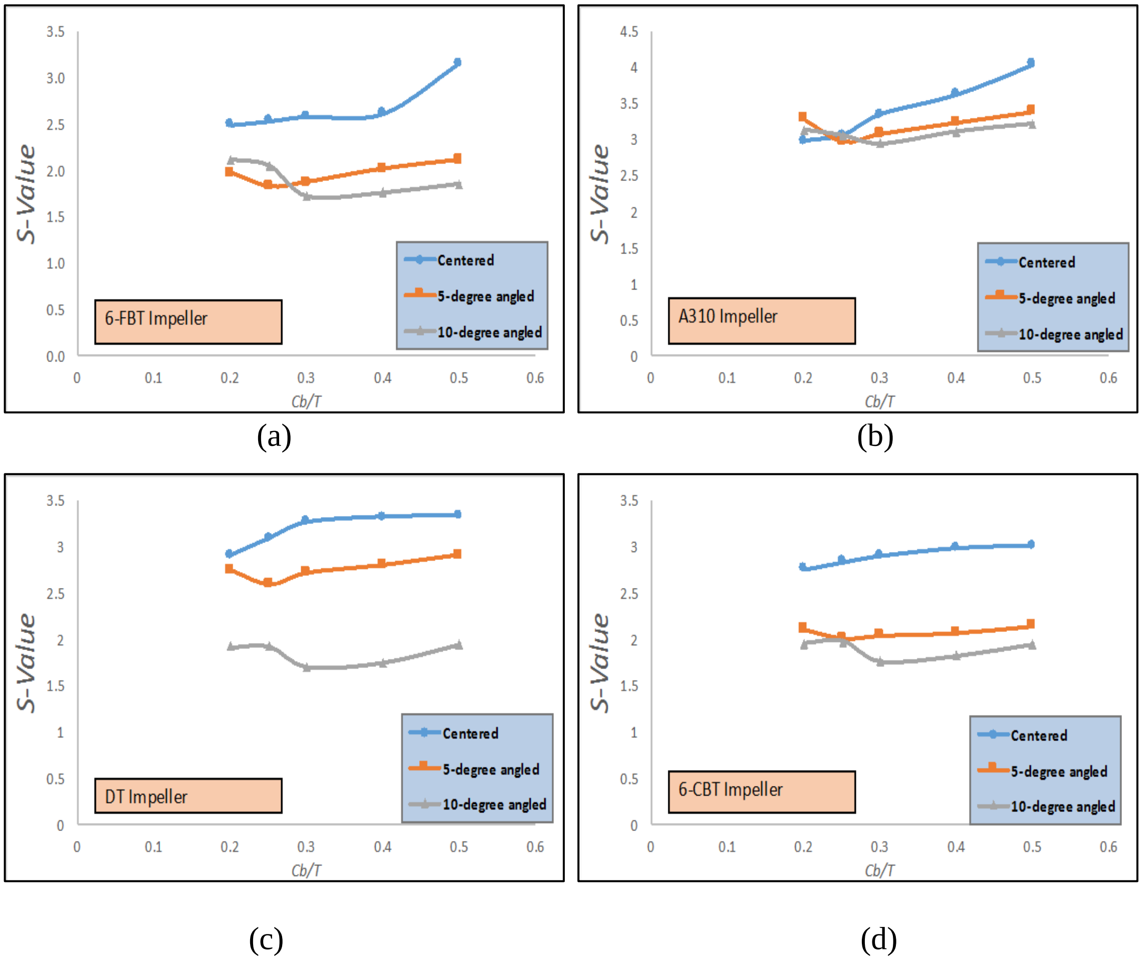


Figure 3.11 S-Value for *Zwietering* Equation. (a) 6-FBT Impeller; (b)A310 Impeller; (c)DT Impeller; (d) 6-CBP Impeller.

CHAPTER 4

CONCLUSION

In this work, N_{js} was obtained in a hemispherical bottomed vessel for a number of impeller types, impeller clearances, and impeller vertical positions. To do so a novel method was applied to the experimental determination of Njs, based on the experimental measurement of the diameter of the zone occupied by the solids at different agitation conditions. N_{js} values obtained with this approach (called $N_{js\text{-}Ds\text{-}Method}$) agree well with the visually determined values of N_{js}.

N_{js} was obtained for all systems investigated here. In general it was found that the value of N_{js} decreases when the impeller is placed at an angle rather than in a central position. Typically, N_{js} decreases when the C_b/T ratio decreases, but the N_{js} curve has a minimum at C_b/T =0.2.

The S-values in Zwietering's equation were also obtained for different C_b/T values, impellers types, and agitator configurations.

The results obtained in this work are directly applicable to the pharmaceutical industry to determine N_{js} in angle-mounted impeller system.

APPENDIX

Table A1 S-value For 6-FBT impeller

C_b/T	Particle size(μm)	Impeller type	Angle(°)	N_{js}-$_{A\sim D\text{-}method}$	S-value
0.5	200	6-FBT	0	419	3.15087
0.4	200	6-FBT	0	338.56	2.60817
0.3	200	6-FBT	0	378.21	2.57577
0.25	200	6-FBT	0	296.71	2.52718
0.2	200	6-FBT	0	317.4	2.48668
0.5	200	6-FBT	5	338.56	2.10598
0.4	200	6-FBT	5	305.25	2.00878
0.3	200	6-FBT	5	306.37	1.86298
0.25	200	6-FBT	5	335.15	1.81438
0.2	200	6-FBT	5	309.5	1.96018
0.5	200	6-FBT	10	342.82	1.84678
0.4	200	6-FBT	10	291.75	1.74958
0.3	200	6-FBT	10	306.37	1.70908
0.25	200	6-FBT	10	252.1	2.04118
0.2	200	6-FBT	10	280.16	2.10598

Table A2 S-value For A310 impeller

C_b/T	Particle size(μm)	Impeller type	Angle(º)	$N_{js\text{-}A\sim D\text{-}method}$	S-value
0.5	200	A310	0	489.4	4.03776
0.4	200	A310	0	396.64	3.61532
0.3	200	A310	0	373.13	3.34024
0.25	200	A310	0	376.91	3.04552
0.2	200	A310	0	302	2.96692
0.5	200	A310	5	329	3.37954
0.4	200	A310	5	319	3.23218
0.3	200	A310	5	309	3.07499
0.25	200	A310	5	303	2.96692
0.2	200	A310	5	310	3.27147
0.5	200	A310	10	320	3.20270
0.4	200	A310	10	313	3.10446
0.3	200	A310	10	300	2.94727
0.25	200	A310	10	305	3.05534
0.2	200	A310	10	314	3.12411

Table A3 S-value For DT impeller

C_b/T	Particle size(μm)	Impeller type	Angle(°)	$N_{js\text{-}A\sim D\text{-}method}$	S-value
0.5	200	DT	0	530.29	3.32907
0.4	200	DT	0	418.02	3.31287
0.3	200	DT	0	425.22	3.25617
0.25	200	DT	0	481.26	3.08607
0.2	200	DT	0	423.7	2.90787
0.5	200	DT	5	366.07	2.89977
0.4	200	DT	5	337.94	2.79447
0.3	200	DT	5	344.92	2.71347
0.25	200	DT	5	328.38	2.59197
0.2	200	DT	5	489.58	2.73777
0.5	200	DT	10	342.02	1.94398
0.4	200	DT	10	315.29	1.74148
0.3	200	DT	10	349.2	1.69288
0.25	200	DT	10	289.95	1.91968
0.2	200	DT	10	337.16	1.91158

Table A4 S-value For 6-CBT impeller

C_b/T	Particle size(μm)	Impeller type	Angle(º)	N_{js}-$_{A\sim D\text{-}method}$	S-value
0.5	200	6-CBT	0	474.84	3.01028
0.4	200	6-CBT	0	442.74	2.98033
0.3	200	6-CBT	0	500.03	2.89796
0.25	200	6-CBT	0	478.4	2.83056
0.2	200	6-CBT	0	455.31	2.75568
0.5	200	6-CBT	5	291.69	2.14164
0.4	200	6-CBT	5	315.06	2.06676
0.3	200	6-CBT	5	293.8	2.03681
0.25	200	6-CBT	5	289.61	2.00685
0.2	200	6-CBT	5	273.79	2.10420
0.5	200	6-CBT	10	252.28	1.93197
0.4	200	6-CBT	10	277.31	1.81216
0.3	200	6-CBT	10	270.2	1.75225
0.25	200	6-CBT	10	289.61	1.96941
0.2	200	6-CBT	10	261.4	1.94695

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