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SOLID SUSPENSION AND GAS DISPERSION IN MECHANICALLY AGITATED VESSELS

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DEDICATION

به نام بهترین دوست که هرچه دارم از اوست

I dedicate this thesis to Mania,

whose unconditional love has always supported me.

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I dedicate this thesis to my family and sincerely seek their blessing for the path ahead.

Rouzbeh JAFARI

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RÉSUMÉ

La conception et l'opération réussies de cuves agitées mécaniquement liquide-solide (LS) ou gaz-liquide-solide (GLS) requièrent la détermination précise du niveau adéquat de suspension solide qui est essentiel pour le procédé. Les ingénieurs et les scientifiques doivent définir des conditions géométriques et opératoires pour un milieu spécifique (propriétés physiques spécifiques) afin de fournir le niveau optimal de suspension solide. Ceci nécessite une connaissance approfondie de comment l'état de la suspension solide peut être influencé par des variations des paramètres physiques, opératoires et géométriques. De même, des corrélations empiriques ou des concepts théoriques précis sont nécessaires pour atteindre cet objectif. Le fait de ne pas concevoir la cuve agitée pour atteindre les conditions optimales et pour maintenir le système dans ces conditions durant l'opération peut amener des inconvénients significatifs concernant la qualité du produit (sélectivité et rendement) et le coût.

Cette étude implique un travail expérimental et théorique extensif sur la suspension et la dispersion du solide dans un système de mélange liquide-solide. Le système étudié a été une cuve agitée mécaniquement. En utilisant différentes techniques de mesure, comme la densitométrie aux rayons gamma et les fibres optiques, il a été possible d'obtenir des résultats très intéressants qui permettront d'améliorer la conception et la montée en échelle de systèmes de mélange liquide-solide.

Une revue de la littérature approfondie à propos de la suspension de solide en cuves agitées et des discussions détaillées avec des partenaires industriels nous ont menés à nous concentrer sur trois objectifs principaux pour améliorer la connaissance des systèmes de mélange liquide-solide denses :

1. Introduire une méthode prometteuse pour la caractérisation fine de la vitesse de suspension (Njs) dans un système de mélange liquide-solide à haute concentration.

2. Caractériser la suspension de solide et le degré d'homogénéité dans un système de mélange liquide-solide à haute concentration.
3. Évaluer la procédure de montée en échelle pour la suspension et la dispersion de solide dans une cuve agitée.

Le paramètre N_{js} a été caractérisé dans deux cuves agitées à des échelles du laboratoire différentes sur une large gamme de concentrations de solide (5-50 % wt/wt) pour trois types d'agitateurs à l'aide d'une nouvelle méthode de densitométrie aux rayons gamma. Les résultats ont été comparés avec une grande quantité de données collectées dans la littérature et obtenues à partir des corrélations empiriques publiées. L'effet du dégagement de l'agitateur et du débit de gaz a également été étudié. Les paramètres de la corrélation de Zwietering (Zwietering 1958) ont été modifiés pour permettre de réaliser une prédiction précise de N_{js}. En outre, un modèle semi-empirique a été proposé pour prédire N_{js}, sur base d'un bilan de quantité de mouvement global pour les particules reposant au fond de la cuve. Un nouveau modèle et des corrélations empiriques modifiées ont été comparées aux données de la littérature (lorsque possible) et la précision de la nouvelle technique a été évidente. Il a été démontré que les agitateurs axiaux sont plus avantageux pour suspendre les solides, mais qu'à partir d'un certain dégagement l'agitateur axial devient totalement inapproprié. L'effet des conditions opératoires, du dégagement de l'agitateur et de la concentration de solide sur la dispersion de solide et sur la hauteur du nuage a également été étudié. Des études de montée en échelle ont également été menées pour évaluer les différentes techniques de montée en échelle pour N_{js} et pour la dispersion de solide dans les cuves agitées.

Cette thèse présente également un travail expérimental pour évaluer les procédures de montée en échelle dans les systèmes de mélange gaz-liquide (GL) et gaz-liquide-solide (GLS). Le système étudié était une cuve agitée mécaniquement pour laquelle l'agitation était réalisée par différents types d'agitateurs. L'aérateur était de type annulaire situé en-dessous de l'agitateur. Des expériences ont

été réalisées pour différents débits de gaz, vitesses d'agitateur et pour deux échelles différentes afin de caractériser le coefficient de transfert gaz-liquide (k_La). La partie principale du travail a été consacrée à évaluer la procédure de montée en échelle dans les systèmes GL et GLS. D'abord, la contribution des échelles de mélange pour différentes conditions opératoires (vitesse d'agitateur et débit de gaz) ont été déterminées. Dans une cuve agitée, deux échelles de mélange peuvent être déterminées (macro-mélange et micro-mélange). Les données expérimentales ont été comparées avec les prédictions théoriques (en utilisant le concept de micro-mélange et de macro-mélange) et il a été démontré que le micro-mélange joue un rôle important dans le transfert de matière gaz-liquide. En outre, différentes procédures de montée en échelle ont été évaluées pour identifier les changements du k_La durant la montée en échelle. Les résultats ont montré une différence significative entre le k_La dans une petite cuve et dans une large cuve pour différentes procédures de montée en échelle. Pour décrire ces différences, la distribution de k_La dans une cuve et l'effet de la procédure de montée en échelle sur cette distribution ont également été caractérisées en utilisant des données locales obtenues à l'aide de mesures par fibre optique (pour déterminer le hold-up de gaz et la taille des bulles). Les résultats indiquent qu'une montée en échelle réussie peut être réalisée en considérant la distribution de k_La dans une cuve agitée, ce qui nécessite des mesures ou des prédictions locales détaillées de k_La.

ABSTRACT

The successful design and operation of liquid-solid (LS) and gas-liquid-solid (GLS) mechanically agitated vessels require the accurate determination of the proper level of solid suspension that is essential for the process at hand. Engineers and scientists must define geometrical and operating conditions for a specific medium (specified physical properties) in such a way that provides the optimum level of solid suspension. This requires comprehensive knowledge about how the state of solid suspension may be affected by changing physical, operational, and geometrical parameters. Also, accurate empirical correlations or theoretical concepts are necessary to fulfill that objective. Failure to design the agitated vessel to achieve optimum conditions and maintain the system at these conditions during operation may cause significant drawbacks concerning product quality (selectivity and yield) and cost.

This research involves extensive experimental and theoretical work on solid suspension and dispersion in a liquid-solid mixing system. The system under study was a mechanically agitated vessel. By using different measurement methods, i.e., Gamma Ray Densitometry, and Optical Fibre, attention-grabbing results have been obtained, which will help to improve the design and scale-up of liquid-solid mixing systems.

A thorough literature survey on solid suspension in agitated tanks and comprehensive discussions with industrial partners led us to focus on three major objectives to improve our knowledge of dense liquid-solid mixing systems:

1. To introduce a promising method for accurate characterizing just off-bottom suspension speed (Njs) in high concentration liquid-solid mixing system.
2. To characterize solid suspension and the degree of homogeneity in a high concentration liquid-solid mixing system.

3. To evaluate the scale-up procedure for solid suspension and dispersion in stirred tanks.

N_{js} was characterized in two laboratory-scale agitated vessels over a wide range of solid concentrations (5-50% wt/wt) for three types of impellers with a new gamma ray densitometry method. Results were compared with a wide range of data collected from the open literature and published empirical correlations. The effect of impeller clearance and gas flow rate were also studied. Parameters of the Zwietering (Zwietering 1958) correlation were modified to be able to make a precise prediction of N_{js}. In addition, a semi-theoretical model proposed for predicting N_{js} based on general momentum balance for particles resting at the bottom of the vessel. A new model and modified empirical correlations were compared with literature data (whenever possible) and the accuracy of the new technique was evident. It was shown that axial flow impellers are more advantageous for suspending solids, but at certain impeller clearances the axial flow impeller becomes totally inappropriate. The effect of operating conditions, impeller clearance and solid concentration on solid dispersion and cloud height were also studied. Scale-up studies have also been carried out to evaluate different scale-up concept techniques for N_{js} and solid dispersion in agitated vessels.

This thesis also examines experimental work on evaluating scale-up procedures in gas-liquid (GL) and gas-liquid-solid (GLS) mixing systems. The system under study was a mechanically agitated vessel for which agitation was provided by different types of impellers. The sparger was a ring type located underneath the impeller. Experiments were carried out for various gas flow rates, impeller speeds and two different scales to characterize the gas-liquid mass transfer coefficient (k_La). The main part of the work was dedicated to evaluating the scale-up procedure in GL and GLS systems. First of all, the contribution of scales of mixing for different operating conditions (impeller speed and gas flow rate) was determined. In an agitated vessel two scales of mixing can be determined (macro-mixing and micro-mixing). The experimental data were compared with the theoretical predictions (using micro-mixing and macro-mixing concept) and it was demonstrated that micro-mixing plays an important role in gas-liquid mass transfer. In addition, different scale-up procedures were evaluated to identify the k_La changes during scale-up. Results show significant differences between k_La in small and large vessels for different scale-up procedures. To describe these differences the distribution of k_La in a vessel and the effect of the scale-up procedure on this distribution also was characterized using local data gathered from fibre optic measurements (to define gas hold-up and bubble size). Results indicate that a successful scale-up can be done by considering k_La distribution in an agitated vessel, which requires detailed local k_La measurements or predictions.

CONDENSÉ EN FRANÇAIS

Tous les procédés chimiques industriels sont développés pour transformer les matières premières bon marché en produits de qualité avec une valeur ajoutée. Habituellement, les réacteurs chimiques sont employés pour atteindre ce but. Un réacteur, dans lequel de telles transformations chimiques ont lieu, doit avoir plusieurs fonctions comme mettre les réactifs en contact intime pour laisser se produire des réactions chimiques et fournir les conditions appropriées (température, pression, etc.) pendant un temps suffisant. Les réacteurs de type cuve agitée, dans lesquels une ou plusieurs turbines sont utilisées pour réaliser le mélange, sont employés couramment dans une grande variété d'industries de transformation. Lorsque les cuves agitées satisfont à certaines des conditions suivantes les ingénieurs préfèrent les employer selon leurs avantages à des fins industrielles plutôt que les autres contacteurs tels que la colonne à bulle:

- L'écoulement de gaz est grand par rapport à l'écoulement liquide
- Le bon transfert de masse pour des gaz à faible solubilité des gaz est exigé
- Le bon transfert de chaleur est exigé (pour des réactions exothermiques ou endothermiques élevées)
- La phase liquide est fortement visqueuse ou des liquides non newtoniens sont traités
- Les solides doivent être suspendus ou dispersés.

L'inconvénient le plus important de la cuve agitée reste la complexité mécanique de sa construction en comparaison avec les autres types de réacteurs. L'axe et la turbine présentent leurs propres difficultés opérationnelles, particulièrement sous des conditions extrêmes (haute pression, concentration élevée des solides, matériaux toxiques). Les cuves agitées peuvent également présenter des difficultés pour leur opération à grande échelle à cause de la taille des unités de dérivation et des pièces en rotation. Elles exigent également une énorme quantité d'énergie.

Une meilleure compréhension fondamentale de l'hydrodynamique, de la dynamique des fluides et du mécanisme de mélange dans de tels systèmes permettrait une conception plus efficace et plus compacte. En outre, la montée en échelle de tels systèmes a toujours été un des plus grands défis pour les ingénieurs et l'obtention d'informations détaillées au sujet leur hydrodynamique aidera à traiter ces défis efficacement.

En raison de l'hydrodynamique complexe produit par la rotation de la turbine, l'opération des cuves agitées demeure un problème entier. La conception et le fonctionnement exacts de ces réacteurs peuvent être décisifs pour la rentabilité du processus en vertu de son influence sur le rendement ou la productivité des réactions. Traditionnellement, ceci est basé sur des corrélations empiriques décrivant des paramètres macroscopiques, tels que la demande d'énergie, la masse et le coefficient de transmission de chaleur global ou la rétention de solide dans la phase dispersée. Beaucoup d'études ont été publiées pour décrire ces paramètres principaux en fonction de variables de fonctionnement et de conception, telles que la vitesse de la turbine, la géométrie de la cuve, le diamètre de la turbine, la hauteur du liquide et le nombre de turbines. Il n'est pas rare qu'un procédé réussi à l'échelle de laboratoire ne fournisse pas l'exécution attendue à l'échelle industrielle. La cause d'un tel échec est un couplage inadapté entre les phénomènes qui ont lieu dans les systèmes de mélange et les phénomènes de transferts à l'échelle industrielle.Un bon exemple de ceci est le procédé de cyanuration d'or. La cyanuration est le procédé le plus commun pour extraire l'or à partir de son minerai. L'or est extrait à partir de son minerai par la réaction entre l'or et le cyanure en présence d'oxygène. C'est une réaction triphasée où l'oxygène doit être dissout dans la phase liquide. Ensuite l'oxygène dissout et le cyanure doivent être transférés à la surface du solide et, finalement, la réaction a lieu dans/sur les particules solide.

La chimie de la réaction de cyanuration de l'or est très complexe. Des détails au sujet du procédé de cyanuration et des théories cinétiques des réactions sont discutés dans l'annexe A. Dans la présente partie nous nous concentrons seulement sur le procédé du point de vue de la conception. Le type de réacteur utilisé dans ce procédé est une cuve mécaniquement agitée. Afin de réaliser une récupération élevée d'or plusieurs cuves agitées sont employés. Généralement 8 à 10 réacteurs sont utilisés. Les réacteurs de cyanuration souffrent fréquemment de quelques inconvénients généraux dont les principaux sont 1) une consommation élevée des réactifs (cyanure et oxygène), 2) un temps de séjour élevé et 3) la sélectivité du produit.

La synthèse primaire sur ce procédé (pour quelques usines industrielles) spécifie que la quantité de cyanure et d'oxygène consommés dans ce procédé est très haute en comparaison de ce qui est théoriquement exigé. Cette valeur est de 600 fois plus de cyanure et 6000 fois plus d'oxygène pour une tonne d'or. La conséquence de ce taux élevé de consommation de réactif est le coût élevé de matériel et d'opération. Comme mentionné ci-dessus, la réaction de cyanuration est une réaction triphasée et il y a différents phénomènes qui contribuent à réaliser la réaction, comme le transfert de masse de réactif à l'emplacement de réaction. La cyanuration d'or elle-même est une réaction très rapide. Ainsi l'étape de contrôle dans la réaction est le taux de transfert de masse, ce qui pourrait être le taux de transfert de masse liquide-solide ou gaz-liquide. Le long temps de séjour exigé pour réaliser la récupération élevée de l'or est un fait important pour confirmer ceci. En outre le contenu de l'or dans le minerai est très bas (1-20ppm) et dans certains cas il est enfermé dans la matrice forte d'autres minerais. Puisque le cyanure peut réagir avec tous les minerais, dans le minerai la sélectivité de la réaction désirée est basse et habituellement les produits finis contiennent une quantité importante de cuivre ou d'autres minerais ce qui exige un processus additionnel de purification.

De nombreuses études de recherche au sujet de la cyanuration d'or ont été faites pour comprendre le mécanisme de réaction et tout autre phénomène chimique qui a lieu pendant la réaction. Malgré tous ces efforts aucune amélioration significative de tels procédés n’a été réalisée. Bien que quelques réacteurs originaux aient été présentés où le cyanure a été remplacé par d'autres matériaux de extraction, la cyanuration dans la cuve agitée reste toujours le choix le plus approprié du point de vue industriel.

Pour identifier les problèmes associés au procédé de cyanuration, quelques essais primaires ont été effectués. L'objectif était de déterminer si les conditions de fonctionnement ont un effet sur le procédé de cyanuration ou pas. À cette fin la réaction de cyanuration a été effectuée dans le réacteur au laboratoire (T=0.4, H=0.4). Différents paramètres ont été changés comme le type de mélangeur, la vitesse, le débit de gaz et le type d'aérateur. Les résultats ont prouvé que dans tous les cas le procédé s'est amélioré de manière significative. Par exemple, la vitesse croissante de la turbine a diminué le temps de séjour exigé de 50% et la consommation cyanurée de 30%. Sur base de ces résultats on a conclu que la technologie courante pour le réacteur de cyanuration souffre de mauvaises conditions de mélange. Pour surmonter ce problème et fournir des meilleures conditions de mélange il était nécessaire de remodeler la cuve agitée et de définir correctement des conditions de fonctionnement.

La réussite du procédé dépend du choix approprié de l'équipement et de définition adéquate des conditions de fonctionnement. Des conditions géométriques et opérationnelles doivent être définies de telle manière qu'elles fournissent les conditions optimales de réaction. Ceci exige une connaissance complète de la façon dont le procédé peut être affecté par la variation des paramètres physiques, opérationnels et géométriques. Le fait de fonctionner en dehors des conditions optimales mène à des inconvénients considérables. Le manque de méthodes expérimentales précises et de données exactes sur la suspension des solides et sur la dispersion de gaz ainsi que les incertitudes associées avec les procédures de montée à l'échelle industrielle ont fourni une bonne motivation pour réaliser cette recherche. La conception et le fonctionnement réussis des cuves mécaniquement agitées de LS et de GLS exigent la détermination précise du niveau de la suspension des solides qui est essentiel pour le proécédé actuel. Les ingénieurs et les scientifiques doivent définir des conditions géométriques et de fonctionnement pour un milieu spécifique (propriétés physiques spécifiques) de telle manière qu'il fournisse le niveau optimum de la suspension des solides. Ceci exige une connaissance complète au sujet de la façon dont l'état de suspension des solides peut être affectée par la variation des paramètres physiques, opérationnels et géométriques. En outre, des corrélations empiriques précises ou des concepts théoriques sont nécessaires pour atteindre cet objectif. Le fait de ne pas concevoir la cuve agitée pour réaliser des conditions optimales et pour maintenir le système à ces conditions lors du fonctionnement peut causer des inconvénients importants en ce qui concerne la qualité du produit (sélectivité et rendement) et les coûts.

Une recherche bibliographique complète sur la suspension des solides dans les réservoirs agités et des discussions approfondies avec les associés industriels nous ont menés à nous concentrer sur quatre objectifs importants pour améliorer la connaissance des systèmes de mélange liquide-solides denses :

1. Présenter une méthode prometteuse pour caractériser la vitesse de la suspension des solides (N_{js}) et sa caractérisation.
2. Caractériser la suspension des solides et le degré de l'homogénéité dans une cuve liquide-solides agitée.
3. Evaluer le procédé de montée à l'échelle industrielle pour la suspension des solides et la dispersion dans les réservoirs agités.

Le paramètre Njs a été caractérisé dans deux cuves agitées de laboratoire à diverses concentrations de solides (5-50% wt/wt) avec une nouvelle méthode de densitomètrie au rayons gamma pour trois types de turbine. Les résultats ont été comparés à un éventail de données rassemblées dans la littérature et de corrélations empiriques publiées. L'effet du dégagement du mélangeur et du débit de gaz ont été également étudiés. Des paramètres de la corrélation de Zwietering (Zwietering 1958) ont été modifiés pour pouvoir faire une prévision précise de Njs. En outre, un modèle théorique a été proposé pour prédire Njs en se basant sur un bilan de quantité de mouvement global pour les particules reposant au fond de la cuve. Le nouveau modèle et les corrélations empiriques modifiées ont été comparés aux données de littérature (autant que possible) et l'exactitude de la nouvelle technique était évidente. Il a été démontré que les mélangeurs axiaux sont plus avantageux pour suspendre des solides, mais à certains dégagements, le mélangeur axial devient totalement inadéquat. L'effet des conditions de fonctionnement, du dégagement de la turbine et de la concentration des solides sur la dispersion des solides et sur la hauteur du nuage a également été étudié. Des études sur le transfert du procédé à l'échelle industrielle ont également été effectuées pour évaluer différentes techniques de conception pour le transfert à l'échelle industrielle de Njs et de la dispersion des solides dans les cuves agitées.

Cette recherche présente également un travail expérimental sur l'évaluation des procédures de transfert à l'échelle industrielle dans les systèmes de mélange gaz-liquides (GL). Le système à l'étude était une cuve mécaniquement agitée pour laquelle l'agitation a été donnée par différents types de mélangeur. L'aérateur est un type d'anneau situé sous la turbine. Des expériences ont été effectuées pour différents débits de gaz. Le coefficient de transfert de masse gaz-liquide (kLa), a été caractérisé. La partie principale du travail a été consacrée à l'évaluation du procédé de transfert à l'échelle industrielle pour les systèmes de GL. Tout d'abord, la contribution des échelles de mélange pour différentes conditions de fonctionnement a été déterminée. Dans une cuve agitée, deux échelles de mélange peuvent être déterminées (macro-mélange et micro-mélange) et le kLa est théoriquement prédit comme si chacune d'elles était responsable de la dispersion de gaz. Les données expérimentales ont été comparées aux prévisions théoriques et il a été démontré que le micro-mélange joue un rôle important dans le transfert de masse gaz-liquide. En plus, différentes procédures de transfert à l'échelle industrielle ont été évaluées pour identifier les changements de k_La pendant le transfert à l'échelle industrielle. Les résultats montrent des différences importantes entre le $k_L a$ dans de petites et grandes cuves pour différentes procédures de transfert à l'échelle industrielle. Pour décrire ces différences la distribution du $k_L a$ dans une cuve et l'effet du procédé de transfert à l'échelle industrielle sur cette distribution ont été caractérisés aussi en utilisant des données locales recueillies par des mesures à fibres optiques (pour définir le holdup de gaz et la taille de bulle). Les résultats indiquent qu'un transfert à l'échelle industrielle réussi peut être fait en considérant la distribution de $k_L a$ dans une cuve agitée, ce qui exige des mesures ou des prévisions locales détaillées de $k_L a$.

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a	*Constant of equation 5-3 (-), Specific area (1/m) chapter 7*
A	*Surface area (m^2), Attenuation of vessel and environment (-)*
A_p	*Particle surface area (m^2)*
b	*Constant of equation 5-3 (-)*
C	*Impeller clearance (m), or solid concentration (kg/m^3)*
C_D	*Drag coefficient (-)*
C_{O2}	*Oxygen concentration in liquid phase (mg/l)*
D	*Impeller diameter (m)*
D_{ab}	*Diffusivity (m^2/s)*
Ds	*Sparger diameter (m)*
d_b	*Bubble size (m)*
do	*Orifice diameter (m)*
dp	*Mean particle diameter (m)*
fl	*Gas flow number: Q/ND^3 (-)*
Fr	*Impeller Froude number (N^2D/g)*
g	*Gravity acceleration (m/s^2)*
H	*Liquid height (m)*
I	*Count rate (count/sec)*
k_L	*Mass transfer coefficient in liquid phase (m/s)*
k_La	*Gas-liquid mass transfer coefficient (1/s)*
L	*Scanning length (m)*
M	*Mass of solid or liquid (kg)*

n	*Parameter equation 6-2*
N	*Impeller speed (1/sec)*
N_{js}	*Impeller speed (1/sec) at just suspended condition for LS system*
N_{jsg}	*Impeller speed (1/sec) at just suspended condition for GLS system*
N_{CD}	*Critical impeller speed for complete gas dispersion (rpm)*
Np	*Impeller Power number (-)*
P	*Power dissipation in Chapter 7 (W), Pressure in chapter 5 (Pa)*
P_g	*Aerated power input (W)*
Q_g	*Gas flow rate (m^3/s)*
r	*Local radial position (m)*
R	*Vessel radius (m)*
Re	*Reynolds number $\rho ND^2/\mu$(-)*
RSD	*Relative standard deviation (-)*
S	*Zwietering correlation constant (-)*
t	*Time (s)*
T	*Vessel diameter (m)*
t_c	*Circulation time (sec)*
U_{core}	*Core velocity of the jet (m/s)*
v or v_s	*Gas superficial velocity (m/s)*
$v_{l\text{-}js}$	*Liquid velocity at the bottom of the vessel at just suspended condition*
V	*Volume (m^3)*
Vp	*Particle volume (m^3)*
v_s	*Superficial gas velocity (m/s)*
W	*Blade width (m)*
X	*Solid loading Ms/Mt×100 (% wt/wt-),*

	or Mass of unsuspended solid (Chap. 2)
z	*Local axial position (m)*

Greek letters

$\alpha, \beta, \gamma, \delta, \theta$	*Constants of equation 5-3*
μ	*Mass attenuation coefficient (kg/m²)*
ν	*Liquid kinematic viscosity (m²/s)*
ρ	*density (kg/m³)*
ε	*Solid hold-up or volume fraction (-), Chapter 5*
ε_g	*gas hold-up (-)*
ε_s	*Solid hold-up (-)*
$\varepsilon_{s,0}$	*Solid hold-up for fully settled bed (-)*
ε_{ave}	*Specific power (w/kg) in chapter 7, average solid hold-up in chapter 6 (-)*
ΔN_{js}	N_{jsg}-N_{js}
φ	*Gas volume fraction (-)*
σ	*Surface tension (N.m)*
θ	*Mixing time (s)*

Subscribes

0	*Initial condition (N=0 rpm)*
ave	*average*
i	*Impeller, in Chapter 5 represents scanning section*
imp	*Impeller*
js	*Just suspended*

jsg	*Just suspended in gassed condition*
l	*Liquid*
ls	*Slurry*
s	*solid*

Abbreviations

CBT	*Concave Blade Turbine*
PBT-D (n)	*Pitched blade turbine down pumping flow (number of blades)*
PBT-U (n)	*Pitched blade turbine up pumping flow (number of blades)*
DT (n)	*Disk turbine (number of blades)*
GLS	*Gas-liquid-solid system*
LS	*Liquid-solid system*
MFU	*Mixed flow impeller, up-pumping*
MFD	*Mixed flow impeller, Down-pumping*
RT (n)	*Rushton turbine (number of blades)*

INTRODUCTION

All industrial chemical processes are developed to transform cheap raw materials into high value products. This goal is usually achieved using chemical reactors. A reactor in which such chemical transformations take place has to carry out several functions, such as bringing the reactant into intimate contact to let chemical reactions occur and providing an appropriate environment (temperature, pressure, etc.) for an adequate length of time. Stirred tank reactors in which one or more impellers are used to provide mixing within the vessel are widely used in a variety of process industries. When some of the following conditions are met, depending on their advantages, agitated vessels (stirred tanks) are often chosen by engineers for industrial purposes instead of other types of contactors, like a bubble column or packed bed reactors:

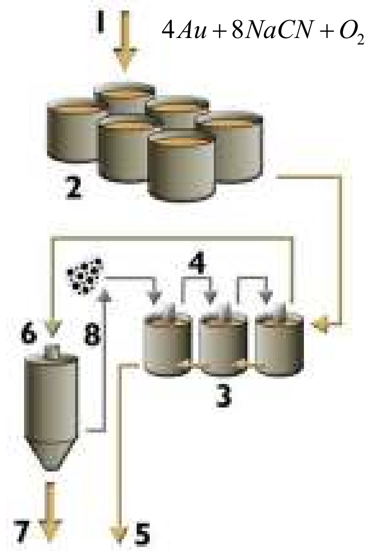
- Gas flow is greater compared to liquid flow
- Good mass transfer for low solubility gases is required
- Good heat transfer is required (for high exothermic or endothermic reactions)
- Liquid phase is highly viscous or non-Newtonian liquids are required to be processed
- Solids are required and need to be suspended or dispersed

The most vital drawback of the agitated vessel is its mechanical complexity of construction in comparison with other types of reactors. The rotating shaft and impeller present their own difficulties from an operational perspective, especially while operating under extreme conditions (high pressure, high solid concentration, toxic materials). Agitated vessels can also present difficulties on a very large scale operation due to the size of the units and rotating parts. An increased understanding of the hydrodynamics in such systems would allow a more compact and efficient design. In addition, scale-up has always been a great challenge for engineers. Detailed information about the scale effect on the hydrodynamic of the systems will help to deal with those challenges effectively.

The correct design and operation of these reactors can be crucial to the profitability of the process by virtue of its influence on the reaction yield or productivity. Traditionally this is based on empirical correlations describing macroscopic parameters, such as power demand, overall mass and heat transfer coefficient or dispersed phase hold-up. Many studies have been published to describe those key parameters as a function of operational and design variables, such as impeller speed, vessel geometry, the diameter of the impeller, liquid height and the number of impellers. It is not uncommon that a successful process on a laboratory scale fails to provide the desired performance on an industrial scale. The cause of such failures is an incomplete understanding of the phenomena taking place in the mixing systems and the effect of scale-up procedures on them. One process in which engineers deal significantly with those challenges is the gold cyanidation process. Cyanidation is the most common process for extracting gold from its ore. Gold is extracted from ore by a reaction among gold and cyanide in the presence of oxygen. This is a three-phase reaction where oxygen should be dissolved in the liquid phase and the dissolved oxygen and cyanide should be transferred to the surface of the solid where the reaction takes place in/on the solid particles. The chemistry of the gold cyanidation reaction is very complicated. The type of reactor used in this process is a stirred tank reactor. In order to achieve a high recovery of gold a cascade of agitated vessels are used, generally 8-10 reactors. Cyanidation reactors commonly suffer from some general drawbacks. The major drawbacks are 1) high consumption of reactants (cyanide and oxygen), 2) high residence time of the process 3) undesirable product selectivity.

Process synthesis specifies that the amount of cyanide and oxygen consumption in this process is very high compared to what is theoretically required (Jafari et al. 2008). This high rate of reactant consumption results in high material and operating costs. As mentioned before, the cyanidation reaction is a three-phase reaction and different phenomena contribute to complete the reaction, in particular mass transfer of reactants to the reaction site. Gold cyanidation itself is a very fast reaction. So the controlling step in the reaction is the mass transfer rate, which could be either the liquid-solid or gas-liquid mass transfer rate. The long residence time required to achieve a high recovery of gold is an important factor to confirm this. On the other hand, the content of gold in ore is very low (1-20 ppm) and in some cases it is locked in a strong matrix of other minerals. Since cyanide can react with all the minerals in ore the selectivity of the desired reaction is low and usually the final product comes with a large amount of copper or other minerals, which requires an additional purification process.

$$4Au + 8NaCN + O_2 + 2H_2O \leftrightarrow 4NaAu(CN)_2 + 4NaOH$$



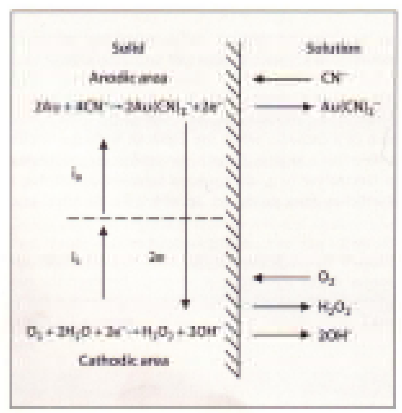


Figure I-1 Gold cyanidation process, reaction stoichiometry and mechanism

Most research studies on the subject of gold cyanidation aimed to understand the reaction mechanism and other chemical phenomenon that take place during the course of the reaction. Despite all of these efforts no significant improvements in these processes has been achieved. Although some novel reactors have been introduced or cyanide has been replaced by other extracting materials, cyanidation in an agitated vessel is still the most appropriate choice from an industrial standpoint.

To recognize the problems associated with the cyanidation process some primary tests were carried out[1]. The objective was to realize whether operating conditions have an effect on the cyanidation process or not. For this purpose the cyanidation reaction has been carried out in a laboratory scale reactor (T=0.4, H=0.4). Different parameters have been changed, such as impeller type, rotational speed, gas flow rate and sparger type. Results showed that in all cases the process improved significantly. As an example, increasing the impeller speed decreased the required residence time by 50% and decreased cyanide consumption by 30%. Based on these results it was concluded that the current technology for the cyanidation reactor is suffering from poor mixing conditions. To overcome this issue and provide better mixing conditions it was necessary to redesign the agitated vessel and properly define the operating conditions. As listed in Figure 2, many parameters should be defined.

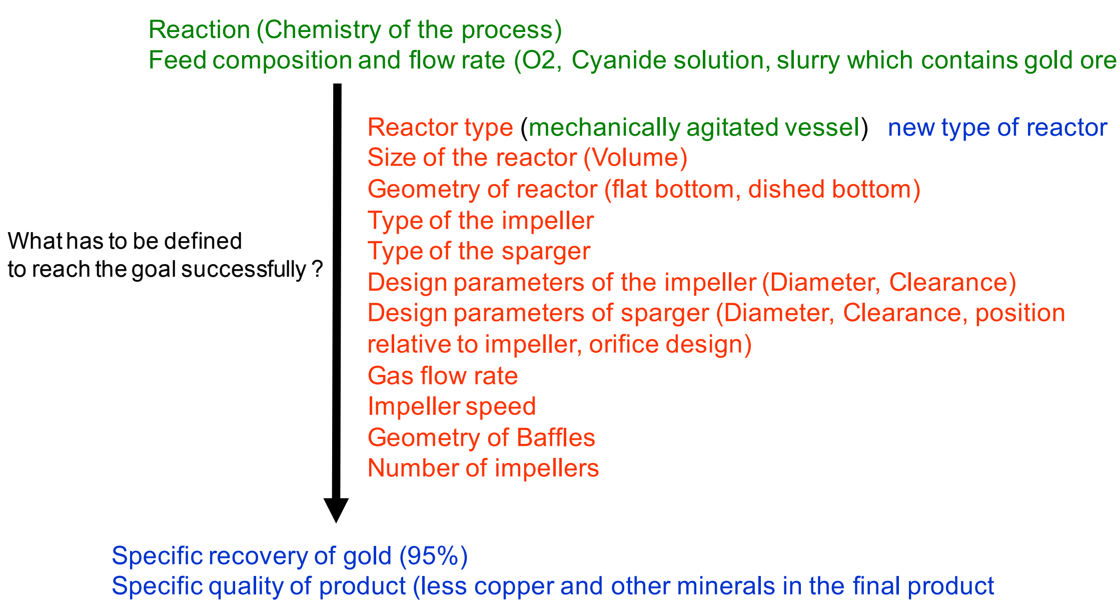


Figure I-2 design procedure for a gold cyanidation reactor

Proper selection of equipments or defining the operating conditions is critical for process success. Geometrical and operating conditions must be defined in such a way that provides optimum

[1] Tests have been done in COREM

reaction conditions. This requires comprehensive knowledge about how the process may be affected by changing physical, operational, and geometrical parameters. Failure to operate at optimal conditions leads to considerable drawbacks. A lack of accurate experimental methods and precise data on solid suspension, gas dispersion, and uncertainties associated with scale-up procedures provided the necessary motivation to undertake this research.

CHAPTER 1

LITERATURE REVIEW

All industrial chemical processes are developed to transform cheap raw materials into a high value product. This goal usually is achieved by employing chemical reactors. A reactor in which such chemical transformations take place has to carry out several functions, such as bringing a reactant into intimate contact to let chemical reactions occur and providing the appropriate environment (temperature, pressure etc.) for an adequate length of time. Chemical reactor engineering includes necessary activities to determine the best possible hardware and operating protocol of the reactor. These parameters may lead the reactor to carry out the desired transformation of raw materials (reactant) to products (value added products). A reactor engineer has to ensure that the reactor hardware and operating protocol of the reactor satisfy the various process demands without compromising safety, the environment and economics. The reactor engineer has to establish a relationship between reactor hardware, operating protocols, and various performance issues. To establish the relationship between reactor hardware and reactor performance, it is necessary to use a different tools (models). Creative application of the best possible tools is necessary to develop the best hardware configuration and define the best operating protocol for the considered reactor. Reactor engineers deal with this matter by using comprehensive knowledge about reaction kinetics and, fluid dynamics and hydrodynamic of each reacting system.

Multiphase flow processes are key elements of several important reactor technologies. These technologies cover a wide range, from very large scale operations, such as fluid catalytic cracking reactors, to specialized reactors to produce high value, low volume specialty chemicals. The presence of more than one phase raises several additional questions. Multiphase flow processes exhibit different flow regimes depending on the operating conditions and the geometry of the process equipment. It is often necessary to evaluate the operability of the multiphase flow process under specified conditions and to identify the operating regime. The fluid dynamics of the multiphase reactors are especially sensitive to reactor configuration and operating conditions. Even small scale hardware details, such as the design of the distributor, may have a dramatic influence on the resulting flow regime.

1.1 Mechanically Agitated Vessel

Stirred reactors, in which one or more impellers are used to generate flow and mixing within the reactor, are among the most widely used reactors in chemical and related industries. Stirred reactors offer unmatched flexibility and control over transport processes occurring within the reactor. Parameters such as reactor shape, aspect ratio, number, type, location and the size of the impellers and the degree of baffling provide effective handles to control the performance of stirred reactors. However, the availability of such a large number of parameters makes the job of selecting the most suitable configuration for the stirred reactor quite difficult. Mixing and contacting in agitated tanks can be accomplished in continuous, batch or fed-batch mode. A good mixing result is important for minimizing investment and operating costs, providing high yields when mass transfer is limiting and, thus, enhancing profitability. Fluid mixing is carried out in a mechanically stirred vessel for a variety of objectives, including homogenization for single or multiphase systems, in terms of concentration of components, physical properties and temperature. The fundamental mechanism involves the physical movement of the materials between various parts of the entire mass using rotating impeller blades.

Over 50% of the world's chemical productions involve these stirred vessels for manufacturing high-added-value products. These vessels are commonly used for the following:

I. Blending of homogenous liquids

II. Suspending solids

III. Dispersion of gas in liquid

IV. Homogenous viscous complex liquids

V. Transferring heat through a jacket and/or internal coils for heating or cooling

Table 1-1, Some industrial applications of stirred reactors (Ranade 2002)

Phases handled	**Application**
Liquid	Alkylations, sulfonations, esterification, Bulk and solution polymerization (styrene, acrilonitrile, ethylene, propylene)
Gas-liquid	Oxidation (ethylene paraffin), chlorination (acetic acid, dodecane), Carbonylations (methanol, propanol), esterifications, manufacture of sulfuric acid, adipic acid, oxamide and so on
Gas-liquid-solid	Hydrogenations (olefins, edible oils, several chloro and nitro aromatics), Oxidation (p-xylene), Fermentation (alcohol, single cell proteins, antibiotics) waste water treatment and so on
Liquid-solid	Calcium hydroxide (from calcium oxide), regeneration of ion exchange resin, anaerobic fermentations

A conventional stirred tank consists of a vessel equipped with a rotating impeller. The vessel is generally a vertical cylindrical tank. A mechanically agitated vessel can be operated in three different regimes (i.e., Laminar flow, Turbulent flow and Transitional flow).

Turbulence is a major phenomenon responsible for mixing and all the typical processes, like mass transfer, heat transfer, liquid-liquid dispersion, gas dispersion and solid suspension, are significantly affected by its presence. Dealing with the interaction of the nature of turbulent fluctuation and mixing processes requires the understanding of the nature of the turbulence. Turbulence is a state of fluid motion where the velocity fluctuates in time and all three dimensions. These fluctuations reflect the complex layering and interactions of large and small structural elements, such as vortices, sheets, ejections, and sweeps of a variety of shapes and sizes. Fully turbulent conditions are achieved at a very high impeller Reynolds number. Fully turbulent flow is an asymptotic state where the velocity fluctuations are so intense that interfacial forces overwhelm viscous forces.

To accomplish the chemical reaction the initial bulk mixing, effective turbulence and molecular diffusion for the final molecular contact are needed. The outcome of chemical reaction will depend on the rate of mixing compared to the rate of reaction. When the rate of reaction is slow compared to the mixing time, the reaction is not affected by mixing because the mixing is complete by the time reaction occurs. When the rate of reaction is fast compared to the rate of mixing, the system is mixing limited.

In most of the chemical processes where a mechanically agitated vessel is used; components are introduced into the process in different phases. In these cases mechanically agitated vessels are used for dispersing a gas phase in a liquid phase, suspending solid particles in a liquid phase or agitating a three phase system where both gas dispersion and solid suspension are required.

1.2 Solid Suspension

Stirred tanks are commonly used for suspending solids. Suspending solid particles in a turbulent liquid can be considered as balancing energy supplied by a rotating impeller and energy needed to lift the suspended solid. Industrial applications requiring adequate mixing of solids in liquids include coal slurries, a catalyst polymer system, solid dissolution, crystallization, pulp and paper, ore leaching, and so on. Axial flow impellers with high pumping efficiencies are most suitable for solids suspension. These impellers generate a flow pattern which sweeps the tank bottom and suspends the solids. Solid pickup from the vessel base is achieved by a combination of the drag and lift force of the moving fluid on the solid particle and the bursts of turbulent eddies originating from the bulk flow in the vessel. In agitated vessels the degree of suspension is generally classified into three levels; on-bottom motion, complete off-bottom suspension and uniform suspension. In partial suspension some solids rest on the bottom of the tank for short periods. Since particles are in constant contact with the bottom of the vessel, all the surface area of particles is not available for chemical reaction or mass or heat transfer. This state is sufficient for the dissolution of highly soluble solids. Complete motion of all particles is known as off-bottom or complete suspension. Under this condition the maximum surface area of the particles is exposed to the fluid for chemical reaction or mass or heat transfer. The just-suspended condition refers to the minimum agitation condition at which all the particles attain complete suspension. The minimum agitation speed for the just-suspended state in a mechanically agitated vessel is known as N_{js}. Uniform suspension corresponds to the state of suspension at which particle concentration and particle size distribution are practically uniform throughout the vessel. Any further increase in agitation speed or power does not appreciably enhance the solid distribution. Many efforts have been made to characterize these parameters experimentally, and theoretically which result in different equations for predicting these parameters.

Zwietering's work (Zwietering 1958) was one of the first studies in this field. He introduced the visual observation method to determine the N_{js}. The motion of the solid particles on the tank bottom was visually observed through the transparent tank wall and the tank bottom. N_{js} was measured as the speed at which no solids are visually observed to remain at rest on the tank bottom for more than 1 or 2 seconds. The main advantage of visual methods is simplicity. However, only with careful and experienced observation it is possible to achieve ±5% reproducibility in a diluted suspension. Further, visual methods require a transparent vessel, which is feasible for most laboratory-scale studies, but rather out of reach for large-scale vessels. To overcome the limitations of the visual technique other methods have been proposed.

Different concepts have been used for characterizing N_{js}, like variation of power consumption or mixing time by Rewatkar et al. (Rewatkar et al. 1991), solid concentration change directly above the vessel bottom by Bourne and Sharma (Bourne and Sharma 1974) and Musil (Musil and Vlk 1978), ultrasonic beam reflection from the static layer of the solid on the vessel base by Buurman et al (Buurman et al. 1985), pressure change at the bottom of the vessel by Micale et al. (Micale et al. 2000a, Micale et al. 2002).

Experimental techniques have been applied to numerous empirical and semi-empirical investigations on solid suspension, whose results have been critically reviewed in the literature (Armenante and Nagamine 1998, Armenante et al. 1998, Atieme-Obeng et al. 2004, Nienow 1985,1992). Most presented correlations have been developed based on the visual technique. Most of the studies resulted in modifications of model parameters in the Zwietering correlation:

$$N_{js} = S\vartheta^{\alpha}\left[\frac{g_c(\rho_s-\rho_l)}{\rho_l}\right]^{\beta} d_p^{\gamma} D^{\delta} X^{\theta} \qquad \text{Eq.1-1}$$

It was shown that a significant variance appears in the prediction of N_{js} and there is no correlation with universal validity. Bohnet and Niezmak (Bohnet and Niesmak 1980) calculated N_{js} using nine correlations to find that the reported values were in the range of **-56% to +250%** from their own value. Different empirical correlations have been developed based on experimental characterization of N_{js}.

Prediction of just suspended speed was a subject of few CFD studies (Fletcher and Brown 2009, Lea 2009, Murthy et al. 2007, Panneerselvam et al. 2008). Lea (Lea 2009) used CFD-assisted design approach to study effectiveness of mixing tank geometrical configurations to suspend particles. He developed design heuristic that can be applied in process industries. Murthy et al. (Murthy et al. 2007) used CFD simulation to study effect of different parameters on just suspended speed in LS and GLS systems. Their study covers solid loading up to 15% (wt/wt). Fletcher and Brown (Fletcher and Brown 2009) studied the influence of the choice of turbulence models on the prediction of solid suspension by means of commercial CFD codes. Kee and Tan (Kee and Tan 2002) presented a new CFD approach for predicting N_{js} and characterized effect of D/T and C/T on N_{js}. Ochieng and Lewis (Ochieng and Lewis 2006) provided qualitative and quantitative insight into solid suspension by simultaneous investigation by CFD and LDV. In their work suspension studies have been carried out in a Nickel precipitation process and best simulation results were obtained for solid loading lower than 6%.

1.3 Solid Dispersion

According to the process, it is possible to carry out the mixing of liquid-solid system in the state of just suspended or homogeneous suspension. Homogeneous suspension, when solid phase uniformly is distributed in the stirred vessel, is difficult to attain and usually is not required in most industrial applications. Most published studies on liquid-solid agitated vessels have been dedicated to characterizing just suspended condition. Other parameters related to a liquid-solid mixing system, like cloud height, solid concentration profile, power consumption and scale-up, have not been studied extensively in high concentrated systems.

Numerous methods are available for measuring local solids concentration in slurry. One of the popular methods is the optical method. It has been used widely for characterizing solid distribution in agitated vessels (Ayazi Shamlou and Koutsakos 1989, Magelli et al. 1990, Magelli et al. 1991). This non-intrusive method is generally limited to solids concentrations less than 1–2%. This is due to the scattering and blocking of light by the solids between the source and the receiver. Other measurement methods are the sample withdrawal method and the conductivity probes. The sample withdrawal method is the simplest one and has been employed widely (Barresi and Baldi 1987, MacTaggart et al. 1993). However, it is has been shown that Iso-kinetic sampling from stirred tank reactors is extremely difficult because of the complex dynamic behavior of the system (Barresi et al. 1994, MacTaggart et al. 1993, Nasr-El-Din et al. 1996). Another method is conductivity measurement, which is based on the conductivity changes in the suspension according to the quantity of solid particles present. The conductivity method is low cost and accurate in dense systems. But, there is an intrusive effect of the probe in the vessel. The influence of the probe on the suspension process can be eliminated by suitably adjusting the size proportion of the probe versus vessel diameter. (Considine and Considine 1985, Nasr-El-Din et al. , Nasr-El-Din et al. 1996, Spidla et al. 2005). Recently Mann et al. (Mann et al. 2001) applied Electrical Resistance Tomography (ERT) to visualize fluid mixing and solid concentration profile in stirred reactors. Optical fibers also have been used widely for characterizing solid concentration in multiphase systems (Boyer et al. 2002, Chaouki et al. 1997).

Buurman et al. (Buurman et al. 1986) studied a highly concentrated system at relatively homogeneous conditions. They reported no differences in the solid concentration at three sample withdrawal points situated in an axial direction. The significance of the radial concentration gradient has never been analyzed in detail, even though the presence of the radial concentration gradient depends on the stirrer type and speed as well as on impeller off-bottom clearance, particle diameter and solid loading. Literature data suggest that the radial concentration gradients are usually negligible (Barresi and Baldi 1987, Magelli et al. 1991, Montante et al. 2002, Yamazaki et al. 1986). However, this assumption cannot be generalized. The presence of radial concentration gradients has been reported by Micheletti et al. (Micheletti et al. 2003). By measuring solid concentration at different radial positions they indicated that a solid concentration gradient exists and it depends on particle size, solid loading and impeller type. It is negligible only for small particle sizes, but increases significantly when particles of a larger size or density are suspended. Angst and Kraume (Angst and Kraume 2006) determined axial and radial particle distributions using an endoscope system. Spidla et al. (Spidla et al. 2005) measured solid concentration by using a conductivity probe in a large scale vessel. They have also proved the presence of radial solid concentration in an agitated vessel.

Several authors have modeled solid distribution in stirred tank for low concentrations of solid and several fluid dynamic models have been adopted for describing solid distribution throughout the tank. They include one dimensional sedimentation dispersion model (Barresi and Baldi 1987, Magelli et al. 1990, Rasteiro et al. 1994, Sessiecq et al. 1999, Shamlou and Koutsakos 1987), multi-zone sedimentation dispersion model (Yamazaki et al. 1986), two- or three-dimensional network of zones (Brucato et al. 1991, McKee et al. 1995). In addition to these phenomenological models, computational fluid dynamics also have been used with different methods and techniques (for example, (Guha et al. 2008, Khopkar et al. 2006, Micale et al. 2000b, Montante et al. 2001). Both CFD and phenomenological are useful however they are opposite in terms of complexity. CFD potential is still to be developed and carefully checked against experimental data especially in liquid-solid systems with high concentration of solid.

Mass transfer at the interface between suspended particles and liquid is one of the most important factors in chemical and biochemical processes. Therefore many correlations of the solid liquid mass transfer coefficient in a stirred vessel have been reported (Calderbank and Moo-Young 1961, Levins and Glastonbury 1972, Miller 1971). There are few published works about three phase gas-liquid-solid systems (for example (Grisafi et al. 1998)). An extensive review about particle liquid mass transfer coefficient in two- and three-phase agitated vessels has been represented by Pangarkat et al. (Pangarkar et al. 2002). Kato et al. (Kato et al. 2001) measured the solid-liquid mass transfer coefficient in a vessel agitated with a new type of impeller. They used ion exchange resin and a conductivity probe to measure the mass transfer coefficient. The solid-liquid mass transfer coefficient was measured by Grisafi et al. (Grisafi et al. 1998). They reported a comprehensive study about mass-transfer coefficient in a three-phase mechanically agitated vessel.

The effect of the gassing rate on the solid-liquid mass transfer coefficient was studied by Fishwick et al. (Fishwick et al. 2003). They used the positron emission particle tracking technique to model liquid flow in the three-phase system. The solid-liquid mass transfer coefficient is measured using the vacuum sampling technique, which provides a small sample of dissolved solid particle concentration from the agitated vessel. The effect of the gassing rate on liquid phase flow and its influence on the solid-liquid mass transfer coefficient also was discussed. Solid-liquid mass transfer was found to decrease with the dispersion of gas.

1.4 Power Consumption

Another important parameter in the design and operation of mechanically agitated vessels is the amount of power dissipated in the vessel by the impeller. Bujalski (Bujalski et al. 1999) determined the power number for the A-310 impeller (hydrofoil) and showed that by increasing the solid loading power, the number increases. Measurements were made in that study for solid loading up to 40% wt/wt. At solid loading higher than 20% wt/wt the variation of power number showed different trends. At low impeller speeds (N<200 rpm) the power number is lower than that for the single phase. By increasing impeller speed the power number increases until it reaches a maximum value and then it slightly decreased when increasing the impeller speed. The constant value of the power number at higher impeller speeds was higher than that for the single phase and its magnitude related to solid concentration, which affects the mixture density surrounding the impeller. Bujalski et al. (Bujalski et al. 1999) related the lower power variation to bottom shape change by increasing impeller speed. They have mentioned that the presence of a solid layer at the bottom of the vessel redirected the flow and the overall effect is that the power number at low speed is reduced. By increasing impeller speed this layer starts to vanish and the power number increases.

Wu et al (Wu et al. 2002) reported power number variation for PBT-D and RT at extreme solid concentrations (>50 % vol/vol). As reported in their study, the power number of PBT increases at high solid concentrations while that of RT decreases. Increasing the power number for PBT can be described in the same way as Bujalski et al (Bujalski et al. 1999) did, but a reduction in the power number of RT was related to the fact that damping at high solid loading suppresses the dead flow zones at the back of the Rushton turbine blades leading to the reduction of drag. On the other hand there is no dead flow zone behind the blade of PBT-D and increasing solid loading only increases skin friction and, accordingly, drag coefficient. Those results are in contrast with what has been reported by Angst and Karume (Angst and Kraume 2006), who reported the reduction of the power number for the Pitched blade turbine.

1.5 Gas Dispersion

Mechanically agitated contactors are widely used in industrial processes for absorption, striping, oxidation, hydrogenation, chlorination, carbonylation, fermentation and so on. They are also used for carrying out biochemical processes, such as aerobic fermentation, manufacture of protein, and waste water treatment. In all these processes gas must be effectively and efficiently contacted with liquid to provide mass transfer. A gas sparger is used when gas should be introduced into the liquid for efficient gas-liquid contacting for mass transfer and/or reaction. While mixer design and operating conditions control the gas-liquid interfacial area, a well-designed and well-located sparger can enhanced the gas-liquid process result by maximizing contact.

Gas-liquid flow in a stirred reactor depends on operating conditions and impeller design and can be classified into different regimes. These flow regimes correspond to different fluid dynamic characteristics and demonstrate complex interaction of transport and mixing processes. Significant research efforts have been undertaken for developing regime maps and the corresponding design correlations. For very low impeller speed, flow generated by rising gas bubbles is dominant. In such cases stirred tank behaves like a bubble column. In other extremes where flow is dominated by the impeller, gas bubbles follow liquid streamlines and are dispersed all over the reactor. In such a case the gas phase behaves as if it is completely mixed. When the impeller speed is low, the impeller is said to be flooded. As impeller speed increases gas is captured by the vortices behind the agitator blades and the impeller is said to be loaded. A further increase in impeller speed leads to the minimum speed to completely disperse the gas throughout the whole vessel. At high impeller speed gross circulation of gas back into the agitator sets in. The universal applicability of the regime maps and related correlations to design, scale-up, and setting up of operating protocols for industrial systems has been considered by many researches (Middleton and Smith 2004).

The most efforts in multiphase stirred vessels have been made to understand mass transfer between phases in a stirred vessel and the correlating mass transfer coefficient. The most comprehensive study has been carried out by Kawase et al. (Kawase et al. 1997), who characterized the gas-liquid mass transfer coefficient for both Newtonian and non-Newtonian fluids with different solid particles.

Nocentini et al. (Nocentini et al. 1998) studied the gas behavior in a large, gas-liquid agitated vessel with multiple impellers. They proposed that the gas behavior can be simply described by a plug flow model with axial dispersion. They used methane as the tracer for RTD experiments. By studying the RTD experiment response to pulse injection they calculated the Pe number for different impeller speeds and gassing flow rates. They also proposed a scale-up correlation for calculating the Pe number for larger scale vessels. Manikowski et al. (Manikowski et al. 1994) studied the hydrodynamics of a multi-impeller, gas-liquid stirred vessel. They used Rushton turbine and Lightnin A-315 impellers. A ring sparger was used to sparge gas into the system and it was fixed to the bottom of the vessel. Two different types of liquid (different CMC solutions in water) were used as the liquid phase. The bubble rise velocity was measured by the ultrasound droplet technique. Pinelli and Magelli (Pinelli and Magelli 2000) studied the liquid and gas phase macro-mixing behavior in a gas-liquid contactor with a high aspect ratio. They considered the plug flow model with axial gas dispersion and characterized the axial dispersion coefficient at different operating conditions along the bed.

Their studies were followed by Majirova et al. (Majirova et al. 2004), who studied gas-phase hydrodynamics for different impellers and characterized the hold up and dispersion coefficients in a gas-liquid agitated vessel. Pinelli 2005 (Pinelli 2005) proposed a new model for gas phase hydrodynamics. He considered two classes of bubbles, i.e., small and large size bubbles. Small size bubbles behave like a CSTR while large bubbles behave like a plug flow reactor. Based on RTD experiments he evaluated model parameters for both classes of bubbles.

Garcia-Ochoa and Gomez (Garcia-Ochoa and Gomez 2004) presented a theoretical approach for predicting gas-liquid mass transfer coefficient and hold-up in gas-liquid stirred tank reactors. They have developed a method based on theoretical principles for determination of the volumetric mass transfer coefficient, k_La, in stirred tank reactors with Newtonian and non-Newtonian fluids. Their model is based on Higbie's penetration theory, which establishes a relationship between the mass transfer coefficient, k_L, and the contact time between two different phase elements. This exposure time can be estimated from turbulence isotropic of Kolmogorof theory as the eddy length to fluctuation velocity ratio. Volumetric mass transfer coefficient values can be predicted with reasonable accuracy.

Martin et al. (Martín et al. 2008) have proposed a model to predict a mean k_La as a combination of two scales of mixing in stirred tank. Two main scales of mixing can be considered inside a stirred tank: macro-mixing and micro-mixing. Macro-mixing is related to the tank size circulation and is responsible for bubble motion, surface aeration and tank homogenization. Meanwhile, micro-mixing is related to the small liquid eddies, responsible for the concentration gradients surrounding the bubbles, and it prevails around the impeller. They have used experimental results and empirical equations to unveil the contribution of both mechanisms to the volumetric mass transfer coefficient, k_La.

Recently some studies have been done to characterize gas-liquid mass transfer distribution in the stirred tanks (Alves et al. 2004, Laakkonen et al. 2006, Laakkonen et al. 2007). However the effect of scale-up procedure on kLa distribution in the vessel has not been investigated.

1.6 Conclusion

Comprehensive knowledge about the effect of different factors (physical properties, geometrical and operational parameters) is centeral for the proper design and operation of LS and GLS agitated vessels. For the design of agitated concentrated systems, it is important to develop more reliable techniques for characterizing just suspended speed especially for processes where conventional techniques cannot be applied (high concentration LS system). In addition, the evaluation of process performance requires detailed knowledge on solid concentration distribution in the vessel and active volume of the reactor to be able to accurately predict selectivity and yields of the reactor. The power number needs to be characterized to estimate power consumption and it is very important to choose proper scale-up procedures. It is also important to have the correct estimation of gas dispersion in an agitated vessel and know how it may change during the scale-up procedure. In the multiphase reactor, chemical phenomena (reaction kinetics) are usually independent of the vessel dimensions, while many physical phenomena are significantly affected by dimensional change. Intense mixing is easy to achieve in small scale reactors while in larger scale reactors reactants experience different flow patterns, mixing conditions and turbulence structures. In most gas-liquid multiphase reactors non-homogenous distribution of dissolved gas is responsible for poorer performance in large scale operations and causes serious drawbacks on an industrial scale.

Nomenclature

dp	*Mean particle diameter (m)*
D	*Impeller diameter (m)*
g_c	*Gravity acceleration* (m/s^2)
k_L	*Mass transfer coefficient in liquid phase (m/s)*
k_La	*Gas-liquid mass transfer coefficient (1/s)*
N	*Impeller speed (1/sec)*
N_{js}	*Impeller speed (1/sec) at just suspended condition for LS system*
S	*Zwietering correlation constant (-)*
X	*Solid loading Ms/Mt×100 (% wt/wt-),*

Greek letters

$\alpha, \beta, \gamma, \delta, \theta$	*Constants of equation 1-1*
ρ	*density (kg/m3)*
ν	*Liquid kinematic viscosity (m2/s)*

Subscribes

js	*Just suspended*
l	*Liquid*
s	*solid*

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CHAPTER 2

METHODOLOGY

2.1 Problematic and Motivation for Study

Maximum solid-liquid contact is vital for the optimization of many chemical processes. In many processes (especially dissolution, leaching and solid-catalyzed reactions), the main objective of liquid-solid contacting is to maximize the surface area of the solid particles available for reaction or transport processes (heat and/or mass transfer). This can only be achieved by optimizing hydrodynamic conditions where solid particles move freely and do not accumulate at any point in the vessel. At impeller speeds lower than the just suspended condition, mass transfer clearly increases with a higher impeller speed. On the other hand, the observed rate may not increase significantly with impeller speed or mixing intensity beyond the just-suspended condition. This indicates that operating at the just suspended condition is the minimum requirement in cases where mass transfer is controlling the process. It is important to define what level of suspension is required versus the desired process results. While the just suspended condition is optimal for many processes, a high degree of suspension (homogeneity) is required for crystallization or the slurry feed system and partial suspension is sufficient for the dissolution of highly soluble solids. Failure to operate at optimal conditions due to uncertainty in predicting the impeller speed required to achieve and maintain the just suspended condition leads to considerable drawbacks. If the mixing system operates above the minimum speed for solid suspension, the degree of suspension will be improved and the mass transfer rate will be enhanced. A higher speed, however, also provides a higher turbulence shear rate, which for some processes, i.e., biological processes, may cause undesirable particle attrition. Obviously, there is also a practical cost-effective limit on the maximum speed of agitation. For example, in the gold cyanidation process, where a high concentration slurry (up to 50% wt/wt) is processed to achieve a high production rate of gold, operating at a lower impeller speed than the just suspended condition will generate fillets in the vessel, thereby detrimentally affecting the reaction selectivity and yield. In some cases, a small proportion of particles may be allowed to accumulate in corners or on the bottom in relatively stagnant regions to form fillets. This condition may offer advantages from a practical point of view because of a large savings in power consumption compared to what is required for eliminating fillets. This power savings may be greater than the loss of active solids. However, it is important to quantitatively define what portion of solid is left unsuspended. On the other hand, over-predicting N_{js} has significant economical drawbacks. For example, in the gold cyanidation process 8-10 reactors in series are used to achieve a high recovery of gold (up to 96%). Vessel diameter is 8 (m) with the same slurry height. Solid loading is 50% (wt/wt). Although under-estimation of N_{js} or N_{jsg} results in decreasing gold recovery, over-estimation of N_{js} also leads to significant variations in estimated capital and operating costs. 10% to 100% over-prediction of N_{jsg} leads to \$0.5M to \$10.3M/year of additional energy costs[1] while under-estimation of N_{jsg} leads to decreased gold recovery. Also, additional costs for the purchase, installation, and maintenance of larger mechanical parts should be considered. This added capital and operating cost cannot be compensated by additional gold recovered from the process.

Comprehensive knowledge about the effect of different factors (physical properties, geometrical and operational parameters) is vital for the proper design and operation of LS and GLS agitated vessels. Although characterizing N_{js} and N_{jsg} was the subject of much research and many published scientific contributions, few articles (or even books), however, have gathered all the information in one place. There is no article, which has critically reviewed the effect of all parameters on N_{js} and N_{jsg} or considered the theoretical prediction of N_{js} and N_{jsg}. This article intends to do so and provides the proper tool for scientists and engineers to deal with the design assignments of mechanically

[1] Cost of electricity was considered 0.06\$/kWh (for Quebec)

agitated vessels. Also, the subjectivity of conventional measurement techniques leads to extensive uncertainty in predicting N_{js}.

For the design of agitated concentrated systems, it is important to develop more reliable techniques for characterizing just suspended speed. In addition, the evaluation of process performance requires detailed knowledge on solid concentration distribution in the vessel and active volume of the reactor to be able to accurately predict selectivity and yields of the reactor. The power number needs to be characterized to estimate power consumption and it is very important to choose proper scale-up procedures. It is also important to have the correct estimation of gas dispersion in an agitated vessel and know how it may change during the scale-up procedure. In the multiphase reactor, chemical phenomena (reaction kinetics) are usually independent of the vessel dimensions, while many physical phenomena are significantly affected by dimensional change. Intense mixing is easy to achieve in small scale reactors while in larger scale reactors reactants experience different flow patterns, mixing conditions and turbulence structures. In most gas-liquid multiphase reactors non-homogenous distribution of dissolved gas is responsible for poorer performance in large scale operations and causes serious drawbacks on an industrial scale. All of the above-mentioned reasons provided good motivation to undertake this research.

2.2 Objectives

This document includes experimental investigation on gas dispersion and solid suspension in a mechanically agitated vessel. The project aims to accomplish the following:

- To improve knowledge about the solid suspension mechanism,
- To provide an accurate method for characterizing states of solid suspension and identifying the accuracy of modeling tools and theoretical methods for predicting solid off- bottom suspension condition.

- To clarify the effect of operating conditions and design parameters on solid suspension and developing a comprehensive guideline for engineers and scientists to deal with design assignments
- To analyze solid dispersion in agitated vessels for different operating conditions and recommend proper scale-up procedures
- To investigate gas dispersion in gas sparged agitated vessel and evaluate the contribution of macro-mixing and micro-mixing on the gas liquid mass transfer coefficient in order to explain issues associated with conventional scale-up procedures and propose an improved one.

2.3 Methodology: Materials and Methods

2.3.1 Materials

Water was used as the liquid phase and sand as the solid phase (density of 2650 kg/m^3). Particle size distribution of sand was measured by the Horiba laser scattering particle size distribution analyzer (model: LA-950). The mean particle size was 277µm. The operating slurry height was set equal to the vessel diameter.

2.3.2 Experimental setup

Experiments were conducted in a 14 L transparent polycarbonate agitated cylindrical vessel with standard baffles, an open top and a flat bottom. Three different impellers were tested, mounted on central a central shaft, namely a six blade Rushton turbine (RT), a concave blade turbine (CBT), and a four-blade pitched blade turbine in down-pumping mode (PBT-D). Schematic diagrams of the experimental setup were illustrated in Figures 2-1 (a & b). The vessel, impeller dimension, and geometrical details of the mixing system are given in Table 2-1.

Gas was fed into the system using a ring sparger with a diameter of ds=0.75D (as recommended in (Atieme-Obeng et al. 2004). This device was located immediately under the impeller. Air was supplied through eight orifices facing upward, each with a 1 mm diameter. Air flow rate was measured and controlled with an accuracy of ±0.05 L/min using a mass flow meter (Aalborg, Model GFM371) with a flow range of 0-50 L/min.

2.3.3 Measurement methods

The power consumption in the vessel by agitation was measured using a torque meter (Himmelstein Model 24-02 T).

(a)

(b)

Fig. 2-1. Experimental setups, a) Large vessel b) Small vessel

Beer-Lambert's law describes the decay in intensity of the emitted gamma ray by passing through the medium: $I=I_0.\exp(-\rho\mu l)$. Changes in the density or phase of the medium lead to corresponding changes in the gamma-ray intensity recorded by the detector. In multi-phase systems, the ray intensity is related to the volume fraction of each phase.

In practice a source of gamma ray consisting of a 2 mm glass bead filled with scandium oxide was activated in the Slowpoke nuclear reactor of École Polytechnique of Montreal. The source activity was 150 μCi and the half-life time of the tracer was 84 days. The tracer was placed in the holder where it was completely shielded by lead. Emitted gamma rays from this source were collimated by lead support. It passed through a 5 mm hole on the protection shield and went through the vessel. A NaI scintillation detector (Teledyne Isotope, Model S-1212-I) was placed on the other side of the vessel and coupled to an amplifier (EG&G ORTEC Model: 925-SCINT) and a data acquisition system (TOMO MSC plus-17). Both tracer and detector were positioned in order to be able to scan the region about 0.5 cm from the bottom of the vessel. The signals were recorded for 2 minutes with a 200 msec sampling time at each impeller speed (varied between 0 to 1000 rpm with different step sizes). Counts were recorded at each impeller speed and they were converted and processed by home-made codes. It was verified that changing the sampling time and recording period as well as the background noise did not alter the experimental results. The original recorded count rates were related to the solid volume fraction. For this purpose, the same region was scanned without solids (pure water). According to this procedure, the following equations can be established. Equation 2-1 relates the measured intensity to solid hold-up.

$$I_{liquid-solid(N>0)} = I_0 exp\big(-\rho_{water}\mu_{water}L(1-\epsilon_s) - \rho_{solid}\mu_{solid}L(\epsilon_s)\big) \cdot exp(-A) \qquad \text{Eq. 2-1}$$

Typical results of the densitometry technique are shown in Fig. 2-2. This figure shows variation of count rate recorded by detector vs. impeller speed. At N = 0 (rpm), when all the solid particles settled on the bottom of the vessel, the recorded intensity by the detector $I_{N=0}$ is constant. By increasing the impeller speed and as solid particles in the scanning region commence motion and are lifted by the liquid, the recorded intensity increases. At higher impeller speeds, when all the solid particles are experiencing random motion and no solid rests on the bottom of the vessel, the recorded intensity is expected to stabilize.

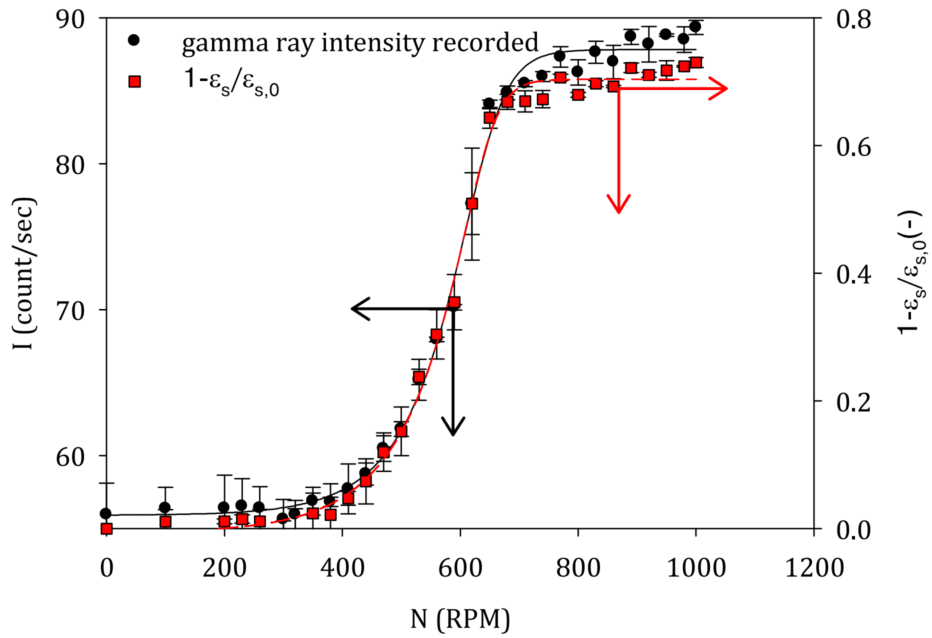

Fig. 2-2. Variation of recorded count rate and average solid hold-up by increasing impeller speed at the bottom of the vessel. Impeller: RT, X: 20%, d_p: 277 μm, C/T: 0.33.

In practice, a slight intensity increase can be observed, which is related to the change in solid particle speed and a decrease of the residence time of the solid in the scanning zone. Solid hold-up can be calculated from recorded count rates by employing equation 5-1. Fig. 2-2 also illustrates the variation of solid hold-up at the bottom of the vessel by increasing impeller speed. As many researchers have mentioned (for example (Musil et al. 1984)) solid concentration at the bottom of the vessel at just suspended conditions exhibits a discontinuity. As shown in Fig. 2-2, based on densitometry data, a discontinuity in solid concentration can be noticed at the bottom of the vessel. The starting point of this discontinuity is considered as N_{js}. $\varepsilon_s/\varepsilon_{s,0}$ represents the normalized solid volume fraction at the bottom of the vessel. By plotting 1- $\varepsilon_s/\varepsilon_{s,0}$ vs. impeller speed the discontinuity in solid concentration at the bottom of the can be identified clearly.

As illustrated in Fig. 2-2, for low impeller speed, all solid particles rest on the bottom of the vessel base. Upon increasing impeller speed, a fraction of the solid particles commences lifting and reaches suspension at a certain height. Partial suspensions correspond to the situation where some solids rest on the bottom of the tank. Since the particles are in constant contact with the bottom of the vessel, not all the surface area of particles is available for chemical reaction, mass or heat transfer.

Table 2-1 Design details of mechanically agitated vessel

Parameter	Value
Small vessel diameter (m)	0.2
Large vessel diameter (m)	0.4
H/T	1
Baffle with	T/10
Number of baffles	4
Material of construction	Plexiglass
Sparger (both setups)	Ring sparger,
	8 holes evenly distributed d=0.75D
Impeller position (both setups)	C=T/3
Geometry (both setups)	Cylindrical with flat bottom
Impellers (both setups)	PBT-D (4 blade), D=T/3
	CBT (6 blade), D=T/3
	RT (6 blade), D=T/3

For characterizing N_{js} with the visual technique, the vessel base was illuminated and the bottom was observed while increasing the impeller speed with a low step-size of 10 rpm. N_{js} was determined according to the Zwietering criteria.

For characterizing N_{js} by the pressure gauge technique, a calibrated pressure transducer (Lucas Schaevitz Model P3061-20wg) was connected to the vessel bottom. LabView software (National Instruments) was used for data acquisition. Signals were recorded with a sampling time of 1 sec for 5 minutes. The recorded signals were then processed based on the procedure explained by Micale et, al. (Micale et al. 2000, Micale et al. 2002).

A fiber optic, consisting of two ports (one emitter and one receiver) was used for characterizing solid concentration in the slurry. When solid particles pass in front of the probe they reflect light. Reflected lights go through the fiber (receiver) and are converted to voltage by PV-4A particle velocity analyzer.

Light intensity of emitter and voltage range of analyzer were adjusted to make sure system has appropriate sensitivity. Measurements were performed with sampling time of 0.5 msec for 160 seconds for 4 axial positions and 4 radial points. Recorded data was then converted to concentration (using calibration curve) and compiled using homemade codes (Esmaeili et al. 2008). It is worth to mention that the fiber optic method is very simple but collecting data from all position in the vessel for this study is quite cumbersome (3 days for each experimental condition).

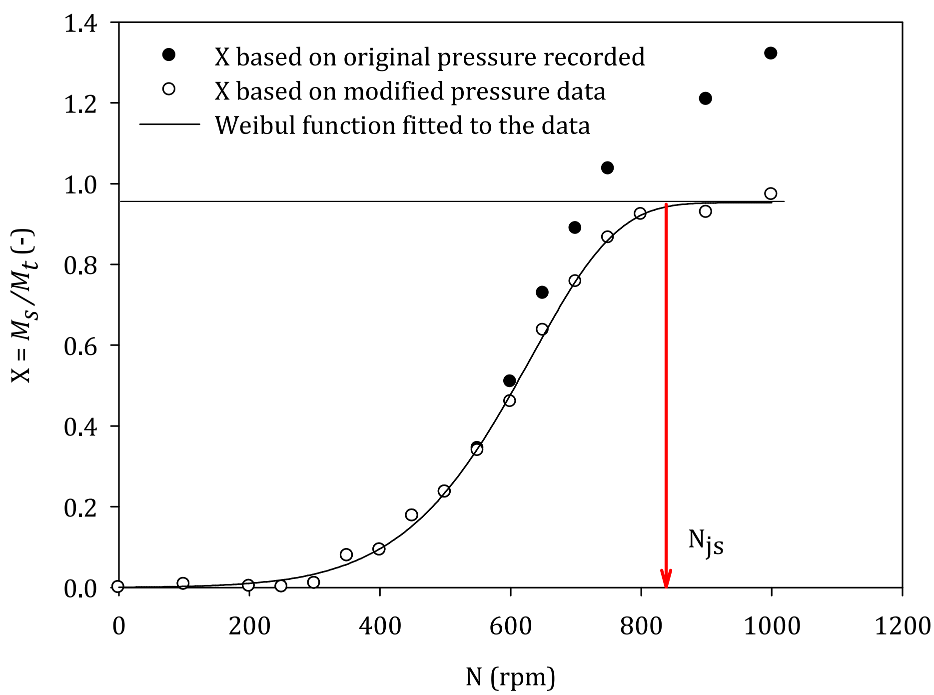

Fig. 2-3. Characterizing Njs by pressure gauge technique

Table 2-2, experimental conditions for characterization of solid distribution

Parameter	Value
Impeller	RT, CBT, PBT-D
Rotational speed (rpm)	600, 800, 950
Solid loading (% wt/wt)	10, 20, 30, 40, 50
Clearance	0.2, 0.25, 0.33, 0.37, 0.45

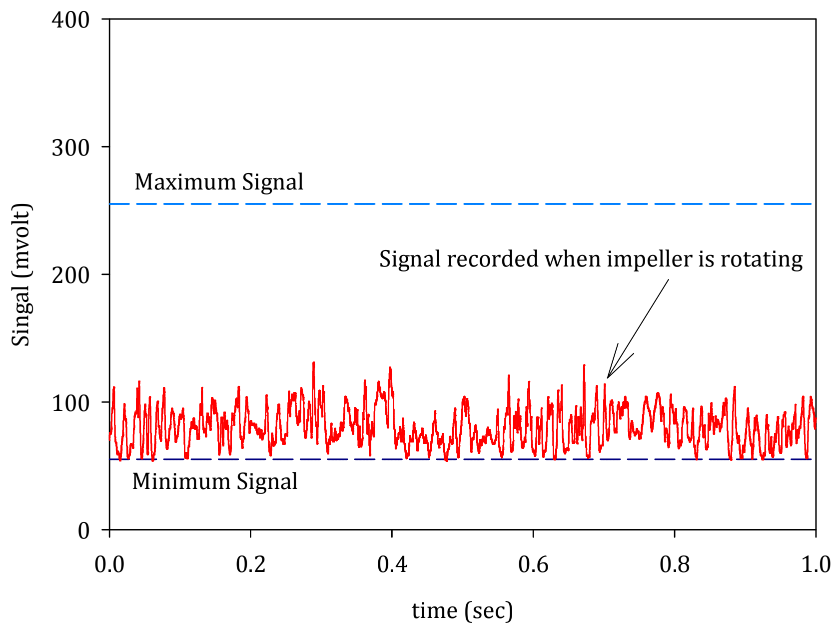

Fig. 2-4. Minimum, maximum and fluctuating recorded signals for liquid-solid mixing system, PBT-D, C/T=0.4, X=20 wt/wt%.

An example of recorded data with a fiber optic probe is shown in Fig. 2-4. Lowest voltage (constant, showing no fluctuation), is related to the condition that there is no solid in front of the probe (ε_s=0) while maximum voltage corresponds to a bed of solid fully settled (ε_s=$\varepsilon_{s,0}$). The data recorded corresponding to fluctuations of solid concentration is also shown in Fig. 2-3. A high increase of voltage was observed when the bulk of solid was reached in the measuring volume of the probe.

Gas-liquid mass transfer coefficient has been measured using the dynamic absorption-desorption technique. At the beginning of an experiment, oxygen present in the liquid was striped out using nitrogen until $C_{O2} \approx 0.5$ mg/L. At this point gas suddenly was changed to air. The time-dependent oxygen concentration was recorded after switching to air using a high precision dissolved oxygen (DO) probe (YSI model 58) and LabVIEW software. By recording the oxygen concentration versus time, measuring the saturated concentration of oxygen in the liquid phase and fitting experimental data to the mass balance equation for oxygen, the gas-liquid mass transfer coefficient can be determined (details can be found in (Sardeing et al. 2004)).

Fiber optics has been used for characterizing gas hold-up and bubble size in gas-liquid reactors. The one used in this study had one light emitter and two light receivers (d=1 mm). When gas bubbles pass in front of the probe they reflect light. Reflected light converted to voltage and amplified by a particle velocity analyzer (PV4A, Institute of Chemical Metallurgy, Chinese Academy of Science) and raw signals were acquired By PV4A software. The signals were processed by home-made codes to calculate the gas hold-up and bubble size. Measurements were carried out for 4 radial positions and 4 axial positions in the both vessels.

Nomenclature

A	*Surface area (m^2), Attenuation of vessel and environment (-)*
C_{O2}	Oxygen concentration in liquid phase (mg/l)
C	*Impeller clearance (m)*
D	*Impeller diameter (m)*
D_s	*Sparger diameter (m)*

I	*Count rate (count/sec)*
L	*Scanning length (m)*
N_{js}	*Impeller speed (1/sec) at just suspended condition for LS system*
N_{jsg}	*Impeller speed (1/sec) at just suspended condition for GLS system*
T	*Vessel diameter (m)*
X	*Solid loading Ms/Mt×100 (% wt/wt-),*
	or Mass of unsuspended solid

Greek letters

ε_s	Solid hold-up (-)
$\varepsilon_{s,0}$	Solid hold-up for fully settled bed (-)
ρ	density (kg/m3)
μ	Mass attenuation coefficient (kg/m2)

Subscribes

0	*Initial condition (N=0 rpm)*
s	*solid*

Abbreviations

CBT	*Concave Blade Turbine*
PBT-D (n)	*Pitched blade turbine down pumping flow (number of blades)*

RT (n) *Rushton turbine (number of blades)*

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CHAPTER 3

ORGANIZATION OF ARTICLES AND THESIS STRUCTURE

Chapters 4 to 7 give the main results of the thesis and corresponding scientific findings. Each of these chapters consists of an individual article. A brief description of each chapter is as follows:

- Chapter 4 presents a comprehensive guideline for designing an agitated vessel which operates at just suspension conditions
- Chapter 5 discusses a new method for characterizing just off-bottom suspension speed in LS and GLS agitated vessels and investigates the effect of operational and design parameters on Njs
- Chapter 6 examines an investigation on solid concentration distribution in an agitated vessel and evaluates scale-up procedures for LS agitated vessels
- Chapter 7 looks at the gas-liquid mass transfer coefficient in agitated vessels, evaluates scale-up procedures for gas sparged agitated vessels

Chapter 8 is a general discussion and summary of results obtained in this work. Finally, the conclusion and recommendations for future works are presented.

Chapter 4: A Comprehensive Review of Just off-bottom Suspension Speed in LS and GLS Stirred Tank Reactors *

4.1 Presentation of Article:

The objective of this article is to provide a comprehensive review on just suspended speed in liquid-solid (LS) and gas-liquid-solid (GLS) stirred tank reactors. It aims to provide the required background for scientists and engineers to design LS and GLS systems, in particular specify the appropriate geometry and operating conditions to achieve the desired quality of solid suspension (just suspended condition) for process at hand. Empirical correlations for predicting critical impeller speed for just suspended condition (N_{js}) have been compared and the effect of different parameters like physical properties of solid and liquid, impeller type, impeller clearance, vessel geometry on N_{js} have been explained. For three-phase system (GLS) effect of gas flow rate and sparger design are discussed. A case study is presented to clarify application of this information.

*Submitted to Chemical Engineering Journal, 2010

4.2: A Comprehensive Review of Just off-bottom Suspension Speed in LS and GLS Stirred Tank Reactors

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4.2.1 Abstract

For successful design and operation of Liquid-Solid (LS) and Gas-Liquid-Solid (GLS) stirred tank reactors engineers and scientists must define geometrical and operating conditions for a specific medium (specified physical properties) in such a way that provides the optimum level of solid suspension. Failure to design the stirred tank reactor to achieve optimum conditions and maintain the system at these conditions during operation may be detrimental to product quality (selectivity and yield) and cost. Successful design and operation require comprehensive knowledge about how the state of solid suspension may be affected by changing physical, operational, and geometrical parameters. Also, accurate correlations are necessary to fulfill that objective. This article intends to provide that background for scientists and engineers. It critically surveys the published work in this field and makes specific recommendations for the appropriate conditions that provide the successful operation of agitated vessels.

KEYWORDS:

Stirred tank reactor, Solid suspension, Liquid-solid, Gas-liquid-solid, Just suspended speed,

4.2.2 Introduction

Efficient solid-liquid contacting is essential for the optimization of many chemical processes. Contact modes include solid dispersion, dissolution, leaching, crystallization, precipitation,

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adsorption, ion exchange, solid-catalyzed reaction and suspension polymerization. In many processes (especially dissolution, leaching and solid-catalyzed reactions), the main objective of liquid-solid contacting is to maximize the surface area of the solid particles available for reaction or transport processes (heat and/or mass transfer). This can only be achieved by optimizing hydrodynamic conditions where solid particles move freely and do not accumulate at any point in the vessel.

Mechanically agitated vessels have been used in the chemical process industry for decades. The energy input provided by the rotating impeller enhances the mass and heat transfer rate compared to other types of contactors. For most of liquid-solid (LS) and gas-liquid-solid (GLS) mixing systems operating at the *just-suspended* condition is the minimum requirement in cases where mass transfer is controlling the process.

Inside a reaction vessel, solid particles in a liquid medium tend to settle toward the bottom as their density is usually higher than that of the liquid. In this scenario, an external force is necessary to lift the solids and retain them in a suspended state. Depending on the unit operation at hand, this force can be provided through various techniques (such as agitation in stirred tanks or gas sparging in three-phase fluidized beds). The energy input creates a turbulent flow field that lifts the solid particles from the vessel base and disperses them throughout the liquid. Solids pickup from the vessel base is achieved by a combination of 1) the drag and lift forces of the moving fluid on the solid particles and 2) the burst of turbulent eddy created in the flow bulk. The quality of solid suspension in stirred tanks is generally classified into different states. With increasing impeller speed a portion of solid particles is picked up from the settled bed of particles and become suspended. When the impeller speed is increased further, a point will be reached where a thin layer of particles is at the bottom of the vessel and moves around. At this stage generation of fillets can be observed, which corresponds to the accumulation of particles in corners or other parts of the vessel. Under this condition, because of the contact between the solid particles and vessel base, all the surface area of the solid particles is not exposed to the liquid phase. On-bottom motion is only sufficient for highly soluble solid materials. Upon reaching a specific impeller speed, all settled particles are continuously in motion on the tank bottom before becoming suspended. The bottom motion prior to suspension may involve a fraction of the settled solids coming to a brief rest before departing from the bottom into suspension. With a slight increase in impeller speed, this stoppage of solid particles is eliminated and particle-bottom contact time is shortened. The impeller speed at which this phenomenon occurs is defined as *critical impeller speed required for just off-bottom solid suspension (N_{js})*. This condition is desired for most solid-liquid applications. A gas-liquid-solid system requires the simultaneous dispersion of gas throughout the vessel and the suspension of solid particles. By increasing impeller speed (at a constant gas flow rate) the quality of gas dispersion improves and more solid particles come into suspension. Similar to the liquid-solid system there is a critical impeller speed at which solid particles do not rest at the bottom of the vessel. They move freely and become suspended. This is referred to as *critical impeller speed for just off-bottom solid suspension* in a gas-sparged system or N_{jsg}. It depends on many parameters (same as N_{js}), especially gas flow rate, sparger design and the distance between sparger and impeller. While designing the liquid-solid or gas-liquid-solid stirred tank reactor it is important to be able to determine N_{js} or N_{jsg} accurately and consider the effect of operating and geometrical parameters. Failure to operate at optimal conditions, due to uncertainty in predicting N_{js}, can be very detrimental for the process.

For example, in the gold cyanidation process, where a high concentration slurry (up to 50% wt/wt) is processed to achieve a high production rate of gold, operating at impeller speed lower than N_{jsg} will detrimentally affect the reaction selectivity and yield (Figure 4-1).

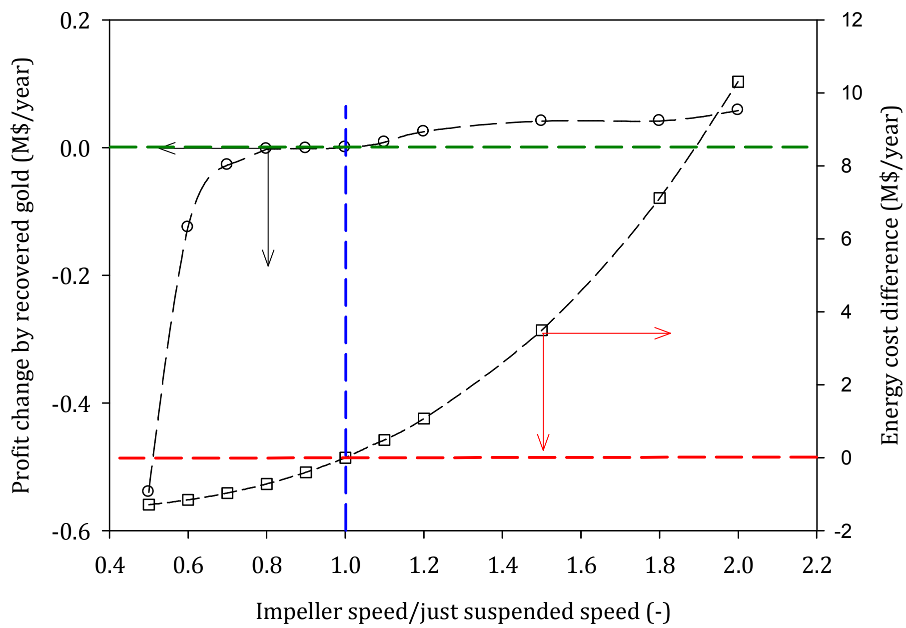


Fig. 4-1. Change in profit gained and energy cost by changing impeller speed

On the other hand, over-predicting N_{js} has significant economical drawbacks. In the gold cyanidation process 8-10 reactors in series are used to achieve high recovery of gold (up to 96%). Vessel diameter is 8 (m) with the same slurry height. As illustrated in Figure 4-1, 10% to 100% over-prediction of N_{jsg} leads to \$0.5M to \$10.3M/year of additional energy costs[2] while under-estimation of N_{jsg} leads to a decrease in gold recovery. Also, additional costs for the purchase, installation, and maintenance of larger mechanical parts should be considered. This added capital and operating cost cannot be compensated by additional gold recovered from the process.

Comprehensive knowledge about the effect of different factors (physical properties, geometrical and operational parameters) is central to the proper design and operation of LS and GLS stirred tank reactors. Although characterizing N_{js} and N_{jsg} was the subject of much research and many published scientific contributions, few articles have gathered all the information in one place. This article

[2] Cost of electricity was considered 0.06\$/kWh (for Quebec)

intends to do so and provides the proper tool for scientists and engineers to deal with the design of LS and GLS stirred tank reactors.

4.2.3. Experimental and Empirical Characterization of N_{js}

Most of our knowledge about solid suspension comes from empirical studies using experimental methods. During the last decades various experimental techniques have been developed and applied for characterizing N_{js}. The earliest and the most common method for characterizing N_{js} is the visual technique. Zwietering (Zwietering 1958) proposed and applied the visual observation method to determine N_{js}. In this method the motion of the solid particles was observed through a transparent tank wall and bottom using a mirror placed directly underneath. N_{js} was defined as the impeller speed at which no solid remains on the tank bottom for more than 1 or 2 seconds. The Zwietering method is the most common approach for characterizing N_{js} and has been used extensively (For example (Armenante and Nagamine 1998, Armenante et al. 1998, Sharma and Shaikh 2003)).

The main advantage of visual methods is simplicity. However, only with careful and experienced observation it is possible to achieve ±5% reproducibility in a diluted suspension. Further, visual methods require a transparent vessel, which is feasible for most laboratory-scale studies, but rather out of reach for large-scale vessels. To overcome the limitations of the visual technique other methods have been proposed.

Different concepts have been used for characterizing N_{js} (Kasat and Pandit 2005), like variation of power consumption or mixing time by Rewatkar et al. solid concentration change directly above the vessel bottom by Bourne and Sharma, ultrasonic beam reflection from the static layer of the solid on the vessel base by Buurman et al, pressure change at the bottom of the vessel by Micale et al. (Micale et al. 2000, Micale et al. 2002), and, recently, a novel technique has been introduced by Jafari et al. (Jafari et al. 2010) based on gamma ray densitometry.

Experimental techniques have been applied to numerous empirical and semi-empirical investigations on solid suspension, whose results have been critically reviewed in the literature and numbers of correlations were developed by employing experimental techniques (Atieme-Obeng et al. 2004, Kasat and Pandit 2005, Nienow 1992). It was shown that a significant variance appears in the prediction of N_{js} and there is no correlation with universal validity. Bohnet and Niezmak (Bohnet and Niesmak 1980) calculated N_{js} using nine correlations to find that the reported values were in the range of **-56% to +250%** from their own value.

Empirical correlations mostly describe the effect of physical properties, impeller type and scale on N_{js}. Solid and liquid density, fluid viscosity and particle size of solid particles are important physical properties that can affect N_{js}. Increasing the viscosity of the liquid does not significantly affect N_{js} since most applications of solid suspension are in the turbulent regime. This also can be concluded from the low exponent on ν in N_{js} correlations (Table 4-1). However, at high fluid viscosity or as the operating regime changes from turbulent conditions to transition, the hydrodynamics near the vessel base change and make solid pick-up more difficult.

For non-Newtonian fluids, there is a wide distribution of apparent viscosity in stirred tank reactor. It makes the hydrodynamics of the system very complicated. The impeller creates high shear rate and the apparent viscosity of a shear-thinning fluid in the vicinity of the impeller is rather low and mixing is relatively good. Under these conditions if the impeller is placed close to the bottom of the vessel, the vicinity of the impeller has a high potential for suspending solid particles, while away from the impeller mixing is poor and momentum transfer is not sufficient to suspend solid particles. Most of the researchers observed no quantifiable effect on N_{js} with increasing kinematic viscosity for a low viscous medium. There are few studies that have been done for viscous or non-Newtonian fluids (Flugg et al. 1977, Hirsekorn et al. 1953, Ibrahim and Nienow 1994, Kawase et al. 1997, Kushalkar and Pangarkar 1995).

Table 4-1 Parameters of Eq. 4-1 for predicting N_{js}

General form of Correlation: $N_{js} = S\vartheta^{\alpha}\left[\frac{g_c(\rho_s-\rho_l)}{\rho_l}\right]^{\beta} d_p^{\gamma} D^{\delta} X^{\theta}$ (Eq. 4-1)

Ref.	Value of the exponent					S	Experimental method
	Θ	α	β	γ	δ		
Zwietering (Zwietering 1958)	0.13	0.1	0.45	0.2	-2.35 (Schmidt) -1.9 (propeller)	As function of C/T and D/T	Visual technique
Nienow (Nienow 1968)	0.12	0.1	0.43	0.21	-2.21 (RT)	$(T/D)^{1.5}$	Visual technique
Baldi et al. (Baldi et al. 1978)	0.125	0.17	0.42	0.14	-0.89	As a function of Re^{*}	Visual technique
Rao et al. (Rao et al. 1988)	0.1	0.1	0.45	0.11	-1.16	$3.3(T^{0.31})$	Visual technique
Takahashi et al. (Takahashi et al. 1993)	0.22	0.1	0.34	0.023	-0.54	2	Visual technique
(Armenante and Nagamine 1998)	0.13	0.1	0.45	0.2	-0.85	As function of C/T and D/T	Visual technique
Micale et al. (Micale et al. 2002)	0.13	---	---	0.428	---	24.1	Pressure gauge technique

Ibrahim and Nienow (Ibrahim and Nienow 1994,1999,2010) investigated the effect of viscosity on the mixing pattern and solid suspension. They concluded that for low viscosity fluids (μ<100 cp) the Zwietering correlation has about a 10% uncertainty, while for more viscous systems (μ<1000 cp) the errors are greater (up to 90%). They also found that the performance of axial flow impellers are more affected by viscosity than radial flow impellers because the flow changes from axial to radial. Kawase et al. (Kawase et al. 1997) showed that shear thinning fluids could exhibit more complicated behavior. Wu et al. (Wu et al. 2001) reported a reduction in N_{js} by increasing non-Newtonian viscosity, which was explained in terms of change of the ratio of the particle settling velocity over the impeller agitation velocity caused by viscosity.

Density difference and particle size are the most important physical properties for solid suspension; whereas liquid density has only a minor effect. By increasing solid density or density difference (ρ_s-ρ_l) solid suspension will be more difficult since solid particles are less buoyant and tend to settle faster. More power will be required to pick-up solid particles and keep them suspended. With an increase in particle size settling velocity increases. Therefore, higher average liquid velocity is required to suspend particles, which leads to higher impeller speed. The effect of particle size on N_{js} and N_{jsg} has been studied by many researchers (for example: (Chowdhury 1997, Myers et al. 1994, Rao et al. 1988). Myers (Myers et al. 1994) indicated that the dependence of the just suspended speed on particle size may have two regimes: one for small particles and one for large particles. For particle size smaller than 1 mm, N_{js} is a strong function of particle size while for d_p higher than 1 mm, N_{js} increases slightly by increasing d_p. Some researchers have proposed a critical value for d_p or d_p/D where N_{js} becomes independent of particle size. Chowdhury (Chowdhury 1997) reported this critical value as dp/D equal to 0.01. There is no information about the effect of particle size distribution on N_{js}. It is expected that N_{js} for solid particles with a narrow particle size distribution is slightly lower than those with a wide particle size distribution.

The shape of the particle and its orientation in the flow affect the settling velocity and suspension condition. The only studies considering N_{js} for solid particles with different sphericities are those of Tay et al. (Tay et al. 1984) and Takahashi et al. (Takahashi et al. 1991). Tay et al. have characterized N_{js} for cylindrical particles with different lengths to diameter ratio. They have found that N_{js} is roughly independent of the size and length to diameter ratio of large cylinders. On the other hand they have reported that sphericity has a significant effect on the maximum suspension height (cloud height). Takahashi et al. (Takahashi et al. 1991) characterized N_{js} for six different particles with spherical, cylindrical, and disk-like shapes. They proposed the following correlation for N_{js},

Other physical properties of the solid and liquid that may have an effect on N_{js} and N_{jsg} are wettability of solid particles, ability of solid to trap air, agglomeration of solid particles, solid particles hardness or attrition, and presence of surface active agents.

$$N_{js} = 1.52\vartheta^{0.1} d_p^{0.04} B^{0.12} \left(\frac{g(\rho_s-\rho_l)}{\rho_l}\right)^{0.31} D^{-0.7} \qquad \text{Eq. 4-2}$$

Prediction of just suspended speed was a subject of few CFD studies (Fletcher and Brown 2009, Lea 2009, Murthy et al. 2007, Panneerselvam et al. 2008). Lea (Lea 2009) used CFD-assisted design approach to study effectiveness of mixing tank geometrical configurations to suspend particles. He developed design heuristic that can be applied in process industries. Murthy et al. (Murthy et al. 2007) used CFD simulation to study effect of different parameters on just suspended speed in LS and GLS systems. Their study covers solid loading up to 15% (wt/wt). Fletcher and Brown (Fletcher and Brown 2009) studied the influence of the choice of turbulence models on the prediction of solid suspension by means of commercial CFD codes. Kee and Tan (Kee and Tan 2002) presented a new CFD approach for predicting N_{js} and characterized effect of D/T and C/T on N_{js}. Ochieng and Lewis (Ochieng and Lewis 2006) provided qualitative and quantitative insight into solid suspension by simultaneous investigation by CFD and LDV. In their work suspension studies have been carried out in a Nickel precipitation process and best simulation results were obtained for solid loading lower than 6%.

4.2.4. The effect of geometrical parameters on N_{js}

4.2.4.1. The effect of vessel geometry on N_{js}

Most of the studies on just suspended speed have been performed in flat bottom vessels. A few work attempted to characterize N_{js} in dish bottom vessels (Buurman et al. 1985) or studied the effect of tank bottom shape (Chudacek 1985,1986, Musil and Vlk 1978). The geometry of the vessel, in particular the shape of the vessel base, affects the location of dead zones or regions where solids tend to accumulate. It also influences the minimum agitation speed required for suspending all the particles from the bottom of the vessel. In flat-bottomed vessels fillet formation tends to occur in the corner between the tank base and the tank wall. In dished bottom vessels solids tend to settle beneath the impeller or midway between the center and periphery of the base. The minimum agitation speed is typically 10% to 20% higher in a flat-bottomed vessel than a dished bottom one. Ghionzoli et al. (Ghionzoli et al. 2007) studied the effect of bottom roughness on N_{js}. They have reported that effect of bottom roughness is related to particle size. For small particles size, suspension is helped by roughness. But by increasing d_p rough bottom begin to hinder suspension.

4.2.4.2. The effect of impeller type on N_{js}

The impeller plays several roles in the stirred tank reactors; to suspend solid particles, to disperse them effectively as well as to disperse gas in GLS systems. Choosing the proper impeller to satisfy the required solid suspension (and gas dispersion in GLS system) with a minimum power requirement is the key for the technical and economic viability of the process.

There are two zones at the tank base where recirculation loops are weak: underneath the impeller and at the junction of the tank base and wall. N_{js} (also N_{jsg}) is affected significantly by the region of the vessel where the final portions of the settled solid particles are lifted into suspension. This region varies for different impeller types. Impellers are classified as axial or radial flow impellers. Flow visualization experiments confirmed that flow pattern of axial and radial flow impellers are completely different (for example(Bittorf and Kresta 2000, Kresta et al. 2001, Kresta and Wood 1993, Montante et al. 1999, Rammohan 2002)). Differences in flow pattern leads to different solid suspension mechanism. Radial flow impellers sweep particles toward the center of the vessel bottom and suspend them from an annulus around the center of the vessel bottom. On the other hand, axial flow impellers tend to suspend solid particles from the periphery of the vessel bottom. It is more difficult to lift particles from the center than drive them toward the corner. The flow pattern of axial flow impellers facilitates suspension in comparison to radial flow impellers. In most empirical correlations, the effect of impeller is included in a dimensionless parameter, i.e. S in equation 4-1.

The effect of the presence of gas on just suspended speed was studied by many researchers (for example (Frijlink et al. 1990, Murthy et al. 2007, Nienow and Bujalski 2002, Nienow et al. 1985, Rewatkar et al. 1991, Zhu and Wu 2002). An important factor in three-phase systems is how the impeller can handle solid suspension and gas dispersion simultaneously. Solid suspension in both two- and three-phase stirred vessels has been traditionally studied with conventional impellers, such as the Rushton turbine and propellers. Propellers are more susceptible to the gassing rate and result in poor gas dispersion compared to the Rushton turbine, resulting in higher values of N_{jsg} (Wiedmann et al. 1980). The Rushton turbine is superior over a propeller for both gas dispersion and solid suspension in a three-phase system. Impellers may suddenly lose their ability to suspend solid if gas is sparged to the system. Also, gas sparging might cause fluctuations in impeller power dissipation. On a large scale it may cause significant mechanical damages. A disk turbine provides stable operation with fewer fluctuations in impeller power over a wide range of gassing rates. However, the disk turbine has some serious deficiencies in gas-liquid-solid mixing operations, such as non-uniform distribution of the energy dissipation rate and gas hold-up in the vessel (Bakker and

Van Den Akker 1994, Chapman et al. 1983a). Compared to disk turbines, axial down-pumping impellers require less energy for solid suspension at low gas flow rates, but they exhibit unstable behavior in terms of large fluctuations in impeller power at high gassing rates (Bujalski 1986, Bujalski et al. 1988). The impeller speed required for re-suspending the solids after the introduction of gas increases linearly with the gas flow rate for the disk turbine, while for other impellers, this relationship is non-linear. For the disk turbine the average liquid circulation time increases by increasing the gas flow rate. Increased circulation time implies decreased linear averaged liquid velocities throughout the vessel, and this probably partly explains the linearity observed between ΔN_{js} and Q_g in the case of the disk turbine (Brayant and Sadeghzadeh 1979, Joshi et al. 1982). Compared to the disk turbine and the up-pumping pitched blade turbine, ΔN_{js} is much less for down-pumping pitched blade turbine at low gas flow rates. However, at high gas flow rates, this impeller loses this advantage and requires a significant increase in the impeller speed to re-suspend the solid particles.

The disk turbine and the up-pumping impellers were found to be relatively insensitive to the increase in the gas flow rate and exhibited stable power behavior. Hence, no sudden collapse of the suspension was observed with these impellers with an increase in the gassing rate (Bujalski 1986, Bujalski et al. 1988, Chapman et al. 1983a, Frijlink et al. 1990, Frijlink et al. 1984). The co-current gas and liquid flows help for reducing the cavity size, resulting in a smaller power drop in gassing if compared with the downward pumping impellers. As an alternative to the conventional radial and axial flow impellers some new impeller designs, like the Lightnin A-310 propeller and BX04, have been introduced (Neale and Pinches 1994, Wong et al. 1987). In comparison with other impellers, the A-310 propeller was found to be unsuitable for three-phase systems due to its easy flooding tendency even at low gassing rates. Thus, this impeller could not be used in a three-phase system, though it consumes very low power when compared to the other impellers (Wong et al. 1987). The BX04 has been found to be less sensitive to the gassing rate compared to the Rushton turbine.

While the relationship between ΔN_{js} and the gassing rate is linear for the 6-bladed Rushton turbine, it appears that the impeller speed required to suspend solids using the BX04 impeller becomes less dependent on the gassing rate as the gassing rate increases (Neale and Pinches 1994). Also, the gassed power number of the BX04 impeller was found to remain virtually unaffected in the presence of gas, which may explain the relative insensitivity of these impellers to the increased gassing rate.

Nienow and Bujalski (Nienow and Bujalski 2002) evaluated the performance of different axial and radial flow impellers. They reported that 6MFU is least sensitive to aeration rate with respect to flooding and solid suspension. The Scaba 6SRGT and up-pumping axial flow hydrofoil, A315, are also difficult to flood and lose little power when gassed. They also reported that for 6SRGT and A315, N_{jsg} is very insensitive to gas flow rate. Their investigation shows that retrofitting RTs by larger diameter A315 or 6SRGT should enable both impeller types to handle more gas without flooding. Also, for these impellers N_{js} is almost equal to N_{jsg}, which makes design and scale-up relatively easy. Although 6SRGT has higher power consumption in gassed condition compared to A315, if high rates of gas-liquid mass transfer along with high gas flow rates are required it is probably the best option. If only solid suspension is required, and liquid-solid mass transfer rate is not the limiting step in the process, impellers with low power numbers (6MFU or A315U) offer distinct energy saving possibilities, while equivalent process results could be achieved.

In general, the selection of an optimum impeller system in three-phase systems depends on the gassing rate. At a low gassing rate, down-pumping mixed flow impellers, such as the pitched blade turbine, are a good choice. If a very high gassing rate is required the disk turbine or up-pumping mixed flow impellers are adequate. The disk turbine could be a better choice due to its superior gas handling capacity compared to the up-pumping mixed flow impellers. New impellers, such as the BX04 and CBT, may be used for the solid suspension in a three-phase system as they are less affected by the presence of gas and require less power compared to the conventional radial flow disk turbine.

4.2.4.3. The effect of impeller diameter

The critical impeller speed for off-bottom suspension was found to decrease with the increasing impeller size at a constant tank diameter. The most efficient application of the disk turbine is when large turbines are used for ungassed suspension (Nienow 1968). Large impellers ($D=T/2$) provide stable operation compared to the smaller ones (D<T/3). Smaller impellers, because of their extreme sensitivity to the gassing rate, show instabilities at high gassing rates and lead to the loss of suspension. Large impellers are less susceptible to such instabilities and, hence, are good for handling suspension at high gassing rates. It has been shown that the sensitivity of the down-pumping axial flow impellers to the gassing rate was less for large diameter impellers compared to the small ones. (Chapman et al. 1983b, Nienow 1968, Rewatkar et al. 1991).

4.2.4.4. The effect of impeller clearance

The impeller clearance has substantial effects on solid suspension. Momentum transfer from the impeller to the particles is maximized in configurations where the impeller operates close to the tank base. Under this condition, the particles trapped at the bottom of the vessel underneath the impeller are driven toward the corners. This centre-to-corner motion faces minimal resistance while accumulating sufficient momentum to lift the particles into suspension after sliding toward the junction of wall and vessel base. By increasing impeller off-bottom clearance, the stagnant zone underneath the impeller increases and more solid particles are trapped in that region. Also, momentum transfer to solid particle decreases and higher impeller speed is required to force particles to move toward the tank corner from where they are suspended.

The influence of the clearance on the flow pattern has been the subject of extensive studies. For RT, Yianneskis et al. (Yianneskis et al. 1987) observed that with a single Rushton impeller (D=0.33T) the inclination of the impeller stream to the horizontal increased with decreasing clearance. Rutherford et al. (Rutherford et al. 1996) reported that with two Rushton impellers, when the lower impeller was located at a clearance of around 0.17 T or less, the impeller stream was not radial, but directed toward the bottom of the vessel. Montante et al. (Montante et al. 1999) investigated transition in flow pattern for RT by decreasing impeller clearance by means of laser-Doppler anemometry. It was found that at impeller clearance around 0.2T the double loop flow pattern of RT undergoes a transition to single loop. Montante et al. (Montante et al. 2009) studied flow regime transition for RT by means of CFD tools. They reported C/T=0.15 as the value when transition from double eight to single eight occurs.

For axial flow impellers, Jaworski et al. (Jaworski et al. 1991) measured changes in circulation pattern with a D=0.33T, PBT at C=T/2 and T/4 clearances. Kresta and Wood (Kresta and Wood 1993) also followed by (Bittorf 2000, Bittorf and Kresta 2000) reported changes in the circulation pattern with clearance for D=0.5T and 0.33T. Clearance was changed from T/20 to T/2. They observed that as off-bottom clearance was increased the angle of the flow discharge changed from the axial toward the radial direction. Mao et al. (Mao et al. 1998) observed that the downward flowing jet from a PBT-D depends strongly on clearance.

The above findings indicate that low clearance radial flow impellers may show promise for practical applications as, for instance, solids suspension may be achieved at lower energy consumption levels in comparison to the standard configuration. Ibrahim and Nienow (Ibrahim and Nienow 1996) reported that the power numbers measured with a Rushton impeller at T/6 clearance are around 25% lower than those with T/3 and T/4 clearance and observed that reducing clearance to T/6 promotes the flow from the impeller towards the tank corner. They reported that in solid-liquid mixing both the agitator speed and mean energy dissipation rate for the just-suspended condition decreased with a decrease in clearance from T/3 to T/6. Gray (Gray 1987) also investigated effect of impeller clearance on just suspended speed and power consumption in liquid-solid systems. For disk impeller, impeller clearance where flow transition can be observed reported as C/T=0.22 and 0.18 for D/W=5, 8 respectively. For axial flow impeller transition was observed at C/T=0.35. He also reported that round bottom tank promotes single-eight pattern.

Three regions have been defined in the N_{js} vs impeller clearance plot (Sharma and Shaikh 2003). In the first region, (C/T<0.1) N_{js} remains constant by increasing impeller clearance. This corresponds to the configuration where the impeller is located very close to the vessel base. This phenomenon is related to the local energy dissipated at the tank base, which remains constant when the impeller operates very close to the vessel base (Baldi et al. 1978). All impellers with very low clearance (C/T <0.1) behave like the axial flow impeller and generate a single-eight loop flow. This low-clearance range is the most efficient condition for the impellers (Sharma and Shaikh 2003). At impeller clearances higher than 0.1, N_{js} increases slightly with the clearance. For radial flow impellers, the flow pattern changes from single-eight to double-eight and, as discussed before, this could change the mechanism of solid suspension. Impeller efficiency decreases by increasing the impeller clearance and, as a result, N_{js} increases. For the axial impeller at a clearance higher than 0.35, N_{js} becomes a strong function of the impeller clearance. This increase in N_{js} is related to the modification of the flow pattern. At any clearance higher than 0.35, flow trajectories originating from the impeller hit the wall before they hit the vessel base. After hitting the wall, they slide downward or upward along the wall. This is typical of the double-eight flow pattern generated by radial flow impellers. It means that the axial flow impeller converts to radial flow impeller.

The variation of off-bottom clearance of the impeller has also a similar effect in three-phase systems to those found in a solid-liquid system (Chapman et al. 1983a, Chapman et al. 1983b). To suspend the solid, 46% savings in the power consumption could be achieved when the impeller clearance was reduced from 1/4th to 1/6th of the liquid height under ungassed conditions. As the gas flow rate was increased from 0.25vvm to 1.25vvm, the power savings decreased to 16%.

Wiedmann et al. (Wiedmann et al. 1980) reported that because of the opposing direction of the liquid flow generated by the ascending gas bubbles and the liquid flow generated by the down-flow impeller, the most favorable position of the stirrer in the two-phase system does not match that in the three-phase system. They reported that optimal conditions for the solids suspension in the three-phase system exist within the range of $1/12 < C/T < 1/1.33$. Chapman et al. (Chapman et al. 1983a) found that the disk turbine exhibits instabilities at low off-bottom clearance ($C=T/6$), which is a recommended configuration for particle suspension in ungassed systems. As the impeller off-bottom clearance is increased beyond $T/4$ flow instabilities occur, which adversely affect the solid suspension process. Chapman et al. (Chapman et al. 1983a) also found that the power savings obtained by reducing the impeller clearance of a large disk turbine (D=T/2) were relatively lower under aerated conditions in comparison to those obtained for the small disk turbine (D=T/3). This again confirmed that large impellers do not show any transition in the liquid phase flow pattern under aerated conditions. Thus, a very large impeller diameter and large impeller clearance may lead to the reversal of the liquid phase flow pattern at the vessel bottom, which adversely affects the ungassed solid suspension. A small impeller diameter and a low off-bottom clearance of the impeller are susceptible to the gassing rate and show unstable power behavior. Optimum clearance for efficient gas dispersion and solid suspension in a three-phase system is recommended as T/4 (Kasat and Pandit 2005).

4.2.4.5 The effect of liquid depth and multi-impeller systems

Liquid depth does not have a significant effect on N_{js}. However, in a system with a single impeller and a high H/T ratio the cloud height is an important parameter. In a standard agitated vessel (H=T) under just-suspended conditions, the system is not homogeneous and there is a region at the top where the solid concentration is very low. The maximum height where solid particles can be dispersed is known as the cloud height. The ratio of cloud height to liquid depth significantly increases by increasing liquid depth. For such cases, a multiple impeller arrangement is usually preferred. Single-impeller stirred tanks are also criticized for the uneven distribution of shear and energy dissipation rates, which can be harmful for some processes involving micro-organisms (bioreactors). However, at equivalent specific power, multiple impeller systems operate at a lower impeller speed, which results in lower shear values and homogenous distribution (Gogate et al. 2000). The multi-impeller system also provides better gas dispersion due to higher mean residence time and re-dispersion of the gas bubbles. Also, the use of multiple impellers has been justified to obtain good distribution of solids in a high aspect ratio three-phase stirred vessel. To satisfy a variety of mixing requirements industrial solid-liquid stirred tank reactors are usually equipped with multiple impellers. Available information on solid suspension in two-phase and three-phase stirred vessels equipped with multiple impellers is limited (Dohi et al. 2001, Dutta and Pangarkar 1995, Harnby et al. 1985, Tatterson 1994). A complete review on multi-impeller system application was provided by Gogate et al. (Gogate et al. 2000).

In a dual impeller system the lower impeller plays a key role in solid suspension. Any interference in the flow pattern of the lower impeller, due to the presence of an additional impeller above it, will result in a higher energy requirement to achieve the same state of solid suspension. N_{js} either remains constant or increases slightly with an increase in the number of impellers for the same liquid height (Dutta and Pangarkar 1995). Wu et. al. (Wu et al. 2000, Wu et al. 2001) observed that N_{js} is sensitive to impeller spacing when dual impellers are used on same shaft. They reported that N_{js} increases with increasing impeller spacing.

The variation in N_{js} with the number of impellers is also strongly dependent on the spacing between the impellers. Bakker et al. (Bakker et al. 1998), examined the effect of flow pattern on solid distribution in dual impeller system by means of CFD simulation. They reported that the second impeller hardly influences the just suspended speed. They found that If the impeller spacing is greater than the impeller diameter, the impellers behave almost independently of each other and, N_{js} for the lower impeller remains unchanged (Bakker et al. 1998). When the impeller spacing is less than the impeller diameter the flow pattern of the upper impeller interferes with the lower one resulting in an increase in the N_{js} value. If the second impeller is placed too far above the first impeller zoning may occur i.e., two impellers generate two separate and non-interacting flow loops, and the solid particles are not able to reach the upper impeller. If the spacing between impellers is equal to 3D one large flow loop extending to about 75-80% of the liquid level is generated. However, when the impeller spacing is increased to 3.7D, the flow between the impellers separates and two flow loops are observed. When a single impeller is used solids move only up to half the liquid level in the vessel. However, when a second impeller is added such that the one long loop is formed, solid particles reach the level of the second impeller and then again are re-distributed by the upper impeller in the upper part of the vessel. Baudou et al. (Baudou et al. 1997) and Mavros and Baudou (Mavros and Baudou 1997) also showed that there are three types of interaction between two impeller systems depending on impeller clearance. The first two zones have the impellers interacting with each other to different extends; however if the distance between impellers is more than 2T/3 the circulation loops do not interact. The impellers only interact if the impellers are within each others active volume.

Similar to the single impeller system, in multi-impeller three-phase systems, the introduction of gas results in a reduction in impeller power, thus requiring an increase in the impeller speed for re-suspension (Dutta and Pangarkar 1995). In a multi-impeller system the lower impeller acts as a gas distributor and the upper impellers remain relatively unaffected by the gassing rate.

4.2.5. The Effect of Process Operating Conditions on N_{js}

4.2.5.1. The effect of solid loading

As solid loading increases, a higher impeller speed is necessary to achieve the just-suspended conditions. If there are more particles present at the base, more energy is required to suspend them. The just-suspended speed is related to the settling velocity of a particle, and the settling velocity is related to the concentration. Therefore, theoretically N_{js} changes with the solid concentration. Solid loading can be expected to influence the impeller performance by modifying the suspension viscosity, local density and/or vortex structure in the vicinity of the impeller blades. It is well known that in concentrated suspensions exhibiting non-Newtonian properties the circulation around the impeller blades changes drastically.

4.2.5.2 The effect of gas flow rate,

The gas flow rate has a negative effect on solid suspension, because it decreases the pumping capacity and power input and weakens all parameters responsible for solid suspension. An increase in power input from the agitator is required for solid suspension under aerated conditions, which suggests that the presence of gas has an additional effect in terms of dampening the local turbulence and liquid velocities near the vessel base. Sometimes gas is trapped by solid particles or agglomerates, which can increase their tendency to float. At low impeller speed (and high gas flow rate) the gas is only dispersed around the impeller shaft. Solid particles rest at the bottom of the vessel and the vessel behaves like a bubble column (its behavior depends on the type of sparger and gas flow rate). If both the impeller speed and gas flow rate are low, the system operates at a minimum dispersion condition regime. If the gas flow rate is high and impeller speed is low, the system is under flooding conditions. When the impeller speed is increased (up to N_{CD}) gas will be dispersed throughout the vessel in the region above the impeller, but the quality of solid suspension may still not be appropriate (Pantula and N. Ahmed 1997). By further increasing the impeller speed beyond N_{CD}, more and more solid particles become suspended until the just-suspended condition is achieved (N_{jsg}). The solid particles are all suspended, but the system is not homogeneous. There could be a clear liquid layer at the top. The thickness of this clear layer is larger compared to that of the solid-liquid system. Increasing the impeller speed ($N >> N_{jsg}$) makes the system more homogeneous. The solid suspension process in a three-phase system is influenced significantly by the gas-filled cavities formed behind the impeller blades (Warmoeskerken et al. 1984). The cavities reduce the pumping capacity of the impeller to the point where the liquid flow generated by the impeller is no longer sufficient to keep the particles suspended.

The effect of gas sparging on stirred tank hydrodynamics has generated a lot of interest. Traditionally gas dispersion in stirred tanks is carried out using radial disk turbines such as Rushton turbine (RT) or Concave Blade turbine (CBT). There is also an increased interest in axial flow impellers for such operation. Local measurements (Aubin et al. 2004) of liquid phase velocity and CFD simulations (Khopkar et al. 2003) both indicate that presence of gas decreases liquid velocity. Decreasing liquid velocity will lead to higher N_{jsg} values. Aubin et. al. (Aubin et al. 2004) reported that the ability of impeller to provide turbulent energy does not decrease in the presence of the gas. Dutta and Pangarkar (Dutta and Pangarkar 1995) characterized N_{jsg} in multi-impeller system. They reported that ΔN_{js} is depended on gas flow rate, impeller diameter and particle parameters (d_p, X). Influence of particle parameters on N_{jsg} is similar to, but relatively weaker than, that in the ungassed case. The effect of introducing gas to the suspension process also depends on the type and geometry of the impellers. Frijlink et al. (Frijlink et al. 1990, Frijlink et al. 1984) reported similar findings for the disk turbine and inclined blade impellers. They observed that the impellers, such as the disk turbine, are less sensitive to the gassing rate compared to the pitched blade turbine. In a three-phase system, the capacity of the impeller to suspend solids is mainly determined by the impeller hydrodynamic in a gas-liquid system rather than that in a solid-liquid system.

4.2.5.3 The effect of the sparger design

A well designed stirred tank for gas dispersion, should generate a large interfacial area between the gas and the liquid phases, exhibits minimal influence of gassing on the power draw, and does not prone to flooding. These characteristics are shown to be strongly affected by the location and type of the sparger. Many studies have been dedicated to this subject (for example (Birch and Ahmed 1997, Chapman et al. 1983a)). Most of them have been done in gas-liquid systems and results implemented in GLS systems. The impeller behavior under gassed conditions is very sensitive to the gas sparging method, i.e., the type and location of the sparger (Chapman et al. 1983a, Rewatkar et al. 1991, Subbarao and Taneja 1979). The extent of the increase in N_{js} also depends on the sparger design and the sparger-impeller clearance.

Different types of sparger can be used in agitated vessels, i.e., ring sparger, pipe sparger and porous plates. The combination of the propeller or disk turbine with the ring sparger is a better option compared to the pipe sparger (Wiedmann et al. 1980). Frijlink et al. (Frijlink et al. 1990) reported a similar observation. The loss of the axial pumping efficiency and the flow regime transition (loading to flooding) occurs at much higher gassing rates with the ring sparger compared to the pipe sparger. Breucker et al. (Breucker et al. 1988) found that with large sparger rings (ds = 2.5D) it is possible to operate the impeller up to high gassing rates without flooding compared to the small sparger rings (Ds = 0.73D). Also, with the large ring sparger the decrease in the gassed power is significantly lower. In the case of the large ring sparger (Ds = 2.5D) the gassed power consumption at the same rotational speed and superficial gas velocity was found to be approximately 50-60% higher compared to the smaller ring sparger (Ds = 0.73D).

Transition in the flow pattern is strongly affected by the sparger-impeller clearance (Frijlink et al. 1990, Frijlink et al. 1984, Rewatkar et al. 1991). When the sparger is close to the impeller, the cavities are developed faster and an increase in the impeller speed is required for re-suspension when introducing gas. However, with a large sparger-impeller clearance there is a modest increase in N_{jsg}. With the sparger close to the impeller the impeller may easily become flooded resulting in sedimentation of the suspended particles. These results are contradictory with the findings of Subbarao and Taneja (Subbarao and Taneja 1979), as they have located the sparger above the impeller with holes directed in the downward direction.

Birch and Ahmed (1997) showed that larger than impeller spargers, which lead to indirect loading of the impeller, offer superior operating alternatives for gas-liquid system with implication for three-phase system. This is because indirect loading, in turn, hinders the formation of large cavities behind the impeller blades, thus ensuring relatively low power loss with aeration. However, a balance needs to be established, considering that beyond a certain point, the influence of the impeller is insufficient to maintain good overall dispersion of the gas, thus reducing the gas holdup.

For PBT-D they recommend ring sparger with (Ds>D) placed below the impeller and above the impeller for PBT-U. For RT they recommend ring sparger placed level to the impeller and close enough to be influenced by the impeller discharge. As mentioned before, the diameter and location of sparger have significant effect on the impeller performance in GLS system. Sardeing et al. (Sardeing et al. 2004) examined different sparger location for different impellers (in GL system). They recommended adequate sparger-impeller arrangement for each impeller. For RT larger sparger (D_s>D) below the impeller. For A315D Medium sparger (D_s≈D) located below the impeller was recommended.

Overall, it can be said that the sparger design and its location have a significant effect on the impeller hydrodynamics in gas-liquid systems, which in turn affect the impeller efficiency to suspend solids in three-phase systems. From the review of all the studies reported in the literature, it can be recommended that the ring sparger, with a ring diameter larger than the impeller diameter, i.e., ds = 1.5- 2 D, and placed below the impeller or in the output stream of the impeller, gives lower values of N_{js} and N_{jsg} (and thus results in less power requirement) compared to the small ring spargers and the pipe spargers.

4.2.6. Case study

To complete the guideline an industrial case has been studied to clarify the procedures required for proper design. The case under study is the gold cyanidation process. Physical properties of the system and some operational requirements are listed in Table 4-2. In the first step, the standard liquid-solid system is considered. During step two modifications for the GLS system will be made and in the last step some extra tips will be provided.

4.2.6.1 Solid-liquid system design

The gold cyanidation is a continuous process in which reaction takes place in solid particles. Reactants are cyanide (liquid phase), gold (solid phase) and oxygen (gas phase). Reactors in this process are usually stirred tanks (T=8 m, H=D). For this case study we assume that solid particles are completely wet-able, they don't have a tendency to trap air, they do not agglomerate, particle size distribution is narrow, solid particles are hard enough that attrition during the process is not considerable and a baffled vessel has been chosen. The settling velocity of solid, calculated as 0.011 m/s (2.16 ft/min), puts this solid-liquid system in the moderate category for difficulty of solid suspension (Atieme-Obeng et al. 2004). Liquid-solid mass transfer in this system is very low (Jafari et al. 2008), which indicates that partial suspension is not an adequate condition. On the other hand, reaching homogenous conditions will require a large amount of energy input (Atieme-Obeng et al. 2004). Also according to Figure 4-1 it has been shown that increasing gold recovery cannot compensate for extra energy costs if the process is being operated at impeller speeds higher than N_{js}. So it can be concluded that just off-bottom suspension is the proper operating condition for this system.

4.2.6.2 Vessel geometry

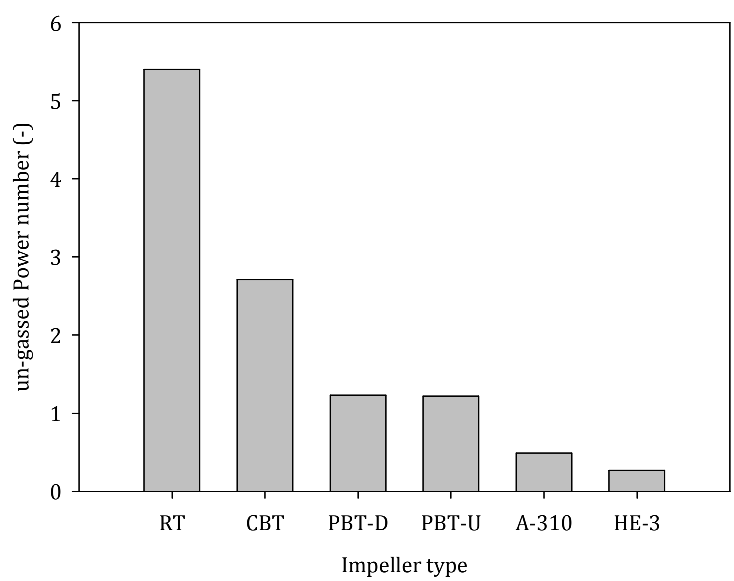
The next step is to define vessel geometry. A dished bottom vessel could be the proper choice since it may provide N_{js} 10 – 20 % lower than a flat bottom, dead zones are limited and the size of the fillet is smaller, but the size of the reactor might make the construction difficult. So for facility of construction a flat bottom vessel can be chosen.

Table 4-2 Physical properties and operational requirements of the gold cyanidation process

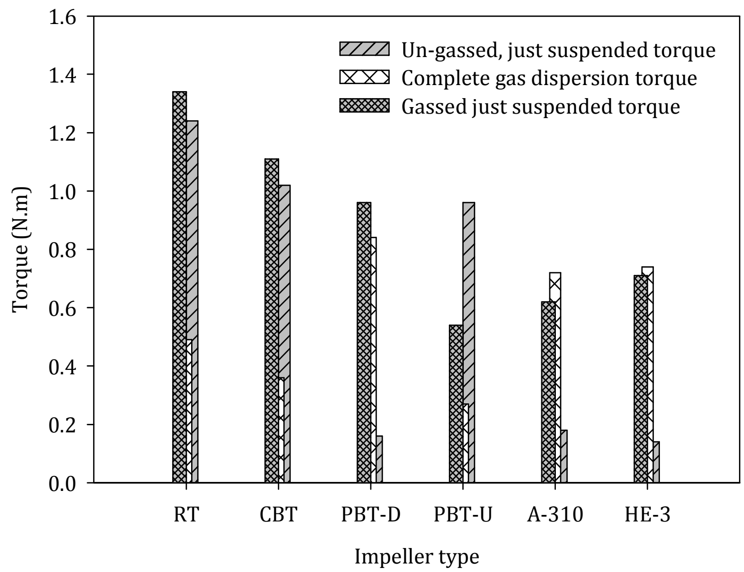
Liquid physical properties	Solid physical properties	Specific requirements
Water-cyanide solution ρ_l:1470 (kg/m^3) μ_l: 100 (cp)	Gold ore d_p: 100 μm ρ_s:3020 (kg/m^3) sphericity: ≈ 1 (-)	Oxygen should be sparged in the system Solid loading 50 (wt/wt%)

4.2.6.3 Impeller selection

For impeller selection it is important to identify which impeller can provide the required hydrodynamics for the process at lower power consumption. The most common correlation that shows global validity for predicting N_{js}, is Zwietering's equation. In Table 4-3 of N_{js} and $P_{js}/\rho V$ for different impellers are listed and compare using Zwietering's equation (Table 4-1). In addition to what is presented in Table 4-3, it is necessary to have information on cloud height, solid concentration distribution, and mixing-reaction contributions to make the proper selection.



(a)



(b)

Fig. 4-2.(a) ungassed power number of impellers in turbulent regime for different impellers, (b) ungassed just-suspended torque , complete gas dispersion torque (v_s=0.01 m/s) and gassed just-suspended torque requirements for different impellers (Lehn et al. 1999)

Table 4-3 Comparing different impeller's N_{js} for the case under study

Impeller type	N_{js} (rpm)	$P_{js}/\rho V$ (w/kg)
RT	37	0.39
CBT	41	0.35
A-310	29	0.011
A-315	31	0.033
A-320	30	0.026
PBT-D (45°)	28	0.044

Also, knowledge on power consumption in LS and GLS stirred tank reactor is central for proper design and operation. In Figure 4-2 (a & b) comparison between impellers, concerning power consumption, was illustrated.

Table 4-4 Comparing impellers for the case under study (at different impeller clearance)

Impeller type	N_{js} (rpm)	$P_{js}/\rho V$ (w/kg)	N_{js} (rpm)	$P_{js}/\rho V$ (w/kg)
	C/T=0.33		C/T=0.1	
RT	37	0.39	35	0.34
CBT	41	0.35	40	0.31
A-310	29	0.011	28	0.01
A-315	31	0.033	29	0.028
A-320	30	0.026	28	0.022
PBT-D (45°)	28	0.044	26	0.035

Table 4-5 Comparing impellers for the case under study, effect of impeller diameter

Impeller type	N_{js} (rpm) D=T/3	$P_{js}/\rho V$ (w/kg)	N_{js} (rpm) D=T/2	$P_{js}/\rho V$ (w/kg)
RT	37	0.39	26	1.06
CBT	41	0.35	29	0.95
A-310	29	0.011	21	0.03
A-315	31	0.033	22	0.09
A-320	30	0.026	21	0.07
PBT-D (45°)	28	0.044	21	0.12

4.2.6.4 Clearance and diameter modification

Clearance and impeller diameter have a significant effect on N_{js} and reactor performance. To have lower N_{js} it has been recommended to place the impeller close to the bottom of the vessel, but this displacement will cause decreasing cloud height. If clearance is higher than 0.35, N_{js} will increase significantly. By placing the impeller at lower clearances it is necessary to using a multi-impeller system to overcome the un-homogeneity of the systems. A second impeller will be necessary to maintain partial homogeneity of the system. The second impeller will not have a significant effect on N_{js}. The proper clearance of the second impeller is an important parameter. This distance should not be higher than 3D to prevent zoning in the vessel. Increasing impeller diameter will lead to decreasing impeller N_{js} but increasing power consumption. We can conclude that the proper choice for this process is a dual impeller system with the first impeller placed very close to the bottom. The second impeller could be the same type. Although the impeller with lower power consumption is favorable, mixing-reaction contribution and solid concentration distributions are parameters that should be reconsidered.

4.2.6.5 Modifications for GLS system

Sparging gas in the system will affect the ability of the impeller to suspend solid particles. For all impellers just-suspended speed will increase by introducing gas into the system. Radial flow impellers show higher stability in the presence of gas at a wide range of gas flow rates. Axial flow impellers do not show significant instabilities at lower gas flow rates; however, at high gas flow rates they become unable to suspend solid.

In the cyanidation process the oxygen flow rate is very low, which means in this case that all types of impellers could be good choices. Another parameter that should be considered, however, is the torque fluctuation on the shaft. The presence of the cavity formed behind the impeller blades induces torque fluctuation. In a large agitated vessel the magnitude of fluctuations could be high enough to damage the mechanical parts of the mixing system. In that case an impeller should be chosen that induces the lowest torque fluctuation. Ring sparger is proposed as the proper choice when it is coupled with radial flow impellers.

Table 4-6 Comparing impellers for a GLS system

	N_{jsg} (rpm)	N_{jsg} (rpm)	N_{jsg} (rpm)	N_{jsg} (rpm)
Qg (Nm^3/hr)	100	200	300	400
RT	38	38	39	40
CBT	44	45	46	47
A-310	30	31	32	33
A-315	32	32	33	33
A-320	31	32	33	33
PBT-D (45°)	29	30	30	31

If operating at a high gas flow rate the sparger diameter should be higher than the impeller diameter (1.5D – 2.5D). If the gas flow rate is not very high even the sparger with a diameter smaller than the impeller diameter (0.75D) is suitable. However, a larger diameter will ensure that torque fluctuations will be low. In addition, it is necessary to evaluate the gas-liquid mass transfer rate, liquid-solid mass transfer rate, solid concentration distribution, and cloud height in a GLS system to ensure those parameters will satisfy process requirements.

4.2.6.6 Other modifications

In the gold cyanidation process the physical properties of the solid (or slurry) may change during the process time. For example, the mineralogy or composition of the ore might change, which leads to ore density change, or the presence of clay in the ore will cause a changing viscosity or rheology of the slurry. These changes will affect the process performance and, in some cases, it will be necessary to adjust operating conditions. Increasing the viscosity of the system will increase N_{js}. In gold cyanidation based on ore composition the viscosity of the slurry could increase up to 200 cP. In this case, Njs increases to 150%. In some gold mines, the type of ore is refractory, which means that the ore contains some minerals, which increase the strength of the rock and make it very difficult to reduce the particle size. N_{js} will increase if d_p increases. As a result, it is important to compare energy savings in milling with extra energy requirements for agitation and the effect of particle size on gold recovery (final product) to be able to define which particle size is sufficient. During the milling process it is possible to achieve non-spherical solid particles. For certain ores, part of the rock can be grinded easily, but other parts are stronger. This may cause a wide range of particle size distribution after the milling process. If particle size distribution is very wide, a design can be made based on a large class of particles to assure the suspension of all solid particles.

Results of this design have been compared with some industrial plants currently operating in Canada (Quebec). Although for confidentiality concerns the details of this comparison cannot be mentioned, it is worth it to warn others that those plants are operating far below optimum conditions and as discussed in some technical reports (Jafari et al. 2008) they are suffering from bad mixing conditions. Impeller selection, sparger design and operating conditions should be modified to overcome mixing problems in those plants. The successful design of a mixing system for such complex conditions is still state-of-the-art. There are many subjects to be studied further to be able to improve the process.

4.2.7. Summary

This review presents a critical survey of the experimental and numerical works reported in the literatures. It also provides comprehensive knowledge about how the state of solid suspension may be affected by changing physical, operational, and geometrical parameters. Such knowledge is central for the successful design and operation of LS and GLS stirred tank reactors and is useful to prevent significant drawbacks concerning product quality (selectivity and yield) and costs. This article critically surveyed all the published works in this field and made specific recommendations for appropriate conditions that provide the successful operation of stirred vessels. This critical review has also identified certain gaps in the data reported in the literature. Data on solid distribution and homogeneity only covers a limited range of operating conditions. Hydrodynamic parameters in multi-impeller systems have not been studied extensively. Operating at high solid concentration is still an important industrial issue and, last but not least, CFD prediction of N_{js} and N_{jsg} is an important challenge to face in the future.

Acknowledgements

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Nomenclatures

C	*Impeller clearance (m)*
D	*Impeller diameter (m)*
Ds	*Sparger diameter (m)*
dp	*Mean particle diameter (m)*
fl	*Gas flow number: Q/ND^3 (-)*
Fr	*Impeller Froude number: N^2D/g (-)*
g	*Gravity acceleration (m/s^2)*
H	*liquid height (m)*
P	*Pressure (Pa)*
M	*Mass of solid or liquid (kg)*

N *Impeller speed (1/sec)*

N_{CD} *Critical impeller speed for complete gas dispersion (rpm)*

N_{js} *Impeller speed (1/sec) at just suspended condition for LS system*

N_{jsg} *Impeller speed (1/sec) at just suspended condition for GLS system*

Np *Impeller Power number (-)*

Q_g *Gas flow rate (m^3/s)*

Re *Reynolds number: $\rho ND^2/\mu$ (-)*

S *Zwietering correlation constant (-)*

T *Vessel diameter (m)*

W *Blade width (m)*

v_s *Superficial gas velocity (m/s)*

X *Solid loading: Ms/Mt (% wt/wt-)*

Greek letters

ν *Liquid kinematic viscosity (m^2/s)*

ρ *density (kg/m^3)*

ε_g *gas hold-up (-)*

ΔN_{js} *N_{jsg}-N_{js}*

Subscribes

i or imp	*Impeller*
js	*Just suspended*
jsg	*Just suspended in gassed condition*
l	*Liquid*
s	*solid*

Abbreviations

RT (n)	*Rushton turbine (number of blades)*
DT (n)	*Disk turbine (number of blades)*
PBT-D (n)	*Pitched blade turbine down pumping flow (number of blades)*
PBT-U (n)	*Pitched blade turbine up pumping flow (number of blades)*
MFU	*Mixed flow impeller, up-pumping*
MFD	*Mixed flow impeller, Down-pumping*

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Chapter 5: Characterization just suspension speed in solid-liquid mixing at high solid concentration with gamma ray densitometry *

5.1 Presentation of the article

The objective of this second article is to develop a new method for the accurate characterization of the just suspended speed (N_{js}) in liquid-solid mixing systems at high solid concentration. In this article the advantages and disadvantages of current measurement techniques for characterizing N_{js} have been explained and a new method is introduced to overcome their limitations. Effects of solid loading, gas flow rate, vessel size and impeller clearance on N_{js} are characterized for different impellers. An improved correlation for N_{js} calculation is proposed and a semi-theoretical approach for predicting N_{js} is introduced.

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5.2, Characterization just suspension speed in solid-liquid mixing at high solid concentration with gamma ray densitometry

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5.2.1 Abstract

The successful design and operation of Liquid-Solid (LS) and Gas-Liquid-Solid (GLS) Stirred tank reactors requires an accurate determination of the level of solid suspension needed for the process at hand. A poor design of the agitated vessel to achieve optimum conditions and maintain the system under these conditions during operation may cause significant drawbacks concerning product quality (selectivity and yield) and cost. In this paper, the limitations of applying conventional measurement techniques for the accurate characterization of critical impeller speed for just off-bottom suspension (N_{js}) at high solid concentrations are described. Subsequently, the Gamma-Ray Densitometry technique for characterizing N_{js} is introduced, which can overcome the limitations of previous experimental techniques. The theoretical concept of this method is explained and experimental validation is presented to confirm the accuracy of the Gamma-Ray Densitometry technique. The effects of impeller clearance, scale, type, and solid loading on N_{js} for several impellers are discussed. Experimental N_{js} values are compared with correlations proposed in the literature and modifications are made to improve the prediction. Finally, by utilizing the similarity to the incipient movement of solid particles in other systems, a theoretical model for N_{js} prediction is presented.

Keywords:

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Mechanically-agitated vessel, Gamma-ray densitometry, Critical impeller speed for solid suspension, Pressure gauge technique, Visual technique, Radial and axial flow impellers, High solid loading, Incipient movement of particle, Theoretical prediction of Njs.

5.2.2 Introduction

Maximum solid-liquid contact is essential for the optimization of many chemical processes. Contact modes include solid dispersion, dissolution, leaching, crystallization, precipitation, adsorption, ion exchange, solid-catalyzed reaction and suspension polymerization. In many processes (especially dissolution, leaching and solid-catalyzed reactions), the main objective of liquid-solid contacting is to maximize the surface area of the solid particles available for reaction or transport processes (heat and/or mass transfer). This can only be achieved by optimizing hydrodynamic conditions where solid particles move freely and do not accumulate at any point in the vessel. Under these conditions, the system can be described to be under "just off-bottom suspension or just-suspended" conditions.

Inside a reaction vessel, solid particles in a liquid medium tend to settle towards the bottom as their density is usually higher than that of the liquid. In this scenario, an external force is necessary to lift the solids and retain them in a suspended state. Depending on the unit operation at hand, this force can be provided through various techniques such as: agitation in stirred tanks or gas sparging in three-phase fluidized beds. The energy input creates a turbulent flow field that lifts the solid particles from the vessel base and disperses them throughout the liquid. Solids pickup from the vessel base is achieved by a combination of 1) the drag and lift forces of the moving fluid on the solid particles and 2) the burst of turbulent eddy created in the flow bulk.

Mechanically agitated vessels have been used in the chemical process industry for decades. The energy input provided by the rotating impeller enhances mass and heat transfer rates compared to other types of contactors. For liquid-solid agitated vessels, mass transfer rate is increased by increasing impeller speed. However, two contrasting trends can be observed; at impeller speeds lower than just-suspended conditions, mass transfer clearly increases with higher impeller speeds. On the other hand, the observed rate may not increase significantly with impeller speed or mixing intensity beyond the *just-suspended* condition. This indicates that operating at just-suspended conditions is the minimum requirement for processes where mass transfer is controlling the process [3]. It is therefore important to define what level of suspension is required versus the desired process results. While just suspended conditions are optimal conditions for many processes, a high degree of suspension is required for crystallization or slurry feed system. For the dissolution of highly soluble solids, partial suspension is sufficient. Failure to operate at optimal conditions due to uncertainty in predicting the impeller speed required to achieve and maintain the just suspended conditions leads to considerable drawbacks. If a mixing system operates above the minimum speed for solid suspension, the degree of suspension will be improved and the mass transfer rate will be enhanced. Higher speed, however, yields a higher turbulence shear rate, which for some processes, i.e., biological processes, may cause undesirable particle attrition or cell mortality. Obviously, there is also a practical economic limit on the maximum speed of agitation. For example, in the gold cyanidation process, where a high concentration slurry (up to 50% wt/wt) is processed to achieve a high production rate of gold, operating at an impeller speed lower than the just-suspended conditions will generate fillets in the vessel, thereby detrimentally affecting the reaction selectivity and yield. In some cases a small proportion of particles may be allowed to accumulate in corners or on the bottom in relatively stagnant regions to form fillets. This condition may offer advantages from the practical point of view because of a large savings in energy consumption compared to what is required for complete suspension. This energy savings may be more than the effect of the loss of active solids. However, it is important to quantitatively define what portion of solid is left unsuspended. On the other hand, over prediction of N_{js} causes significant economical drawbacks. For example, in the gold cyanidation process, 5 to 50% over-prediction of N_{js} leads to $150,000 to $2,200,000 /year in supplementary energy expenses. Also, the added cost for the purchase, installation, and maintenance of larger mechanical parts should be considered. This extra capital and operating costs cannot be compensated by additional gold recovered from the process. Furthermore, comprehensive knowledge about the effect of different factors (physical properties, geometrical and operational parameters) is central to the proper design and operation of LS stirred tank reactors. Although characterizing N_{js} was the subject of much research and many published scientific contributions, the subjectivity of conventional measurement techniques leads to a high degree of uncertainty in the prediction of N_{js}. It was shown that a significant variance appears in the prediction of N_{js} and there is no correlation with universal validity. Bohnet and Niezmak [5] calculated the critical impeller speed of the suspension using nine correlations and found that the reported values were in the range of **-56% to +250%** from their own values. In addition, only a few studies deal with high concentration solid suspensions in agitated vessels and current experimental methods show their limitations in terms of accuracy. For the design of concentrated systems, it is important to develop more reliable techniques for characterizing just suspended speed. In this work the Gamma-Ray Densitometry technique is proposed. It will be shown that this new technique minimizes the subjectivity of N_{js} characterization techniques and is not affected by the mixing system.

5.2.3. Background

At constant loading of solid particles, if the impeller speed is increased incrementally, bottom particles become increasingly suspended and the fraction of settled solids decreases. Upon reaching a specific impeller speed, all settled particles are continuously in motion on the tank bottom before becoming suspended. The bottom motion prior to suspension may involve a fraction of the settled solids coming to a brief rest before departing from the bottom into suspension. With a slight increase in impeller speed, this stoppage of solid particles is eliminated and particle-bottom contact time is shortened. The impeller speed at which this phenomenon occurs is defined as the critical impeller speed required for solid suspension (N_{js}). The earliest and most common method for characterizing N_{js} is the visual technique. Zwietering [56] proposed a visual observation method to determine N_{js}. The motion of the solid particles was observed through the wall and bottom of transparent tank using a mirror placed directly underneath it. N_{js} was defined as the impeller speed at which no solids remain on the tank bottom for more than 1 or 2 seconds. This method allows determining N_{js} with an accuracy of ±5% for the same observer.

However, only with careful observation is it possible to achieve ±5% reproducibility in a diluted suspension. Furthermore, visual methods require a transparent vessel, which is feasible for most laboratory-scale studies, but rather complicated for large-scale vessels. To overcome the limitations of the visual technique, other methods have been proposed. In Table 5-1, experimental methods for characterizing N_{js} have been listed. Their limitations and advantages have been explained and they are ranked based on their accuracy and applicability. Those experimental techniques were applied to numerous empirical and semi-empirical investigations on solid suspension, whose results were critically reviewed in the literature (for example [15, 44]). To provide more insight about the suspension mechanism, researchers have introduced theoretical models to predict N_{js}. These models are generally classified into different categories. The first category describes particle pickup by turbulent eddies [4], while with the second category, particles are assumed to be picked up by fluid flow [48]. There also exists a third category in which a suspension model is based on analogy to other multi-phase systems, like minimum fluidization of the gas-liquid-solid fluidized beds [29, 46]. Theoretical methods are listed and explained in Table 5-2. Although these theoretical methods are applicable for a first estimation of operating conditions, most of these methods still require empirical characterization of some parameters. There have been few efforts to predict N_{js} by means of commercial CFD codes [14, 19, 31, 39, 40]. CFD tools could provide a valuable opportunity for studying solid suspension phenomena and characterizing N_{js} but the validity of computational methods in highly concentrated turbulent flow is still questionable.

Table 5-1 Experimental methods for characterizing N_{js} (adapted from [3, 18])

Method Proposed by:	Concept	Advantages	Disadvantages	Applicability	Accuracy
Zwietering	Visual observation of particles that do not rest at the vessel bottom for more than 1-2 sec	Simplicity, Non-intrusive	Not applicable for opaque system, High uncertainty for high solid loading systems, Careful and skilled observation is necessary	1	3
Einenkel and Mersmann	Visual observation of the height of the slurry compared to the total height	Non-intrusive, Simplicity	Small particles suspended, come to the top of the tank, results in vanishing interface while larger particles are still resting at the bottom	1	5
Rewatkar et. al.	Variation of impeller power consumption by increasing the amount of solid suspended	Non-intrusive, Can be used for opaque systems	Requires accurate measurement of power consumption, expensive for large scale vessels, The criteria is not clear,	3	5
Rewatkar et. al.	Variation of liquid phase mixing time by increasing amount of solid suspended	Can be used for opaque system	Requires accurate measurement of mixing time, Not applicable for large scale vessels, The criteria is not clear, In high solid loading or three-phase systems accurate measurement of mixing time is challenging	3	5
Rewatkar et. al.	Decrease in count rate recorded from radioactive tracer inside the vessel by increasing impeller speed	Non-intrusive Can be used in opaque system	Decrease in recorded count rate could be because of tracer dispersion not just off-bottom suspension The criteria is not clear	5	5
Musil et al	Discontinuity in solid concentration close to the bottom of the vessel by increasing impeller speed	Can be used in opaque system	Intrusive, Accurate measurement of concentration is challenging	3	4

Chapman et al.	peak in solid concentration measured close to the bottom of the vessel by increasing impeller speed	Can be used in opaque system	Intrusive Accurate measurement of concentration is difficult	3	4
Buurman et al.	Use of Doppler effect at vessel bottom	Independent to material and scale, non-intrusive	Applying the technique is challenging, ultrasound sensor must be installed inside the vessel otherwise signals are scattered by wall	4	2
Micale et al.	Change in the pressure recorded at the bottom of the vessel by increasing impeller speed	Independent to material, non-intrusive	Proper selection of pressure recording port is important, Method proposed to eliminate effect of dynamic pressure head is not accurate	2	3

Accuracy: 1: most accurate, 5: least accurate, Applicability: 1: easiest to apply, 5: most difficult to apply

Table 5-2 Theoretical methods for predicting N_{js}

Reference	**Concept**	**Remarks**
Kolar (1961) [20]	Energy necessary to suspend particles equals the energy dissipated by the particle moving at its terminal velocity in a still fluid	In a turbulent fluid the settling velocity of a particle is different from that in a still fluid. Very simple model, unable to precisely predict N_{js} Assumptions are more likely similar to homogenous suspension rather than just suspended conditions
Baldi (1978) [4]	Particles are picked up and kept suspended by turbulent eddies	Cannot describe the effect of viscosity nor the effect of solid concentration Cannot describe why the impeller which creates mass circulations (PBT) are more effective for suspending particles than impellers which create a lot of turbulence
Narayanan et al. (1969) [35]	Balance of vertical forces acting on particles	Assumption of no slip between solid and liquid and homogenous distribution of solid particles is questionable. Proposed for very diluted solid concentrations

Subbarao and Taneja (1979) [51]	Balance of forces acting on particles	Particle settling velocity was estimated from a correlation for the porosity of a liquid fluidized bed as a function of liquid velocity
Ditl and Rieger (1985) [9]	Same concept as Baldi et al. [4], Solid particles are picked up by different sizes of eddies	Cannot describe the effect of viscosity nor the effect of solid concentration Cannot describe why the impeller which creates mass circulations (PBT) are more effective for suspending particles than impellers which create a lot of turbulence
Musil and Vlk (1978) [32]	Balance between liquid and particle kinetic energy	The approach followed by them was rejected by Ditl (1980) [8] because of mathematical mistakes
Shamlou and Zolfagharian (1987) [48]	Proposed a model for estimation necessary conditions for incipient motion of solid particles based on average velocity of the liquid near the bottom of the vessel and forces acting on particles, like lift, drag, buoyancy and weight resting at the bottom of the vessel.	Model doesn't need any experimental adjustment but the parameter describing solid arrangement is unknown.
Molerus and Latzel (1987) [28, 29]	Solid suspension governed by two different mechanisms based on Archimedes number. Region responsible for solid suspension is the wall boundary layer of the vessel.	Requires accurate correlation for predicting shear rate at the boundary layer of the vessel
Wichterle (1988) [53]	Difference between the terminal settling velocity of particle and velocity of the liquid.	The ratio between N_{js} and settling velocity allows predicting N_{js} easily
Mersmann et al. (1998) [25]	Power input dissipated by two phenomena: consumption of power to avoid settling and generating discharge flow for suspension.	Values for N_{js} calculated by this method are highly under-predicted compared to experimental data. This could be because of correlations for fluctuating velocity at the bottom of the vessel is not accurate

5.2.4. Materials and Methods

5.2.4.1 Experimental setup

Experiments were conducted in a 14 L transparent polycarbonate agitated cylindrical vessel with standard baffles (Fig. 5-1), an open top and a flat bottom. Three different impellers were tested, mounted on central a central shaft, namely a six blade Rushton turbine (RT), a concave blade turbine (CBT), and a four-blade pitched blade turbine in down-pumping mode (PBT-D). The vessel, impeller dimension, and geometrical details of the mixing system are given in Table 5-3. Water was used as the liquid phase and sand as the solid phase (density of 2650 kg/m^3). Particle size distribution of sand was measured by the Horiba laser scattering particle size distribution analyzer (model: LA-950). The mean particle size was 277μm. The operating slurry height was set equal to the vessel diameter.

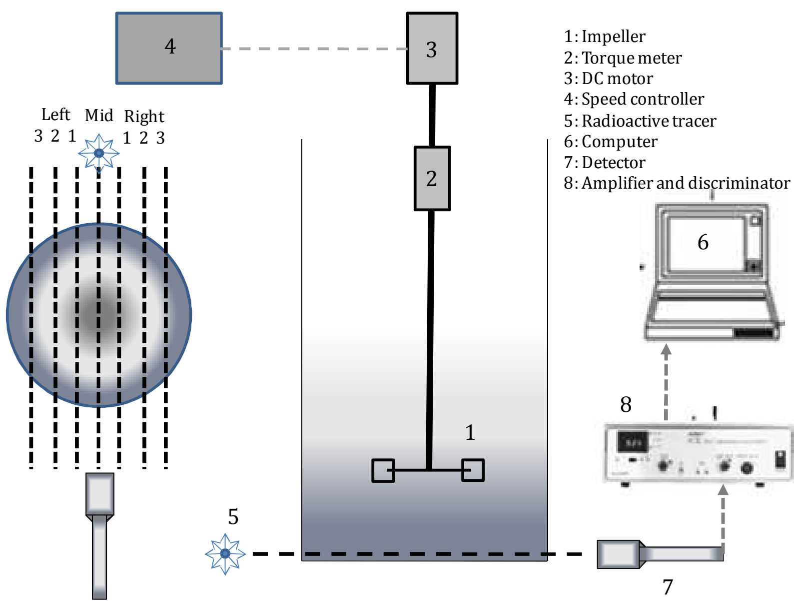

Figure 5-1. Experimental setup and gamma ray densitometry data acquisition system.

5.2.4.2 Methods

The use of radioactive sources (radioisotopes) to characterize the fluid dynamics and hydrodynamics of single-phase and multi-phase systems has an extensive history. A detailed review of these methods can be found in Chaouki et al. [6]. In the present work, we use the concept of densitometry described and applied above [6, 10] to propose a new technique for characterizing N_{js}. If a radioactive source is placed on one side of the vessel and a detector on the other side, based on the material between them the detector receives a specific amount of gamma ray. This phenomenon can be modeled by the Beer-Lambert's law that describes the decay in intensity of the emitted gamma ray by passing through the medium: $I=I_0.\exp(-\rho\mu l)$. Changes in the density or phase of the medium lead to corresponding changes in the gamma-ray intensity recorded by the detector. In multi-phase systems, the ray intensity is related to the volume fraction of each phase. This gamma-ray emission-obstruction-detection framework could be the basis of a useful tool for characterizing solid suspension in agitated vessels.

Table 5-3 Design details of a mechanically agitated vessel

Parameter	Value
Vessel diameter (m)	0.2
H/T	1
Baffle with	T/10
Number of baffles	4
Material of construction	Plexiglass
Geometry	Cylindrical with flat bottom
Impeller clearance from the bottom	Varies between 0.5T to 0.2T
Impellers	RT (6 blade), D:T/3
	CBT (6 blade), D:T/3
	PBT (4 blade), D:T/3

In practice a source of gamma ray consisting of a 2 mm glass bead filled with scandium oxide was activated in the Slowpoke nuclear reactor of Ecole Polytechnique of Montreal. The source activity was 150 μCi and the half-life time of the tracer was 84 days. The tracer was placed in the holder where it was completely shielded by lead. Emitted gamma rays from this source were collimated by lead support. It passed through a 5 mm hole on the protection shield and went through the vessel. A NaI scintillation detector (Teledyne Isotope, Model S-1212-I) was placed on the other side of the vessel and coupled to an amplifier (EG&G ORTEC Model: 925-SCINT) and a data acquisition system (TOMO MSC plus-17) – See Fig. 5-1. Both tracer and detector were positioned in order to be able to scan the region about 0.5 cm from the bottom of the vessel. The signals were recorded for 2 minutes with a 200 msec sampling time at each impeller speed (varied between 0 to 1000 rpm with different step sizes). Counts were recorded at each impeller speed and they were converted and processed by home-made codes. It was verified that changing the sampling time and recording period as well as the background noise did not alter the experimental results. The original recorded count rates were related to the solid volume fraction. For this purpose, the same region was scanned without solids (pure water). According to this procedure, the following equations can be established. Equation 5-1 relates the measured intensity to solid hold-up.

$$I_{liquid-solid(N>0)} = I_0 exp\left(-\rho_{water}\mu_{water}L(1-\epsilon_s) - \rho_{solid}\mu_{solid}L(\epsilon_s)\right) \cdot exp(-A) \qquad \text{Eq. 5-1}$$

N_{js} was also characterized by two conventional techniques for comparison: the visual technique and the pressure gauge technique. For characterizing N_{js} with the visual technique, the vessel base was illuminated and the bottom was observed while increasing the impeller speed with a low step-size of 10 rpm. N_{js} was determined according to the Zwietering criteria. For characterizing N_{js} by the pressure gauge technique, a calibrated pressure transducer (Lucas Schaevitz Model P3061-20wg) was connected to the vessel bottom. LabView software (National Instruments) was used for data acquisition. Signals were recorded with a sampling time of 1 sec for 5 minutes. The recorded signals were then processed based on the procedure explained by Micale et, al. [26, 27]. In the experiments, various solid loading and impeller clearance conditions were investigated. The effect of the gas flow rate on solid suspension was studied as well. Different scale-up procedures were evaluated to identify which procedure may provide proper scale-up conditions. All experiments were repeated at least three times to ascertain the reproducibility.

5.2.5. Results and discussion

5.2.5.1 Main features of solid suspension

Typical results of the densitometry technique are shown in Fig. 5-2. This figure shows variation of count rate recorded by detector vs. impeller speed. At N = 0 (rpm), when all the solid particles settled on the bottom of the vessel, the recorded intensity by the detector $I_{N=0}$ is constant. By increasing the impeller speed and as solid particles in the scanning region commence motion and are lifted by the liquid, the recorded intensity increases. At higher impeller speeds, when all the solid particles are experiencing random motion and no solid rests on the bottom of the vessel, the recorded intensity is expected to stabilize. In practice, a slight intensity increase can be observed, which is related to the change in solid particle speed and a decrease of the residence time of the solid in the scanning zone.

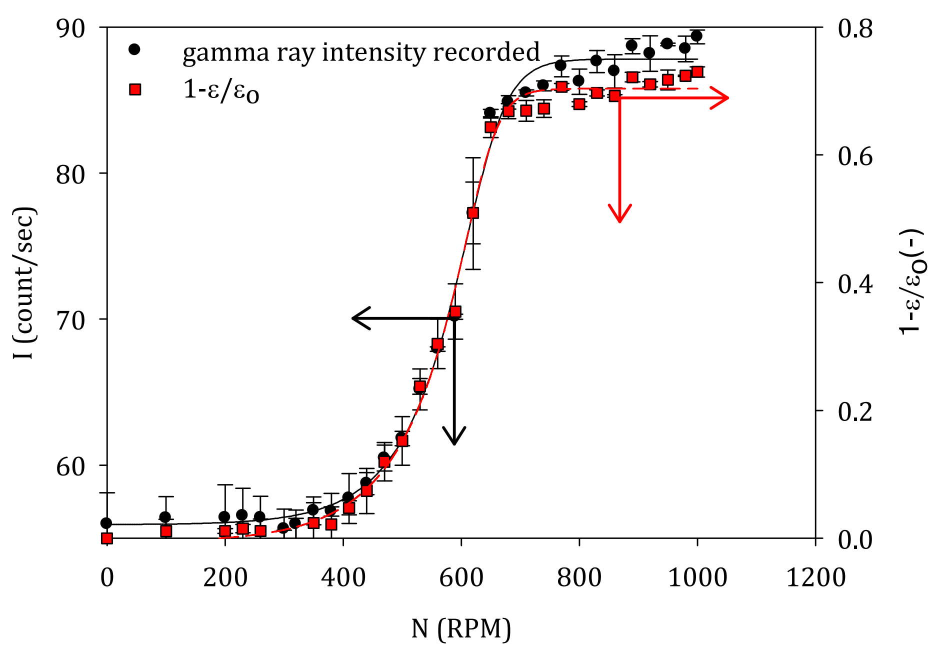

Figure 5-2. Variation of recorded count rate and average solid hold-up by increasing impeller speed at the bottom of the vessel. Impeller: RT, X: 20%, d_p: 277 μm, C/T: 0.33.

Solid hold-up can be calculated from recorded count rates by employing equation 5-1. Fig. 5-2 also illustrates the variation of solid hold-up at the bottom of the vessel by increasing impeller speed.

As many researchers have mentioned (for example [33]) solid concentration at the bottom of the vessel at just suspended conditions exhibits a discontinuity. As shown in Fig. 5-2, based on densitometry data, a discontinuity in solid concentration can be noticed at the bottom of the vessel. The starting point of this discontinuity is considered as N_{js}. $\varepsilon/\varepsilon_0$ represents the normalized solid volume fraction at the bottom of the vessel. By plotting $1-\varepsilon/\varepsilon_0$ vs. impeller speed the discontinuity in solid concentration at the bottom of the can be identified clearly. As illustrated in Fig. 5-2, for low impeller speed, all solid particles rest on the bottom of the vessel base. Upon increasing impeller speed, a fraction of the solid particles commences lifting and reaches suspension at a certain height. Partial suspensions correspond to the situation where some solids rest on the bottom of the tank. Since the particles are in constant contact with the bottom of the vessel, not all the surface area of particles is available for chemical reaction, mass or heat transfer.

As the impeller speed is increased, the partially suspended solid yields three distinct zones: a clear liquid layer at the top; a non-suspended solid layer at the bottom; and a region with a suspended mixture in between. The relative size of the three zones depends on how easily particles can be picked up by the fluid and how efficiently the impeller is agitating the liquid. Increasing impeller speed results in conditions where no particle stagnates at the bottom of the vessel. Although virtually all solid particles are suspended, the system is not yet homogeneous, with a clear interface between the solid-rich and solid-lean regions. By increasing impeller speed beyond the just suspended condition, the degree of homogeneity reaches a maximum value.

5.2.5.2 Comparing densitometry with the pressure-gauge and visual observation techniques

Results of the gamma ray densitometry technique were compared with those of the two conventional techniques in Fig. 5-3.a and 5-3.b. As explained in previous section (5.2.4.2) for determination of N_{js} by visual technique, the bottom of the vessel was monitored and for pressure gauge technique, the pressure at the vessel base was recorded when impeller speed was increased incrementally from 0 to 1000 rpm.

As illustrated in Fig. 5-3.a the pressure-gauge technique quite systematically overestimates the just-suspended speed compared to the densitometry technique. This could be related to the fact that the method for eliminating the dynamic head effect is not valid for high solid concentration and for axial flow impellers. As discussed by Micale [26, 27] the dynamic head effect can perturb experimental data significantly. At low solid loading, N_{js} determined by the pressure-gauge technique, and the visual method are in good agreement. Differences in N_{js} values obtained using the different methods do not exceed 5%. This difference is well within the range of experimental uncertainty. However, for high solid loading and special cases like an axial flow impeller or low off-bottom impeller clearance, both conventional techniques exhibit larger differences compared to the new method.

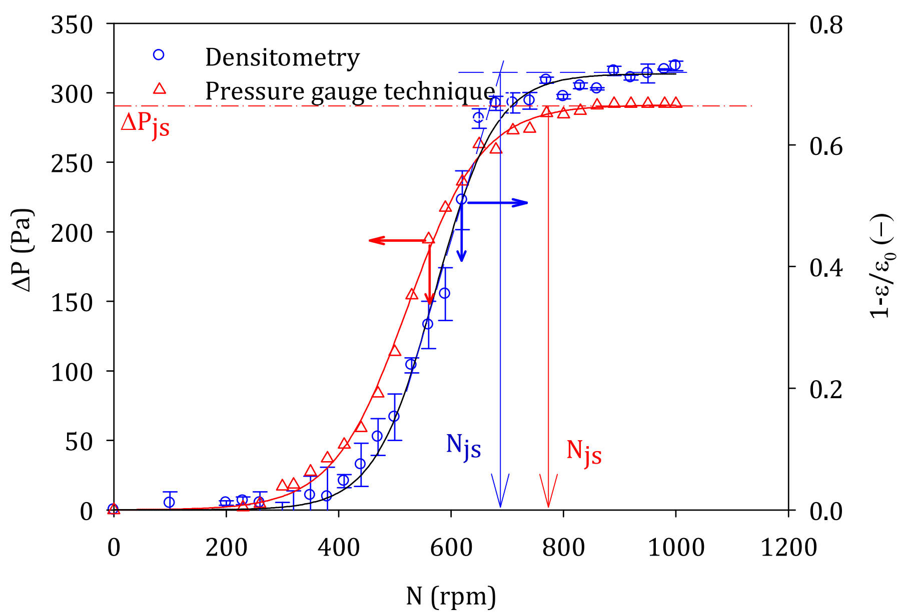

Figure 5-3.a. Comparison of gamma ray densitometry technique with pressure technique.

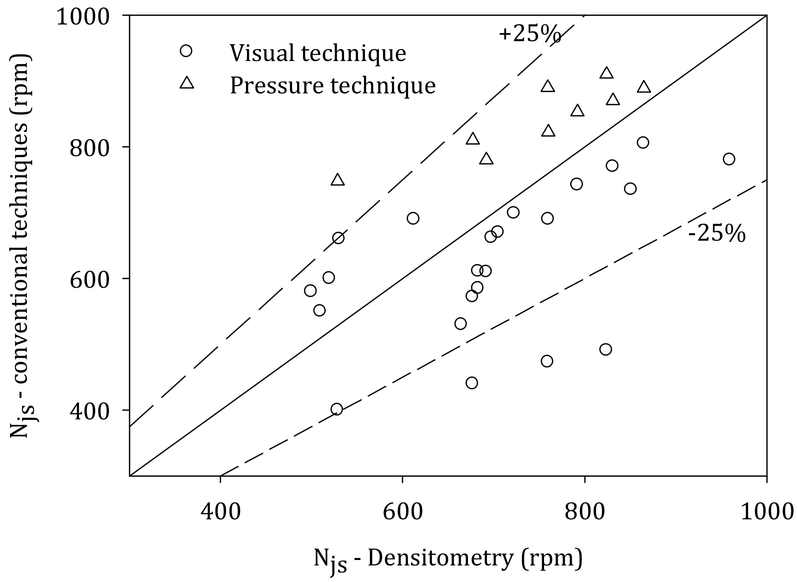

Figure 5-3.b. Comparison of gamma ray densitometry technique with two conventional methods.

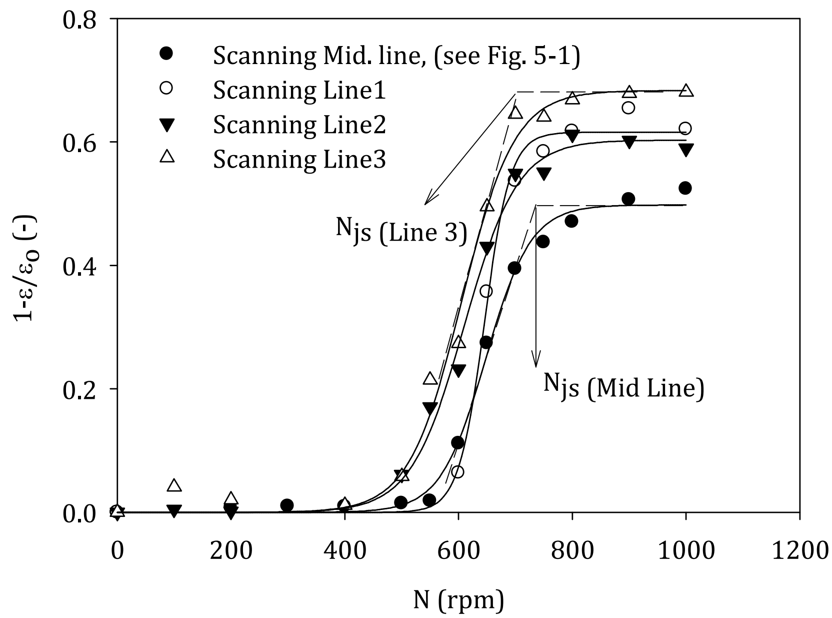

Figure 5-4.a. Solid hold-up variation at the bottom of the vessel by increasing impeller speed, for 4 different scanning lines, Impeller: RT, X: 30%, dp: 277 μm, C/T: 0.33.

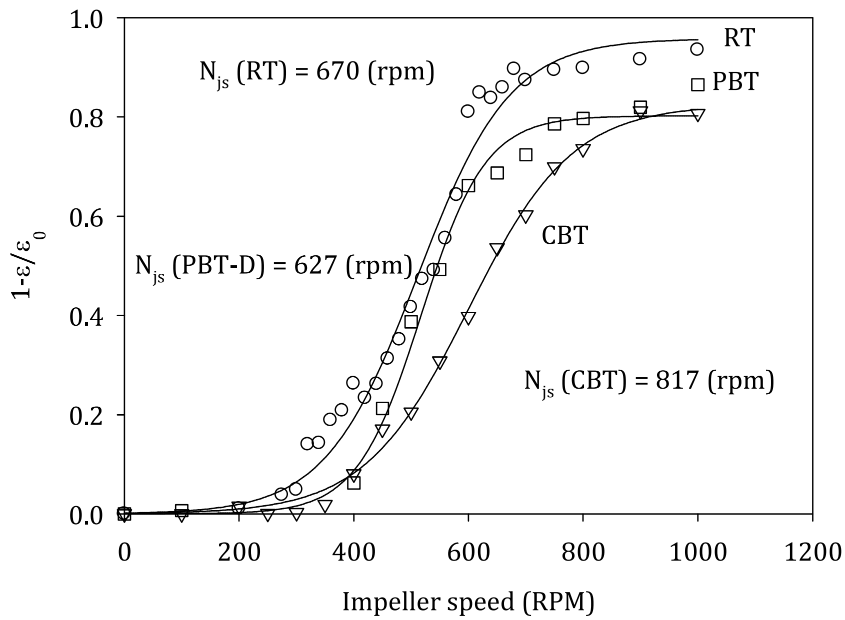

Figure 5-4.b. Comparison of just suspended speed for different impellers.

X: 10% wt/wt, , C/T: 0.33.

5.2.5.3 Effect of impeller type

The degree of solid suspension in agitated vessels is strongly related to the specific power, pumping capacity and flow pattern. The main source of power dissipation and pumping is the impeller rotation. Researchers have studied a variety of impellers for solid suspension. The choice of a given impeller to achieve maximum solid suspension with minimum power requirement is the key for the technical and economic viability of the process. N_{js} is affected significantly by the region of the vessel where the final portion of settled solid particles is brought into suspension. This region varies for different impeller types and vessel geometry.

Three types of impellers have been studied in this article: Rushton Turbine (RT), Pitched Blade Turbine in down-pumping mode (PBT-D) and Concave Blade Turbine (CBT). Axial flow impellers (like PBT-D) are more favorable for liquid-solid mixing processes since they can provide a good quality of solid suspension at lower impeller speed compared to radial flow impellers [2, 3], but their instability for being applied in a three-phase system (Gas-liquid-solid) leads us to study RT and CBT as well.

There are two zones on the tank base where recirculation loops are weak: underneath the impeller and at the junction of the tank base and wall. As illustrated in Fig. 5-4.a for the Rushton turbine, the final settled solids were suspended from underneath the impeller at the centre of the tank. At the same impeller speed, more solids were suspended from other regions compared to the centre. There are many characterization studies regarding the flow pattern of radial and axial flow impellers (for example [1, 22, 23]). The radial flow generated with radial flow impeller first hits the wall and change direction, moving upward and downward [21, 23]. Downward jet hits bottom of the vessel and is redirected to the center. Thus, the radial flow impeller sweeps particles toward the center of the vessel bottom and lifts them from an annulus around the center of the vessel bottom. As illustrated in Fig. 5-4.a solid concentration is lower at third scanning line, which corresponds to periphery of the vessel, compared to Mid. line.

On the other hand axial flow impellers tend to suspend solid particles from the periphery of the vessel bottom. The flow generated by axial flow impeller (in down pumping mode) first hits the bottom of the vessel. It is then redirected to the wall and generates liquid wall jet moving upward, which could push solid particle forward and lift them from the periphery of the vessel [16, 22, 47, 54]. Wall jet generated by axial flow impeller at the wall is stronger than the one generated by radial flow impeller. Accordingly it is more difficult to lift particles from the center than drive them toward the corner. The flow pattern of axial flow impellers facilitates suspension in comparison to radial flow impellers. As shown in Fig. 5-4.b, radial flow impellers require higher impeller speeds for solid suspension compared to axial flow impellers.

5.2.5.4 Effect of solid loading

The effect of solid concentration on N_{js} has also been studied. As shown in Fig. 5-5.a, higher solid loading causes an increase in required N_{js}. Upon increasing solid loading, more power is required to suspend large portions of solid. In the plateau region (Fig. 5-5.a), solid hold-up at the bottom of the vessel is higher at X=40 (% wt/wt) compared to the other case, because the power draw of the impeller is not high enough to disperse the solid. The effect of solid loading on N_{js} for the various impellers is summarized in Fig. 5-5.b.

5.2.5.5 Effect of impeller clearance

The effect of the impeller clearance on the just-suspended speed is illustrated in Fig. 5-6.a and 6.b. Experimental results show that the clearance has a substantial effect on solid suspension especially for PBT-D. Critical impeller speed for off-bottom suspension increases as the clearance is increased. Based on impeller clearance and type of impeller two radically different flow patterns could be observed: (1) a double-loop shape in which two recirculation loops circulate above and below the impeller, and (2) a single-loop shape in which the lower recirculation loop is suppressed. Single-loop flow is typical for axial flow impellers while double-loop is typical for radial flow impellers. Variation of the flow pattern leads to different solid suspension regimes, which, in turn, affect N_{js} as discussed previously. Energy transfer from the impeller to the particles is maximized in configurations where the impeller operates close to the tank base [2]. When the impeller is placed close to the vessel base, the particles trapped at the bottom of the vessel underneath the impeller are initially driven toward the corners. This centre-to-corner motion faces minimal resistance while accumulating sufficient momentum to lift into suspension after sliding to the junction of wall and vessel base. By increasing the impeller off-bottom clearance, the stagnant zone underneath the impeller increases, more solid particles are trapped in that region as less momentum is transferred to the particles. A higher speed (more power) is necessary to force particles to move toward the tank corner from where they become suspended. Fig. 5-6.b illustrates the variation of solid hold-up at the bottom of the vessel for radial flow and axial flow impellers at two different impeller clearances.

The variation of N_{js} as function of impeller clearance is shown in Fig. 5-6.b. Sharma and Shaikh [49] have defined three regions in the N_{js} vs. impeller clearance plot. In the first region, N_{js} remains constant by increasing impeller clearance. This corresponds to the configuration where the impeller is located very close to the vessel base. This phenomenon is related to the local energy dissipated at the tank base, which remains constant when the impeller operates very close to the vessel base [4]. Impellers exhibit a high efficiency for suspending solid particles in this region. According to Sharma and Shaikh [49] this phenomenon can be observed only in conditions where C/T<0.1. All impellers with very low clearance (C/T <0.1) behave as axial flow impellers and generate a single-eight loop flow. This low-clearance range is the most efficient condition for the impellers.

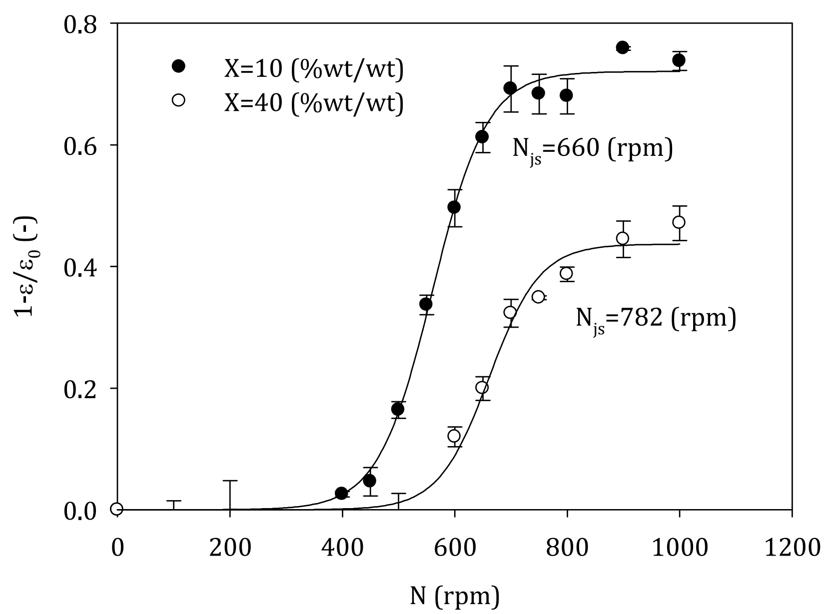

Figure 5-5.a. Variation of solid hold-up at the vessel bottom for low and high solid concentration, Impeller: RT, C/T=0.33.

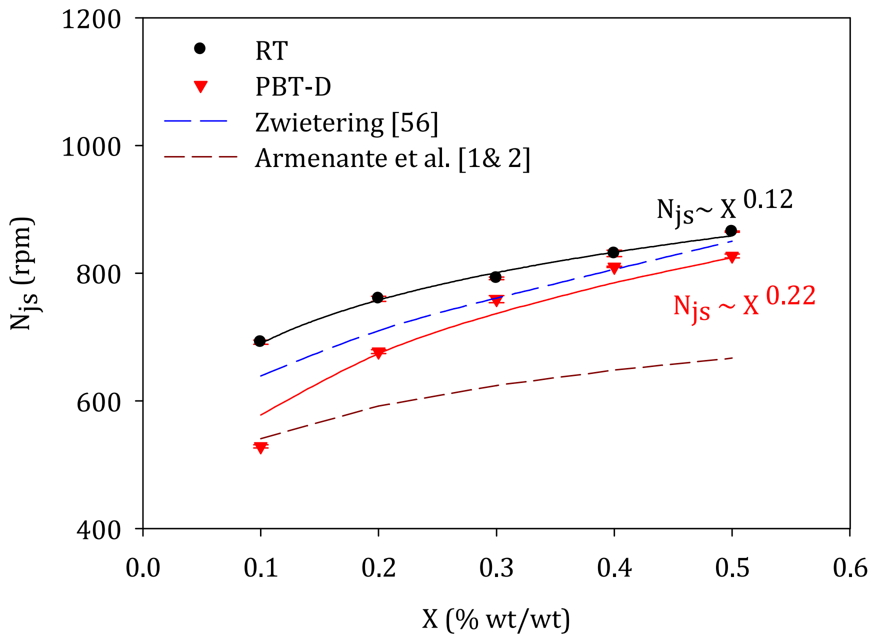

Figure 5-5.b. Variation of N_{js} by increasing solid concentration, C/T: 0.33.

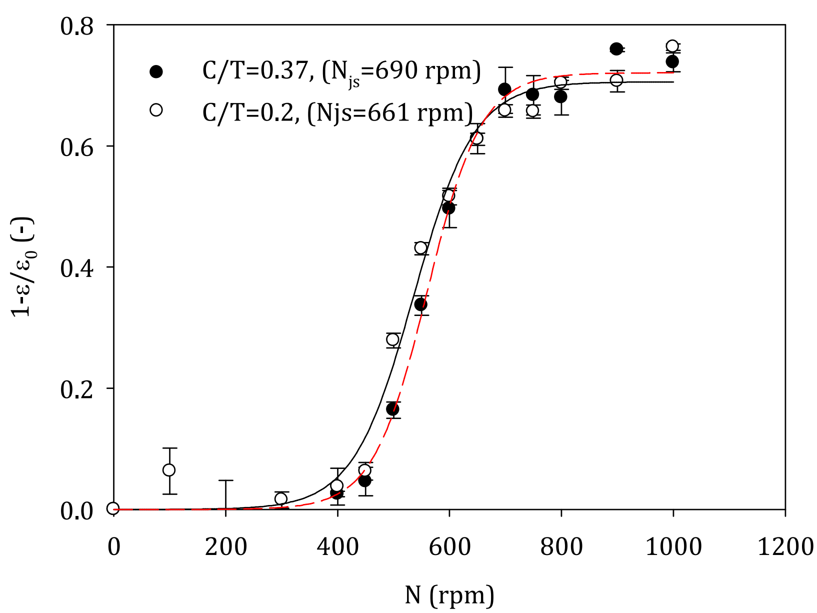

Figure 5-6.a. Variation of solid hold-up at the vessel bottom for low and high impeller clearance,

Impeller: RT.

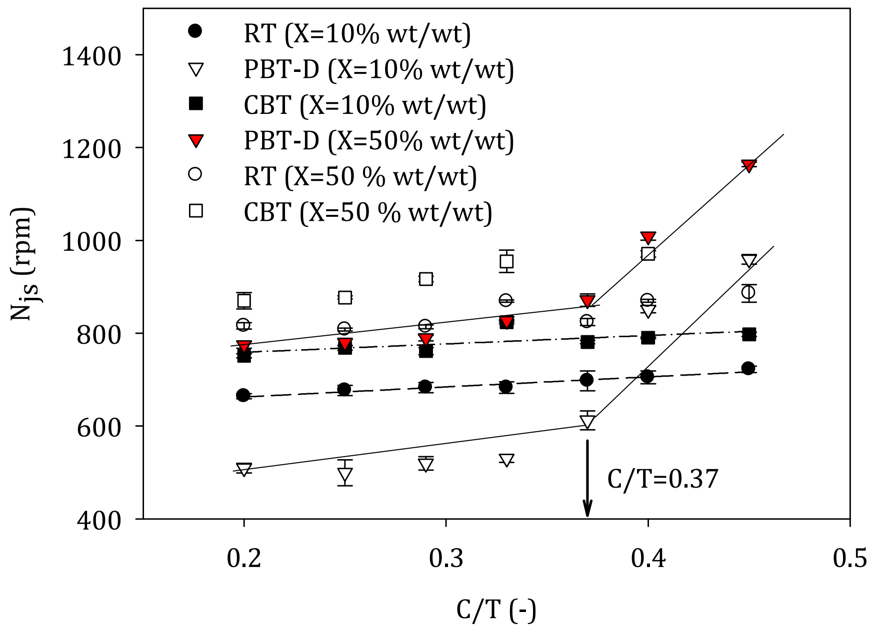

Figure 5-6.b. Variation of N_{js} by impeller clearance.

The difference between N_{js} for different impellers is related to the amount of power dissipated by the impeller in the vessel. As illustrated in Fig. 5-6.b, at impeller clearances higher than 0.2, N_{js} increases only slightly with increasing clearance. For radial flow impellers, the flow pattern changes from single-loop to double-loop [30] and, as discussed before, this changes the mechanism of solid suspension. At higher impeller clearance, the trend is similar. Impeller efficiency decreases by increasing the impeller clearance and, as a result, N_{js} increases.

For the axial flow impeller, a different behavior is observed. At impeller clearances lower than 0.37, N_{js} increases slightly as the clearance increases. This is observed for the same reason as with radial flow impellers with the added feature that at clearances higher than 0.37, N_{js} becomes a strong function of the impeller clearance and the slope in this region is higher compared to other regions. This increase in N_{js} is related to the modification of the flow pattern. At clearances higher than 0.37, flow lines originating from the impeller hit the wall before they hit the vessel base. After hitting the wall, they slide downward or upward along the wall. This is typical of the double-eight flow pattern generated by radial flow impellers. It means that the axial flow impeller converts to a radial flow impeller. Such an increase cannot be observed for radial flow impellers (RT and CBT). For radial flow impellers N_{js} slightly increases since the power available for solid suspension decreases by increasing impeller clearance.

Flow visualization can provide more insight about this phenomenon. As reported in different articles [22, 30, 34], flow transitions occur by increasing impeller clearance for both radial and axial flow impellers. In case of radial flow impeller (RT) transition from double-eight flow patter to single eight happens at C/T=0.15 [30]. When the clearance value is less than or equal to 0.15T the strong inclination of impeller stream can be utilized to promote solid suspension from bottom of the vessel. With clearance higher than 0.15T a double eight flow pattern is produced (for example [21]) and it was shown that an annular wall jet exist at the wall of the tank. By increasing the impeller clearance, downward wall jet weakens, which explains the higher impeller speed required for off-bottom suspension.

For axial flow impeller, at critical value of impeller clearance, impeller's discharge flow will impinge on the vessel wall rather than the base, which leads to two flow loops in the vessel. The primary flow loop moves upward the wall. The secondary flow loop is characterized by low-velocity, radially-inward flow at the base of the vessel, which returns to the impeller via up-flow at the center of the vessel. This flow pattern which is known as reveres flow is not well-suited for solid suspension. Different values have been reported for clearance at which axial impeller undergoes a distinct transition. It is strongly affected by type of impeller, D/T ratio and impeller blade angle [22, 34]. The critical value for PBT-D reported as C/T=0.37 (Fig. 5-6.b) in this work.

5.2.5.6 Comparing with correlations

Different correlations have been proposed for predicting N_{js} (with general format of Eq. 5-2). However, no correlations with global agreement have been presented so far. As illustrated in Fig. 5-

7, the agreement between the prediction and experimental data is not good, which means there is no equation with global validity.

$$N_{js} = Sv^{\alpha}\left[\frac{g_c(\rho_s - \rho_l)}{\rho_l}\right]^{\beta} d_p^{\gamma} D^{\delta} X^{\theta} \qquad \text{Eq. 5-2}$$

The subjectivity of conventional experimental techniques causes significant differences between predicted values for the same system. Most of the studies resulted in modifications of model parameters in the Zwietering correlation. Values determined for the model parameters (α, β, γ, δ and θ in Eq. 5-2) in different studies are almost similar to each other, but in Fig. 5-7 high differences between measured and predicted values can be seen. Therefore, it can be concluded that the difference between measured values and predicted ones can be related to variations of the dimensionless number S in Zwietering's correlation, which is a function of impeller size, type, and clearance. The value of S changes linearly for radial impellers with increasing impeller clearance. For axial impellers, however, S is significantly affected by impeller clearance beyond the critical point [2]. As a result, the Zwietering correlation can be modified as follows:

$$N_{js} = \left(a + b\frac{C}{T}\right) v^{\alpha}\left[\frac{g_c(\rho_s - \rho_l)}{\rho_l}\right]^{\beta} d_p^{\gamma} D^{\delta} X^{\theta} \qquad \text{Eq. 5-3}$$

Values for *a* and *b* for different impellers are given in Table 5-4.

Table 5-4 Values for a and b parameters in equation 5-3 for different impellers

Impeller	A	b
RT (0.1 < C/T)	4.7	1.1
PBT-D (0.1<C/T<0.35)	3.47	1.35
CBT (0.1 < C/T)	5.4	0.98

In Fig. 5-7 comparison between predicted values by new model and previous published models [2, 36, 56] are illustrated.

5.2.5.7 Effect of scale

The effect of scale on N_{js} was also evaluated by the gamma ray densitometry technique. For this purpose experiments were repeated in a larger vessel (T=0.4 m) at different solid concentrations for PBT-D and RT. Results are reported in Fig. 5-8. Different scale-up methods have been proposed for N_{js}. These scale-up methods are divided into two categories. The first category includes two common approaches used by engineers to scale-up agitated vessels, i.e., constant specific power (P/V) and constant tip speed. The second category includes the scale-up procedure developed based on empirical studies or theoretical concepts. As it was shown in Fig. 5-8, applying different scale-up rules leads to significant differences in predicting N_{js} for larger scale. The reader should note that even a small difference in the exponent of D can have large effect on power consumption when N_{js} is scaled-up. At high solid loading, for RT, constant P/V seems promising method but for PBT-D, at same solid loading, tip speed constant may provide better prediction. At low solid loading the criterion proposed by Nienow [34] is more accurate. Clearly, there is no scale-up procedure with global validity and appropriate operating condition at large scale, for any mixing system, should be determined independently.

5.2.5.8 Application of the gamma ray densitometry technique in a three-phase system

Solid suspension is also a key factor in three-phase (GLS) mechanically agitated vessels. In a three-phase system the presence of gas makes the solid suspension a more complex phenomenon. It is common knowledge [3] that the presence of gas decreases the ability of the impeller for solid suspension due to 1) decreasing the power dissipation in the system and 2) affecting the flow pattern of the liquid phase resulting in reducing the liquid-solid slip velocity. Reduction of slip velocity decreases the inter-phase forces (drag and lift), which are responsible for solid pickup from the vessel base. As illustrated in Fig. 5-9 by increasing the gas flow rate, a higher impeller speed is required to achieve just off-bottom suspension. The differences between literature data and densitometry technique are considerable.

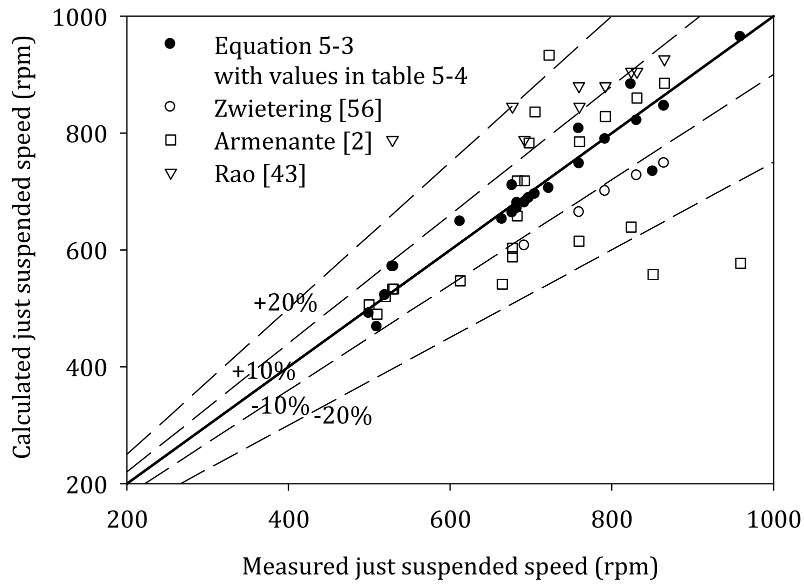

Figure 5-7. Comparing measured values for just suspended speed by densitometry technique and predicted ones with different models [2, 43, 56].

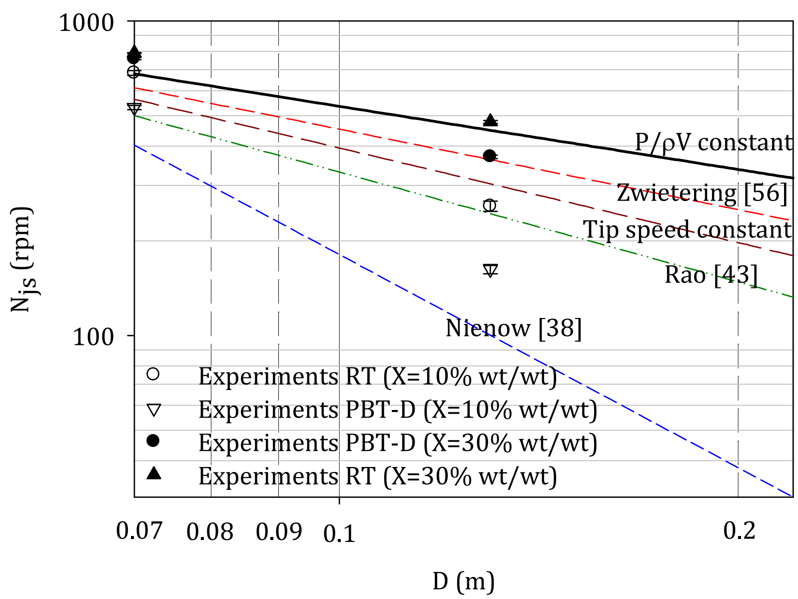

Figure 5-8. Variation of N_{js} by increasing scale and comparison with different scale-up procedures [38, 43, 56].

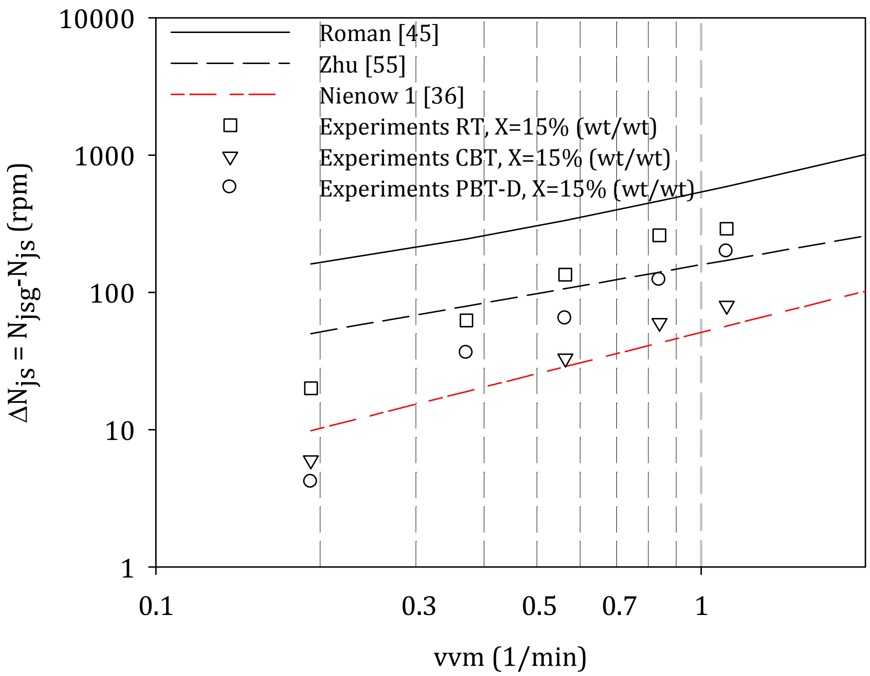

Figure 5-9. Variation of ΔN_{js} in gas-liquid-solid system by increasing gas flow rate, Experimental data compared with correlations [36, 45, 55].

The use of the visual technique in the presence of gas is more difficult. Gas flow (at very low values) could help the suspension of solid particles, but as reported in the illustrated in Fig. 5-9 increasing the gas flow rate directly affects the impeller performance. Accordingly, ΔN_{js} ($=N_{jsg}-N_{js}$) increases by increasing the gas flow rate. Radial and axial flow impellers show different behavior in the GLS system. As can be seen in Fig. 5-9, performance of CBT is less affected by presence of gas compared to two other impellers.

5.2.5.9 Theoretical prediction of N_{js}

The empirical correlations for predicting N_{js}, which typically take the form of equations 5-3, do not facilitate the understanding of the particle suspension mechanism, and theoretical models may provide more insight about the suspension mechanism. The method considered here is based on a force balance acting on a single particle resting at the bottom of the vessel. If we consider a solid particle resting at the bottom of the vessel in liquid, which is under turbulent agitation, by increasing impeller speed particles may start to move by rolling, sliding or lifting. Once particles have lifted up from bottom of the vessel they can be carried away due to the sedimentation-dispersion mechanism. Different forces may act on a single spherical particle when it moves in turbulent media [7]. Based on what is reported in the literatures for solid motion in a stirred tank (for example [7]), also by considering an analogy with particle minimum pickup velocity in pipes (for example [42, 50]); for the particle resting at the bottom of the vessel and at the moment of dislodgment the force balance on single particle can be written as

$$\frac{1}{2}A_p\rho_l C_D v_{l-js}^2 - gV_p(\rho_S - \rho_l) - \rho_{Sl} g H A_p = 0 \qquad \text{Eq. 5-4}$$

which, on solving for velocity yields:

$$v_{l-js} = \sqrt{\frac{4gdp(\rho_s - \rho_l)}{3\rho_l C_D} + \frac{\rho_{ls} H g}{\rho_l C_D}} \qquad \text{Eq. 5-5}$$

$v_{l\text{-}js}$ is the minimum velocity of fluid required to initiate the just suspended condition of solid particles. Equation 5-4 assumes complete "wet-ability" of solids by the liquid and also assumes no slip between particles and fluid. It also assumes spherical particles. Equation 5-5 describes the minimum liquid velocity required at the bottom of the vessel for pick-up particles at conditions close to just suspended speed and it is sensitive to liquid and solid physical properties (dp, ρ_s, ρ_l, ν) and solid concentration (through slurry density and C_D). It is necessary to find an appropriate approach for relating the impeller speed to this minimum liquid velocity. If such a relation exists, it would be possible to determine N_{js} theoretically for any mixing system by knowing physical properties of the liquid and solid phase and solid loading. In light of the lack of accurate data concerning local liquid velocity at the bottom of the vessel for dense liquid-solid systems [11, 12], local liquid velocity could be determined from circulation time in a single-phase agitated vessel. McManamey [24] proposed that the time required for the liquid to circulate once through the flow path should be equal to the maximum length of the circulation path divided by the average liquid velocity in the circulation path ($t = \frac{liquid\ circulation\ path}{liquid\ velocity}$). The liquid circulation path can calculated from the geometry of the vessel and impeller type. For a PBT-D impeller the liquid circulation path is *2H+T/2* [17] and for an RT impeller it is *3T-2C* [35]. Accordingly, we will have $t_c = \frac{3T-2c}{v_l}$ for RT and $t_c = \frac{3T}{v_l}$ for PBT-D. The circulation time can be expressed as a function of impeller speed, liquid properties, and tank and impeller geometry [13]. In agitated vessels, mixing time can be assumed to be some multiple of the circulation time [37, 41, 46]. In this case, accurate correlations for predicting mixing time in dense liquid-solid mixing systems may help to predict the correct N_{js} values. However, by applying this approach, the calculated N_{js} values were highly different compared to current experimental results. This leads to the conclusion that the minimum liquid velocity at the bottom of the vessel required for off-bottom suspension is much lower than the average circulation velocity.

Van der Molen and Van Maanen [52], based on investigations with a laser-Doppler velocimeter, have found that the average velocity at the wall of the agitated vessel could be calculated as $v_l = C_1 U_{tip}(D/T)^{7/6}$. At just suspended conditions we can rewrite this equation as $v_{l-js} = C_1 U_{tip-js}(D/T)^{7/6}$ *or*

$$v_{l-js} = C_1 N_{js} \pi D (D/T)^{7/6} \qquad \text{Eq. 5-6}$$

For the system used in this study minimum liquid velocity at the bottom of the vessel at just suspended condition (v_{l-js}) was calculated from equation 5-5. Calculated v_{l-js} and experimental values for N_{js} were replaced in equation 5-6 and C_1 defined for RT and PBT-D. For RT, at constant solid loading, C_1 increases very slightly by increasing impeller clearance (average value=0.05) however it shows linearly increases by increasing solid loading (slope: 0.18). For PBT-D, C_1 increases linearly by increasing solid loading (slope: 0.13). It is constant for low impeller clearances i.e. C/T<0.37 (C_1=0.07). Results of the theoretical prediction of N_{js} using the current method were compared with experimental data reported by Narayanan [35] and the model they have proposed in Fig. 5-10. Narayanan et al. [35] have done experiments in systems where clearance is 0.5 and solid loading varies between 5% to 20%. Both models exhibit almost same accuracy. The combination of equations 5-5 and 5-6 provides a very simple and semi-theoretical approach for predicting just suspended impeller speed. The uncertainty of predicted N_{js} could be decreased by modifying equations 5-6 based on local measurement of the liquid velocity at the bottom of the vessel.

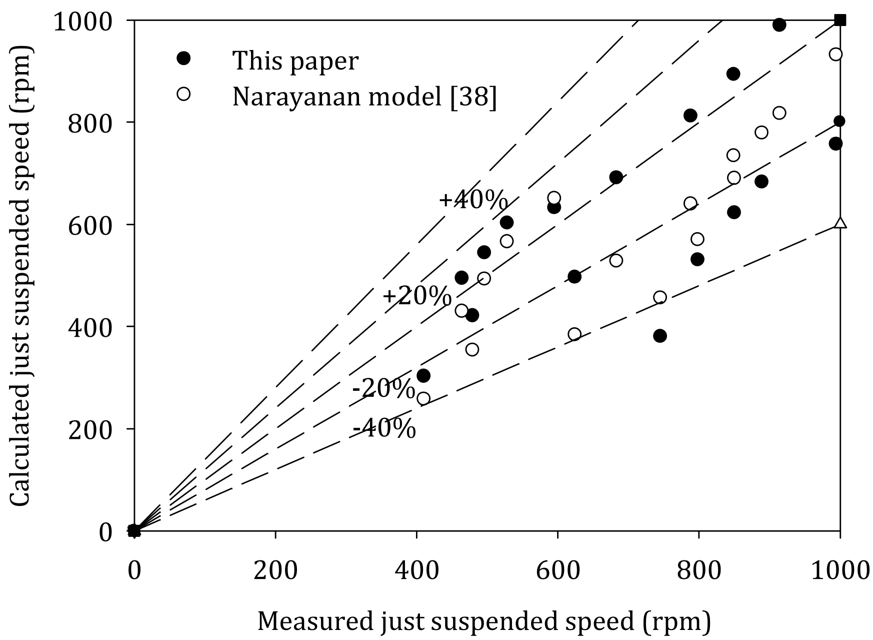

Figure 5-10. Comparing N_{js} calculated from semi-theoretical model proposed in this work with model and experimental data proposed by Narayanan [35].

5.2.6. Summary and conclusions

To overcome limitations of conventional techniques for characterizing just suspended speed in liquid-solid mixing systems, a novel technique was developed based on gamma ray densitometry. This technique represents an original approach and a convenient means of measuring just suspended speed in systems where visual observation is not possible. Even in the systems where other methods are applicable the densitometry technique can provide more accurate measurement.

It was clearly observed that, based on impeller clearance, axial and radial impellers operate differently. All impellers are efficient at low clearance. However, there exists a critical clearance where the flow pattern of the axial flow impeller changes. It was also shown that correlations for predicting N_{js} do not have universal validity. Correlation for predicting N_{js} was modified based on Gamma ray densitometry results. Finally, a theoretical approach was proposed based on the analogy between solid suspension in agitated vessels and the incipient movement of solid particles in pipes. This model shows good agreement with experimental data collected from literature. However, model accuracy could be improved by local solid-liquid characterization close to the bottom of the vessel.

Acknowledgments

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Nomenclatures

a	*Constant of equation 5-3 (-)*
A	*Surface area (m^2), Attenuation of vessel and environment (-)*
A_p	*Particle surface area (m^2)*
b	*Constant of equation 5-3 (-)*
C	*Impeller clearance (m)*
C_D	*Drag coefficient (-)*
D	*Impeller diameter (m)*

dp	*Average particle diameter (m)*
g	*Gravity acceleration (m/s^2)*
H	*liquid height (m)*
I	*Count rate (count/sec)*
P	*Pressure (Pa)*
ΔP	*pressure difference in pressure gauge technique, $P_{N>0} - P_{N=0}$, (Pa)*
L	*Scanning length (m)*
N	*Impeller speed (1/sec)*
S	*Zwietering correlation constant (-)*
t_c	*Circulation time (sec)*
$v_{l\text{-}js}$	*Liquid velocity at the bottom of the vessel at just suspended condition (m/s)*
Vp	*Particle volume (m^3)*
vvm	*Volume of gas per unit volume of liquid per minute (1/min)*
X	*Solid loading Ms/Mt×100 (% wt/wt-)*

Greek letters

α, β, γ, δ, θ	Constants of equation 5-3
ΔN_{js}	N_{jsg}-N_{js} (rpm)
ρ	density (kg/m^3)
μ	Mass attenuation coefficient (kg/m^2)

ε	Solid hold-up or volume fraction (-)
ν	Liquid dynamic viscosity

Subscripts

i	Representation of scanning section
imp	Impeller
js	Just suspended, liquid-solid system
jsg	Just suspended, gas-liquid-solid
l	Liquid
s	solid
ls	Slurry
0	Initial condition (N=0 rpm)

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CHAPTER 6: Experimental Investigation on Solid Dispersion, Power Consumption and Scale-up rules in Moderate to Dense Solid-Liquid Suspensions *

6.1 Presentation of the article

The objective of this third article is to investigate solid concentration distribution in the stirred tank reactor. The solid concentration is measured by means of a fiber optic at different solid loading and impeller clearance for different types of impeller, and three rotational speeds. Radial and axial solid concentration distributions are presented. The mechanism of solid suspension is investigated for axial and radial flow impeller by measuring radial solid concentration close to the bottom of the vessel. For axial flow impellers it is shown that the active volume of the vessel is less than the total volume of the reactor. The solid concentration drop in the vessels is a strong function of impeller speed, clearance and solid loading. The variation of power number for different impellers at different solid loading and clearance also is investigated. Procedures for scale-up of Liquid-Solid (LS) stirred reactor are finally evaluated to identify how scale-up procedure may affect the performance of the reactor.

* Submitted to Chemical Engineering Research and Design (2010)

6.2 Experimental Investigation on Solid Dispersion, Power Consumption and Scale-up rules in Moderate to Dense Solid-Liquid Suspensions

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6.2.1 Abstract

Detailed particle concentration distribution in dense solid-liquid suspension was measured by means of fiber optic probes. The effect of solid loading, impeller speed, and impeller type and clearance was investigated. Results were compared with modeling approaches to show the accuracy of sedimentation-dispersion model and its capability to describe complex phenomena taking place in dense liquid-solid mixing systems. Variation of power numbers by changing impeller clearance and solid loading were also investigated. It was shown that the impeller power number for a slurry system exhibited different trends in a moderate or dense liquid-solid system. In addition, scale-up rules to achieve the same level of homogeneity on a large scale as the laboratory scale were evaluated.

Keywords:

Solid dispersion, fiber optic, liquid-solid, high solid loading, mechanically agitated vessel

6.2.2 Introduction

Mechanical mixing is a common unit operation in chemical, biochemical, mineral processing and many other applications. Suspension of solid particles in a liquid is required in many processes, such as leaching, catalytic reactions, crystallization, and water treatment. According to the process,

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it is possible to carry out the mixing of liquid-solid system in the state of just suspended or homogeneous suspension. Homogeneous suspension, when solid phase uniformly is distributed in the stirred vessel, is difficult to attain and usually is not required in most industrial applications. Proper design of liquid-solid stirred tank reactor requires comprehensive knowledge of local solids concentration profiles in the slurry. Most published studies on liquid-solid agitated vessels have been done for characterizing just suspended condition. Other parameters related to a liquid-solid mixing system, like cloud height, solid concentration profile, power consumption and scale-up, have not been studied extensively in high concentrated systems.

Numerous methods are available for measuring local solids concentration in slurry (for example: (Angst and Kraume 2006, Ayazi Shamlou and Koutsakos 1989, MacTaggart et al. 1993b, Spidla et al. 2005)). One of the popular methods is the optical method. It has been used widely for characterizing solid distribution in agitated vessels (Ayazi Shamlou and Koutsakos 1989, Magelli et al. 1990, Magelli et al. 1991). This non-intrusive method is generally limited to solids concentrations less than 1–2%. This is due to the scattering and blocking of light by the solids between the source and the receiver. Other measurement methods are the sample withdrawal method and the conductivity probes. The sample withdrawal method is the simplest one and has been employed widely (Barresi and Baldi 1987, MacTaggart et al. 1993b). The samples are taken from different locations in the vessel, and the solid phase concentration is determined. However, it is has been shown that Iso-kinetic sampling from stirred tank reactors is extremely difficult because of the complex dynamic behavior of the system (Barresi et al. 1994, MacTaggart et al. 1993b, Nasr-El-Din et al. 1996). Another method is conductivity measurement, which is based on the conductivity changes in the suspension according to the quantity of solid particles present. Most researchers have used the two-electrode conductivity probe (for example (Spidla et al. 2005)). However, four-electrode conductivity probe also have been applied in some studies (Considine and Considine 1985, MacTaggart et al. 1993a, Nasr-El-Din et al. , Nasr-El-Din et al. 1996). The conductivity method is low cost and accurate in dense systems. But, there is an intrusive effect of the probe in the vessel. The influence of the probe on the suspension process can be eliminated by suitably adjusting the size proportion of the probe versus vessel diameter. Recently Mann et al. (Mann et al. 2001) applied electrical resistance tomography to visualize fluid mixing and solid concentration profile in stirred reactors. Optical fibers also have been used widely for characterizing solid concentration in multiphase systems (Boyer et al. 2002, Chaouki et al. 1997, Esmaeili et al. 2008). Details about fiber optic development and application presented by Liu et al. (Liu et al. 2003).

Buurman et al. (Buurman et al. 1986) studied a highly concentrated system at relatively homogeneous conditions. They reported no differences in the solid concentration at three sample withdrawal points situated in an axial direction. The significance of the radial concentration gradient has never been analyzed in detail, even though the presence of the radial concentration gradient depends on the stirrer type and speed as well as on impeller off-bottom clearance, particle diameter and solid loading. Literature data suggest that the radial concentration gradients are usually negligible (Barresi and Baldi 1987, Mak and Ruszkowski 1990, Montante et al. 2002, Yamazaki et al. 1986). However, this assumption cannot be generalized. The presence of radial concentration gradients has been reported by Micheletti et al. (Micheletti et al. 2003). By measuring solid concentration at different radial positions they indicated that a solid concentration gradient exists and it depends on particle size, solid loading and impeller type. It is negligible only for small particle sizes, but increases significantly when particles of a larger size or density are suspended. Angst and Kraume (Angst and Kraume 2004,2006) determined axial and radial particle distributions using an endoscope system. Spidla et al. (Spidla et al. 2005) measured solid concentration by using a conductivity probe in a large scale vessel. They have also proved the presence of radial solid concentration in an agitated vessel.

Several authors have modeled solid distribution in stirred tank for low concentrations of solid and several fluid dynamic models have been adopted for describing solid distribution throughout the tank. They include one dimensional sedimentation dispersion model (Barresi and Baldi 1987, Magelli et al. 1990, Rasteiro et al. 1994, Sessiecq et al. 1999, Shamlou and Koutsakos 1987), multi-zone sedimentation dispersion model (Yamazaki et al. 1986), two- or three-dimensional network of zones (Brucato et al. 1991, McKee et al. 1995). In addition to these phenomenological models, computational fluid dynamics also have been used with different methods and techniques (for example, (Guha et al. 2008, Khopkar et al. 2006, Micale et al. 2000, Montante et al. 2001). Both CFD and phenomenological approaches are useful however they are opposite in terms of complexity. CFD potential is still to be developed and carefully checked against experimental data especially in liquid-solid systems with high concentration of solid.

The principal aim of this paper is to investigate the effect of solid loading, impeller clearance, types of impeller, and rotational speeds on solid concentration distribution in the stirred tank reactor. The variation of power number for different impellers at different solid loading and clearance also is investigated. Procedures for scale-up of liquid-solid stirred reactor are evaluated as well to identify how scale-up procedure may affect the performance of the reactor.

6.2.3. Materials and Methods

Experiments were conducted in a cylinder vessel with an open top and flat bottom. The vessel, impeller dimension, and geometrical details of the mixing system are given in Table 6-1. Water was used as the liquid phase and sand (ρs=2650 kg/m^3, d_p=277μm) as the solid phase. The operating slurry height was set at a value equal to the vessel diameter. Just suspended speed was evaluated by the densitometry technique (Jafari et al. 2010) whenever necessary. The power consumption in the vessel by agitation was measured using a torque meter (Himmelstein Model 24-02 T).

A fiber optic, consisting of two ports (one emitter and one receiver) was used for characterizing solid concentration in the slurry. When solid particles pass in front of the probe they reflect light. Reflected lights go through the fiber (receiver) and are converted to voltage by PV-4A particle velocity analyzer. Light intensity of emitter and voltage range of analyzer were adjusted to make sure system has appropriate sensitivity. Measurements were performed with sampling time of 0.5 msec for 160 seconds for 4 axial positions and 4 radial points. Recorded data was then converted to concentration (using calibration curve) and compiled using homemade codes. It is worth to mention that the fiber optic method is very simple but collecting data from all position in the vessel for this study is quite cumbersome (3 days for each experimental condition).

Table 6-1 Design details of the mechanically agitated vessel

Parameter	Value
Vessel diameter (m)	0.2
H/T	1
Baffle with	T/10
Number of baffles	4
Material of construction	Plexiglass
Geometry	Cylindrical with flat bottom
Impeller clearance from the bottom	Varies between 0.5T to 0.2T
Impellers	PBT (4 blade), D:T/3
	CBT (6 blade), D:T/3
	RT (6 blade), D:T/3

6.2.4. Results and Discussions

Different solid concentrations 4 – 30 vol/vol% (10~50 wt/wt%) were investigated, for different rotational speeds and clearance. For each combination, at the beginning, just off-bottom suspension condition (N_{js}) was verified using the gamma ray densitometry technique (Jafari et al. 2010).

Measurements (with fiber optic) were carried out at different axial and radial positions. To prevent accumulated solid particles around the baffles interfering experimental data, probe positioned at midway of the baffles. All measurements were taken at steady state when the solid concentration profile was fully developed. Identical results were obtained by changing the sampling frequency or data recording time.

Table 6-2 Experimental conditions for this study

Parameter	**Value**
Impeller type	RT, CBT, PBT-D
Rotational speed (rpm)	600, 800, 950
Solid loading (% wt/wt)	10, 20, 30, 40, 50
Clearance	0.2, 0.25, 0.33, 0.37, 0.45

An example of recorded data with a fiber optic probe is shown in Fig. 6-1. Lowest voltage (constant, showing no fluctuation), is related to the condition that there is no solid in front of the probe (ε_s=0) while maximum voltage corresponds to a bed of solid fully settled (ε_s=$\varepsilon_{s,0}$). The data recorded corresponding to fluctuations of solid concentration is also shown in Fig. 6-1. A high increase of voltage was observed when the bulk of solid was reached in the measuring volume of the probe.

In Fig. 6-1, turbulent flow and macro-instabilities create fluctuations in particle concentration that translate into signal fluctuations. It was observed that the solid particle fluctuation (signal fluctuation) is lower in the dense suspension regions compared to lean suspension regions. We believe that it originates from damping turbulent fluctuation in dense regions in the presence of high amounts of solid.

6.2.4.1 Solid radial and axial concentration profile

Typical radial solid concentration profiles at different impeller speeds are presented in Fig. 6-2 which shows the variation of normalized solid volume fraction at different radial location for different axial position. Data presented in this figure includes only measurements at C/T=0.33 and two impeller speeds (600 & 800 rpm).

As illustrated in this figure, the radial concentration profile is non uniform in the vessel and it depends on impeller speed. However radial concentration gradient is not significant. At low impeller speed at the bottom of the vessel, solid concentration is higher for RT underneath the impeller compared to PBT-D. Solid concentration decreases by increasing impeller speed.

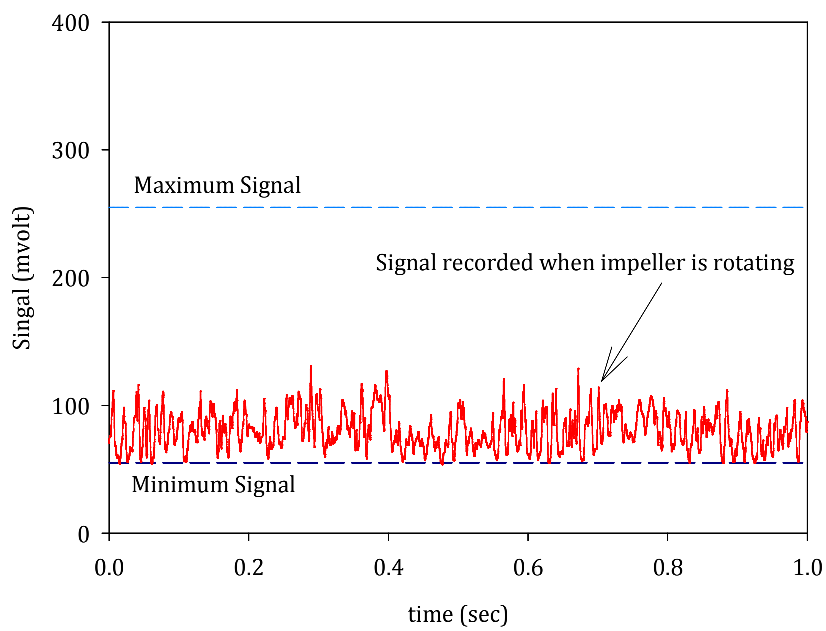
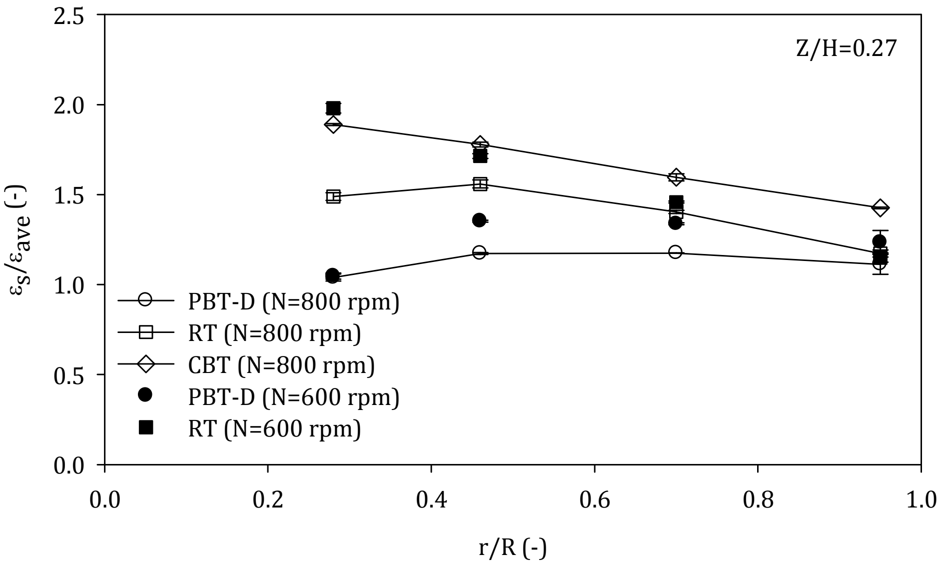
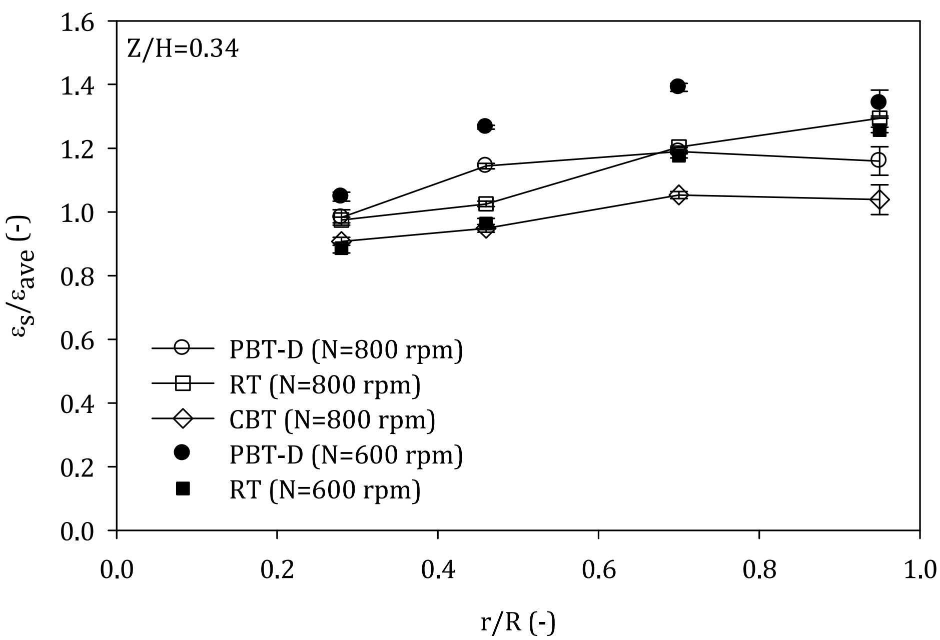


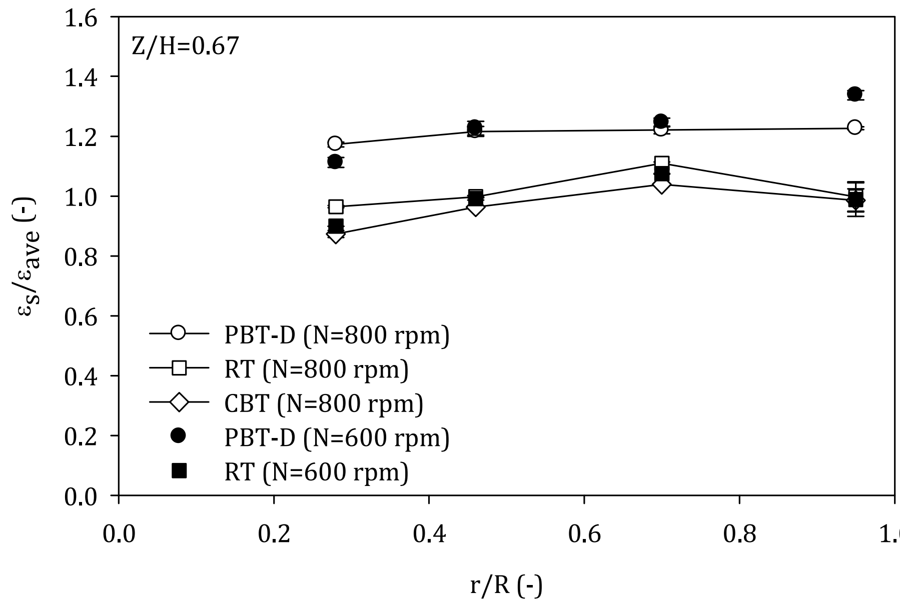
Fig. 6-1. Minimum, maximum and fluctuating recorded signals for liquid-solid mixing system, PBT-D, C/T=0.4, X=20 wt/wt%.


Z/H=0.27
2.5
2.0
1.5
1.0
0.5
0.0
$\varepsilon_S/\varepsilon_{ave}$ (-)
PBT-D (N=800 rpm)
RT (N=800 rpm)
CBT (N=800 rpm)
PBT-D (N=600 rpm)
RT (N=600 rpm)
0.0
0.2
0.4
0.6
0.8
1.0
r/R (-)

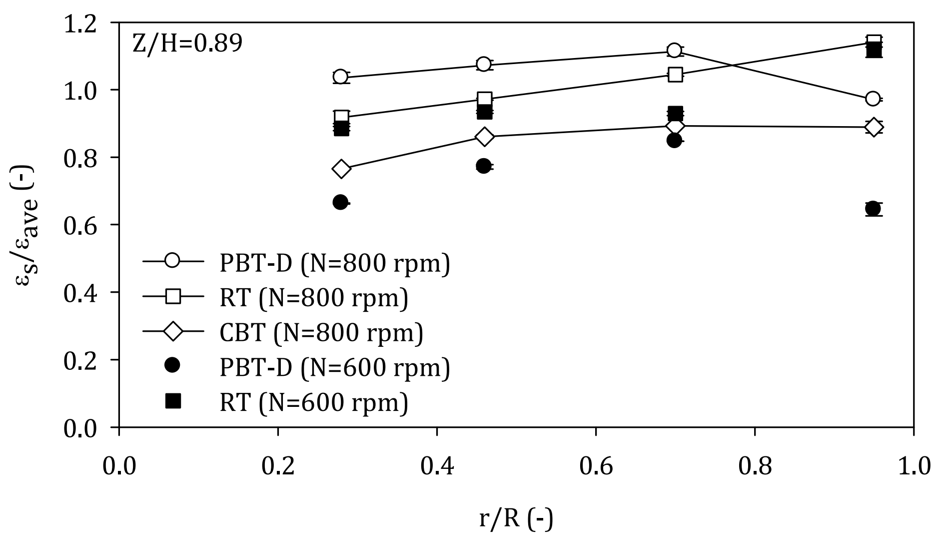
(a)


Z/H=0.34
1.6
1.4
1.2
1.0
0.8
0.6
0.4
0.2
0.0
$\varepsilon_S/\varepsilon_{ave}$ (-)
PBT-D (N=800 rpm)
RT (N=800 rpm)
CBT (N=800 rpm)
PBT-D (N=600 rpm)
RT (N=600 rpm)
0.0
0.2
0.4
0.6
0.8
1.0
r/R (-)

(b)



(c)



(d)

Fig. 6-2. Local particle volume fraction at different radial and axial positions and impeller speeds,

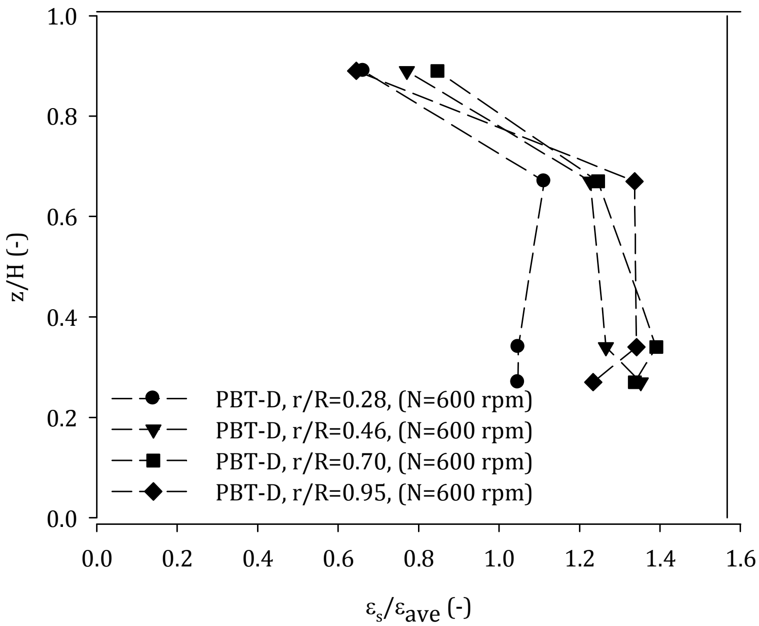
C/T=0.33, X=10 % ($\varepsilon_{ave.}$≈4%).

Radial flow impellers (due to their flow pattern) suspend solid particles from the center of the vessel, which explains why higher solid concentration is observed underneath the impeller. Higher solid concentration at N=600 rpm is related to the generation of fillets at the center for the radial flow impeller. This cannot be seen for PBT-D since it suspends solid at lower impeller speed (N_{js}≈525 rpm). At a higher impeller speed (N=800 rpm) solid concentration for RT decreases and solid concentration gradient close to the bottom of the vessel diminishes since the impeller is operating at a speed higher than N_{js} (N_{js}≈690 rpm). The solid concentration for CBT is higher than for RT and a radial solid gradient was observed for this impeller even at 800 rpm. CBT is a radial flow impeller and it was expected to have a higher solid concentration in the middle compared to other regions. Higher solid concentration compared to that of RT is due to a lower ability of CBT to suspend solid particles.

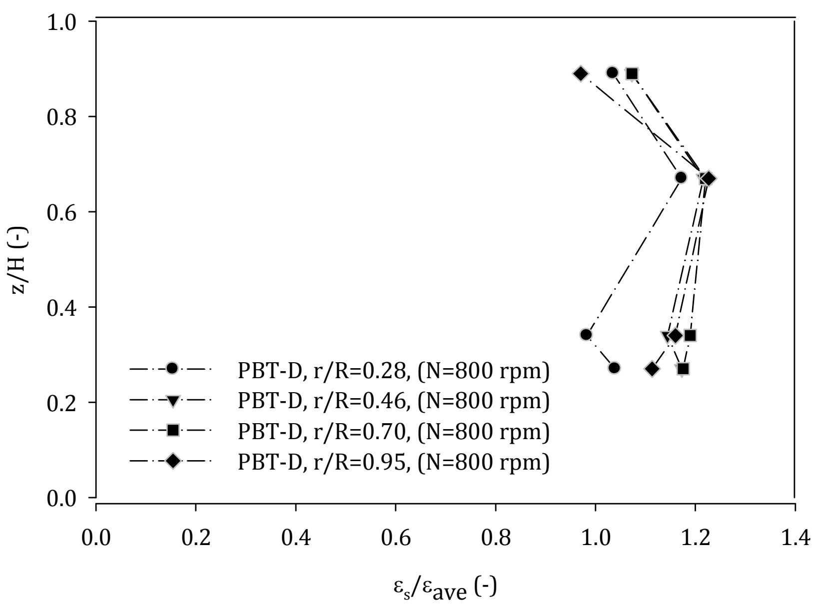
In the upper part of the vessel, a different behavior was observed. At low impeller speed a noticeable solid concentration gradient appears since the power dissipated by the impeller is not enough to disperse the solid particles efficiently. In the upper half of the vessel (Z/H >0.5) solid concentration gradient is also negligible. It is interesting to note that the solid concentration for PBT-D compared to radial flow impellers is higher. This could be related to the large pumping capacity of the axial flow impeller compared to radial impellers, which could lift solid particles to the upper level of the vessel. The amount of solid concentration at the upper level of the vessel is lower than that close to the bottom, which clearly shows the existence of the solid lean region, which decreases the effective volume of the vessel.

Solid distribution in agitated vessels is a function of different parameters, namely impeller type, impeller clearance, impeller speed, solid loading and physical properties. By increasing the impeller speed, the solid concentration profile along the vessel will change. At impeller speeds lower than the just suspended there is a layer of unsuspended solid particles on the bottom of the vessel. A smaller quantity of solid particles is suspended and dispersed in the vessel. At an impeller speed close to just-suspended conditions solid concentration along the vessel becomes more homogenous. However, it strongly depends on the impeller clearance and the solid loading. As illustrated in Fig. 6-2, even for moderate solid concentration (X=10 % wt/wt), the solid concentration suddenly drops at the top of the vessel. This indicates the presence of a solid lean region at the top of the vessel, which can also be observed by with visual inspection. This emphasizes that even if the just off-bottom suspension is usually considered as the desired operating condition it is important to identify the solid concentration profile beyond N_{js} to evaluate the active volume in the liquid-solid system.

The above discussion is well illustrated in Fig. 6-3, where the solid axial profile has been plotted for different radial positions. Solid concentration is high at the bottom while it decreases dramatically along the vessel height. Impellers, based on their power dissipation in the vessel and flow pattern, have the ability to disperse solid particles to a certain level of the vessel. This level is known as the cloud height (Bittorf and Kresta 2003, Bujalski et al. 1999, Hicks et al. 1997, Mak 1992). This is an important parameter in liquid-solid mixing systems and identifies the active volume of the vessel. As illustrated in Fig. 6-3, a drop in solid concentration is more noticeable at lower impeller speeds. The interface between the rich and lean solid region appears where the downward velocity of the particles is balanced by the upward velocity of the fluid at the wall. Bittorf and Kresta (Bittorf and Kresta 2003) explained three phenomena, which are useful for investigating the mechanism behind the formation of this interface: hindered settling stratified flow and negative buoyant jet. They concluded that the location of the clear fluid interface in a stirred tank could be determined if an upward jet in the tank were driving the solid concentration. They have also shown that the flow at the wall of the tank can be characterized by three-dimensional wall jets (Bittorf 2000, Bittorf and Kresta 2000, Kresta et al. 2001).



(a)



(b)

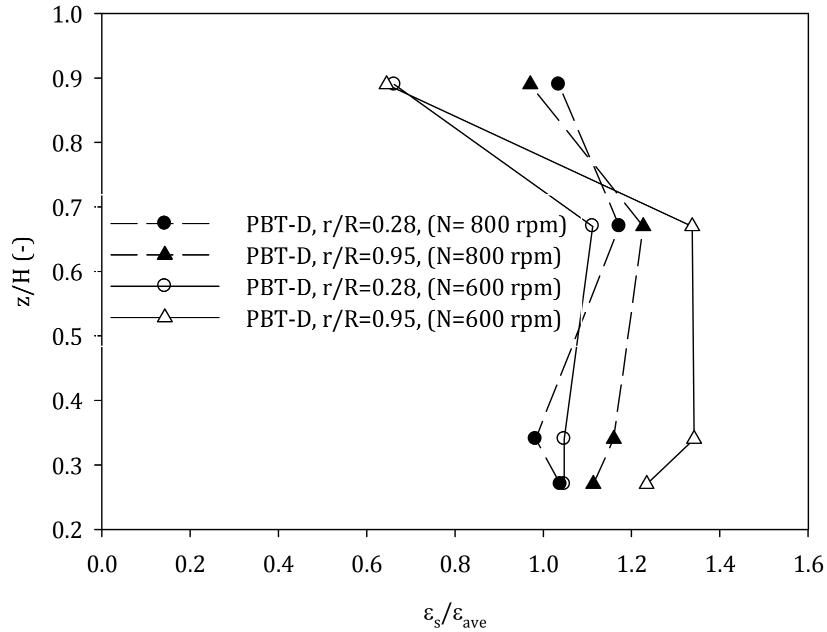
Fig. 6-3. Axial solid distribution at different radial positions, X=10% (N_{js}=525 rpm), C/T=0.33, a) N=600 (rpm), b) N=800 (rpm), They demonstrated that for axial flow impellers maximum jet velocity on the wall decays with $(z/T)^{-1.15}$. They have used this information to develop a model for predicting cloud height for axial flow impellers. This information will be used further to explain the effect of impeller type and clearance on solid concentration distribution.

6.2.4.2 Effect of impeller type

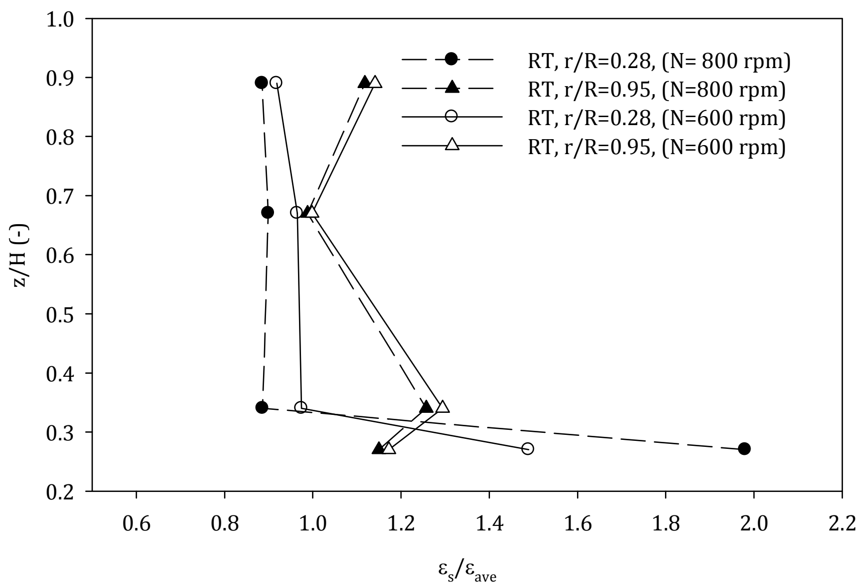
The impeller type has a significant effect on the solid suspension as well as the solid concentration profile. Three types of impellers are used in this work. They belong to two different classes of impellers. PBT-D is a classic example of axial flow impellers, while RT and CBT are classified as radial flow impellers. At standard application conditions (C/T=0.33) the axial flow impeller generates a single loop flow while radial flow impellers generate a double-eight flow pattern. The axial flow impeller pushes solid particles toward the periphery of the vessel where they become suspended and dispersed, while the radial flow impeller pulls them underneath it from where they can be suspended and dispersed. Accordingly, we expect to observe different solid concentration profiles for these impellers.

As illustrated in Fig. 6-4 the shape of the solid concentration profile is different for RT and PBT-D. Near the center of the vessel solid concentration for RT is higher compared to the ones for PBT-D while at the wall solid concentration is higher for PBT-D compared to RT.

The effect of impeller type is also illustrated in Fig. 6-5, for moderate and dense liquid solid systems. The effect of the impeller type is more pronounced at high solid loading, as shown by the different profile shapes obtained for radial and axial flow impellers (notify all impellers were at the same speed). Although N_{js} for PBT-D is lower than RT and CBT, the solid concentration at the bottom for this impeller is higher compared to the other types of impellers. A noticeable difference can be observed at the top of the vessel.



(a)



(b)

Fig. 6-4. Radial solid distribution at different axial positions, comparing solid concentration profile for axial and radial flow impeller,

X=10 (%wt/wt), C/T=0.33, a) PBT-D (N_{js}=525 rpm), b) RT (N_{js}=690 rpm).

Solid concentration for PBT-D dramatically decreases at high solid loading while this dramatic change cannot be observed for radial flow impellers. It indicates that although PBT-D can suspend particles at lower power consumption, it is not able to disperse them up to top of the vessel. Cloud height for the axial flow impeller will be much less than for radial flow impellers. Location of the low concentration solid region in the stirred tank could be determined by employing the information about the upward jet in the tank since solid distribution in the vessel can be driven by the liquid jet in the tank (Bittorf 2000, Bittorf and Kresta 2000, Bittorf and Kresta 2003, Kresta et al. 2001). Since axial and radial flow impellers initialize the jet from different locations different solid concentration profiles can be observed in experimental data reported in this work (for example, Fig. 6-5).

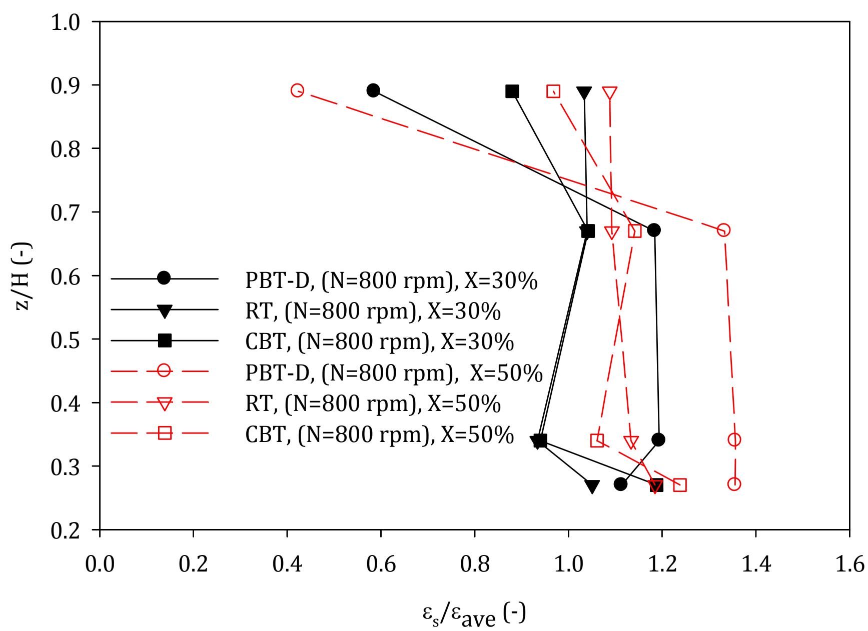


Fig. 6-5. Axial solid concentration profile for different impellers at different solid loadings C/T=0.33, X=30% wt/wt (14% vol) and X=50% wt/wt (27.4 % vol)

Axial flow impellers generate only an upward jet. The wall jet is initialized from the bottom of the vessel (z/H<0.2) and its maximum velocity decays along the vessel wall (approximately starts at z/H=0.37). As illustrated in Fig. 6-5 solid concentration drops significantly in the upper volume of the vessel (z/H > 0.65). This is the point where fluid velocity is not high enough to overcome the settling velocity of solid suspension (Bittorf and Kresta 2003).

Cloud height can be predicted by the equation presented by Bittorf and Kresta (equation 14 in (Bittorf and Kresta 2003).

Table 6-3 Predicted cloud height for PBT-D at N=800 rpm

Solid concentration (X)	Impeller clearance (C/T)	Predicted cloud height by (equation 14 in (Bittorf and Kresta 2003)
30%	0.25	0.76
	0.33	0.63
	0.4	0.45
50%	0.25	0.56
	0.33	0.68

Comparing predicted values with the experimental results of this work cannot be done quantitatively since solid concentration is only measured in four axial positions and cloud height cannot be determined from experimental data accurately. However, values in Table 6-3 are in good agreement with data plotted in Fig. 6-5 and 6-6.

6.2.4.3 Effect of solid loading

Fig. 6-5 also illustrates the effect of solid loading on the solid concentration profile. At the same impeller speed (for the same impeller) increasing the solid loading from moderate to high significantly affects the ability of the impeller to provide homogeneity in the system. The most significant effect was observed for PBT-D. This could be related to the influence of solid particles on the liquid velocity profile. Solid loading has less influence on radial flow impellers. It is worth mentioning that although impellers are rotating at the same speed, the amount of energy dissipation in the system is different. The energy consumption of RT is higher than CBT, which is higher than that of PBT-D. The high amount of energy dissipated in the system by RT could explain why the solid concentration profile slightly changed by increasing solid loading.

6.2.4.4 Effect of impeller clearance

The impeller clearance is another key parameter that could significantly affect the solid concentration profile. Fig. 6-6 shows axial solid concentration for all impellers at three different impeller clearances. By increasing clearance the impeller capability to suspend solid particles will decrease. This is due to the fact that at the same impeller speed the energy dissipated in the slurry will be reduced and the flow pattern will change if the impeller clearance increases (Kresta et al. 2001, Kresta and Wood 1993, Montante et al. 1999). At low clearance, suspension of solid particles can be achieved at lower impeller speeds, but solid particles will not be homogenously dispersed throughout the vessel. As shown in Fig. 6-6 at low impeller speeds cloud height increases by increasing impeller clearance. The effective volume of the vessel is about 50% of the total volume when the impeller is placed close to the bottom of the vessel (C/T=0.2), but it significantly changes by increasing the clearance and the whole volume of the vessel could be considered as effective volume (C/T=0.4). However, average measured solid holdup is lower than what was originally used, which is related to the fact that all solid particles are not suspended and a portion of the solid particles are at rest at the bottom of the vessel. At higher impeller speeds ($N>>N_{js}$) the impeller clearance does not have a significant effect on the solid distribution in the vessel. Although operating at a high impeller clearance (C/T=0.4) results in higher solid concentration at the bottom of the vessel, which is related to diminishing the capability of the impeller for suspending and dispersing solid particles, the interface between the clear liquid and the suspension layer became sharper and more clearly defined as the solid concentration was increased.

Bittorf and Kresta (table 1 in (Bittorf and Kresta 2003)) have also reported a variation of initial velocity at the origin of the jet (U_{core}) as a function of impeller clearance. By increasing impeller clearance U_{core} decreases, which can describe why the cloud height decreases and lower solid concentration is observed at the top of the vessel. In Fig. 6-6 (for PBT-D), the difference between solid concentrations at the top of the vessel for different clearances is not significant. Measurements were made at z/H=0.89, which is much higher than cloud height (see table 6-3). At this axial position average liquid velocity is low and almost similar for different impeller clearances, which leads to the same solid concentration.

6.2.4.5 Relative standard deviation (RSD)

Relative standard deviation is very often used to quantify the distribution quality of solid in liquid-solid suspension (Atieme-Obeng et al. 2004, Montante et al. 2001). It is a deviation of the local solid concentration from the mean solid concentration. The magnitude of RSD (calculated by equation 6-1) decreases as the distribution becomes more homogeneous and perfect homogeneity will give a zero value. The degree of homogeneity increases as agitation is increased. Impeller clearance and rotational speed have a significant effect on the homogeneity of the system. Although at high clearance a higher impeller speed is required to achieve full suspension, a portion of suspended solid is dispersed more homogenously compared to low clearance conditions. An interesting observation can be made in the RSD variation with impeller clearance. At the impeller clearance equal to 0.4, RSD is lower than C/T=0.25. This is not acceptable since we know that at high impeller clearance the unsuspended mass of solid is higher and a noticeable portion of solid particles rest at the bottom of the vessel. The reason is at lower impeller clearance and low impeller speed there is a solid lean concentration layer that exists at the top of the vessel, which increases RSD significantly. This layer disappears at higher impeller speeds or if the impeller is positioned with a higher clearance. It is worth mentioning that when solid loading is increased the system requires more power to achieve the same degree of homogeneity.

$$RSD = \frac{1}{C_{ave}}\left(\frac{1}{n-1}\sum_{i=1}^{n}(C_i - C_{ave})^2\right)^{0.5} \qquad \text{Eq. 6-1}$$

6.2.4.6 Numerical prediction methods

Besides experimental efforts for characterizing solid dispersion in liquid-solid mixing systems, researchers introduced different approaches for predicting solid concentration distribution in such systems. Those efforts can be classified to CFD simulations and phenomenological models. Despite the fact that currently commercial CFD codes provide capabilities for studying liquid-solid mixing systems, due to high uncertainty associated with numerical procedures and the physical description of complex phenomena in concentrated systems, successful results in predicting solid concentration in high concentrated systems are not yet reported.

Since in this article we are dealing with high concentration systems, only phenomenological models have been considered and evaluated.

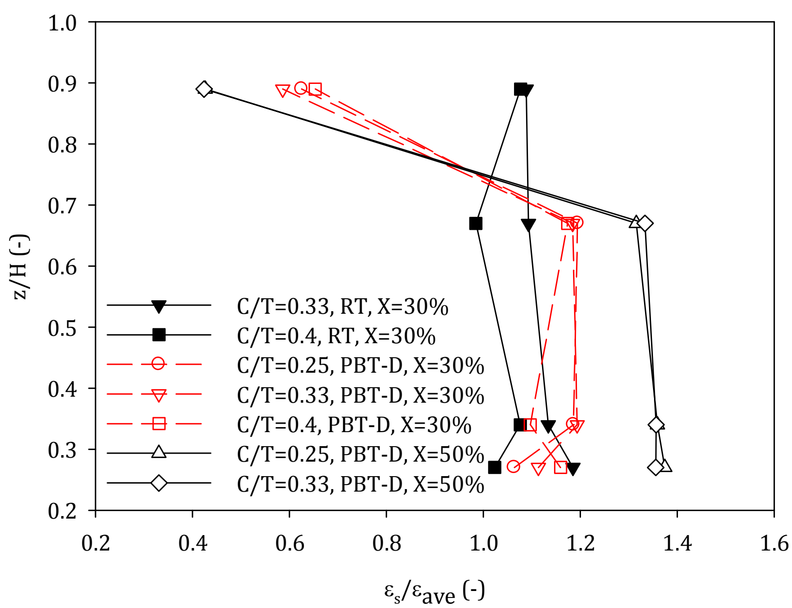


Fig. 6-6. Axial solid concentration profile for different impellers at different clearances, N=800 (rpm)

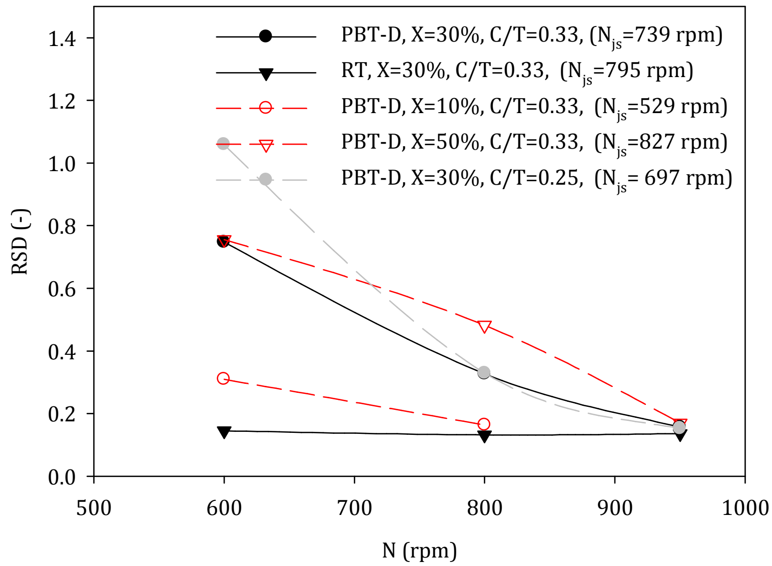
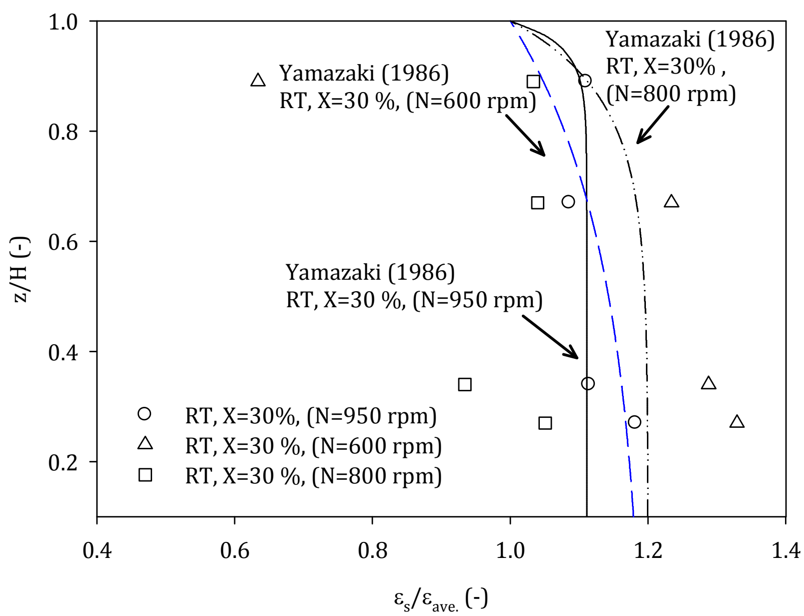
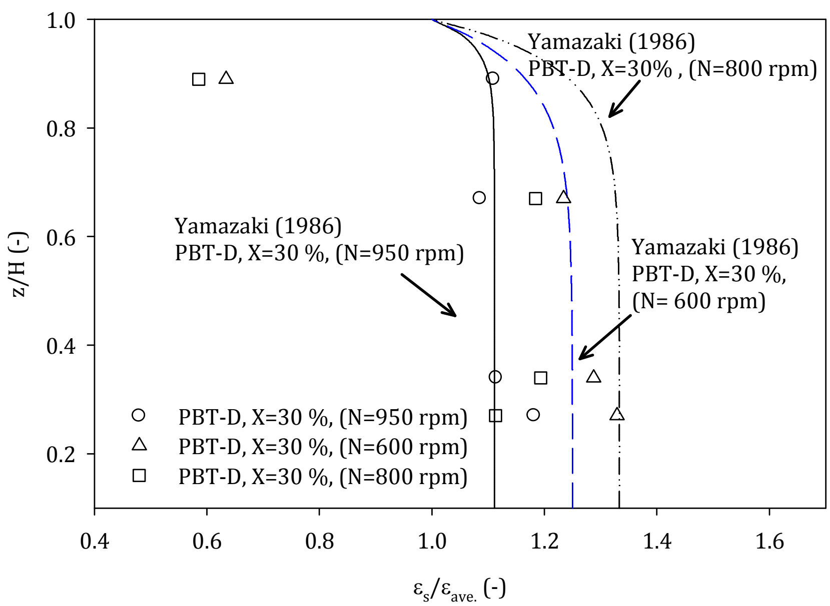


Fig. 6-7. Variation of RSD in a liquid-solid system by changing impeller speed and clearance



(a)



(b)

Fig. 6-8. Comparing experimental data with model proposed by Yamazaki et al. (1986), a)RT, b)PBT-D

In modeling the dispersion phenomena, most researchers based their analysis on one dimensional sedimentation dispersion model. To model the distribution process, both solid and liquid phases are taken as an upward moving continuum and the particle dispersion coefficient is employed to account for the relative movement between two phases. The dispersion coefficient is a function of operating conditions, geometry and physical properties (for example (Yamazaki et al. 1986)).

In this study we used the modified sedimentation dispersion model, which was introduced by Yamazaki et al. (Yamazaki et al. 1986). They considered different regions around the impeller based on a flow pattern generated by the impeller (double eight of single eight). A material balance was written for the differential volume of each region and solved analytically, which finally leads to a series of equations for predicting solid concentration (Table 7 from (Yamazaki et al. 1986)).

Comparing modeling results with experimental data shows that sedimentation dispersion model can provide a good prediction at high impeller speed. At this speed ($N \geq 950$ rpm) the system is almost homogenous. At low impeller speed the hydrodynamics of the system is different and model assumptions are not able to capture the phenomena occuring in the system. The accurate prediction of the solid distribution at impeller speed beyond N_{js} may be achieved by modifying the model concept based on reliable information about the fluid dynamics of the system.

The model is not able to predict cloud height precisely. As mentioned before more comprehensive models for predicting cloud height were proposed by Bittorf and Kresta (Bittorf and Kresta 2003). Current models for predicting cloud height for a high solid concentration system can be improved by having the local measurement of the liquid and solid fluid dynamic in the liquid-solid system.

6.2.4.7 Power consumption in a liquid-solid system

There are some discrepancies between data published in literature about the variation of power numbers in slurry systems and the influence of solid loading. Some of those results have been listed in Table 6-4.

Table 6-4 Reported results for power number variation in slurry systems

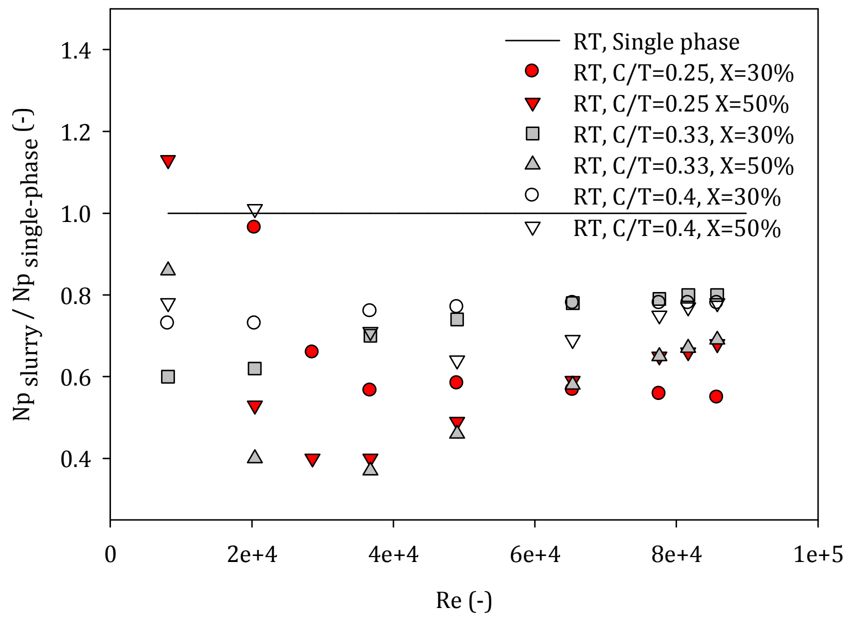
Reference	**Experimental conditions**	**Major observations**
Bujalski (Bujalski et al. 1999)	Impeller type: A-310 Solid concentration: up to 40 % wt/wt	• By increasing solid loading the power number increases. • At solid loading higher than 20% wt/wt the variation of the power number showed different trends; at low impeller speed (N<200 rpm) the power number is lower than that for a single phase. By increasing impeller speed the power number increases until it reaches a maximum value and then slightly decreases by increasing the impeller speed constant. • At solid loading higher than 20% wt/wt the presence of a solid layer at the bottom of the vessel redirected the flow and had the overall effect of reducing the power number at low speed. By increasing impeller speed this layer starts to vanish and the power number

		increases.
Wu et al (Wu et al. 2002)	Impeller : PBT-D and RT Solid concentration: >50 % vol/vol	• The power number of PBT increases at high solid concentration while that of RT decreases. • The increasing power number for PBT can be described in the same way as Bujalski et al. (Bujalski et al. 1999). • For PBT-D increasing solid loading only increases skin friction and, therefore, drag coefficient. • The reduction in the power number of RT was related to the fact that damping in liquid velocity at high solid loading suppresses the dead flow zones at the back of the Rushton turbine blades leading to a reduction of drag.
Angst and Kraume (Angst and Kraume 2006)	Impeller: PBT-D Solid loading: up to 20% wt/wt	• Reduction of the power number for the pitched blade turbine • At a lower impeller speed the power number decreases by increasing impeller speed. This was probably caused by a reduced bottom clearance of the impeller due to settled solid particles resting at the bottom of the vessel. • When particles start to become suspended, the increased density in the vicinity of the impeller leads to an increased power number.

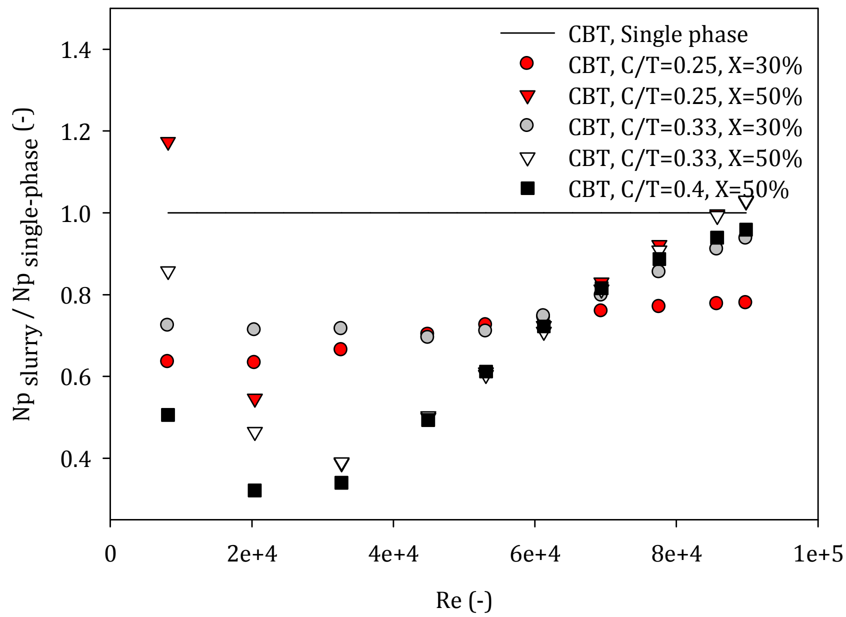
Fig. 6-9 (a-d) shows selected results of the normalized power number vs. Reynolds number for different solid loadings, impeller types and clearances. The results demonstrate that the power number for slurries is lower than that of the single-phase system at high Reynolds numbers. Power measurements have been done for two different solid concentrations, i.e., 30% wt/wt and 50% wt/wt and different impeller clearances. Measurements were made in a small scale reactor (T=0.21 m), but for RT and PBT-D they were also repeated on a large scale for 30% wt/wt solid loading.

In a small vessel (for all impeller clearances), the power number (Np) for the liquid-solid system is lower than that of the single-phase. For the high concentration system (50% wt/wt) the variation of the power number is different. It is high at a lower impeller speed. Then it decreases by increasing impeller speed until it reaches a minimum and then starts to increase again. CBT shows the same trend as RT.

At low impeller speed the power number increases by increasing impeller speed. This reduction can be attributed to the formation of solid fillets at the center and periphery of the wall. These fillets may reduce promote the streamlined flow. The power number reaches the minimum value and starts to increase, which could be due to breaking fillets and the simultaneous commencement of solid suspension.

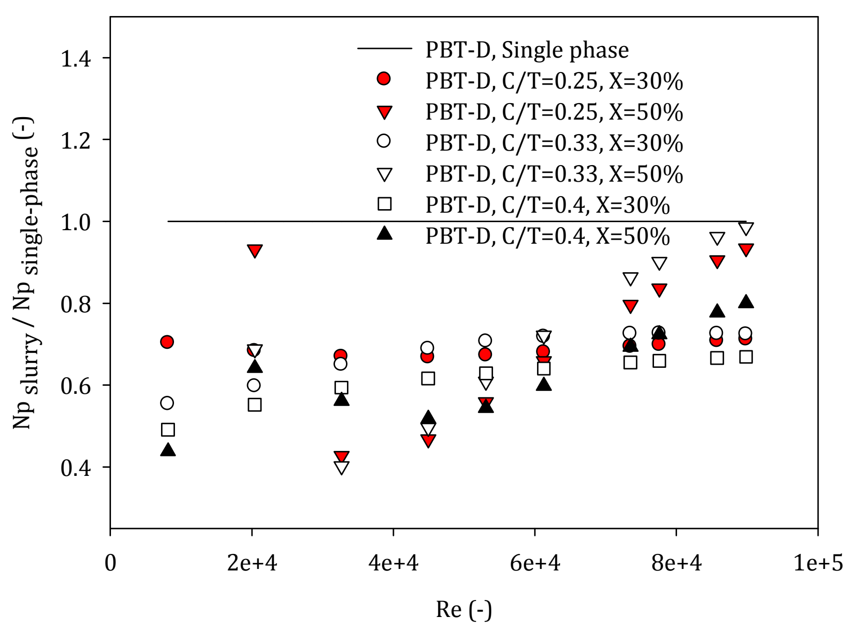


(a)



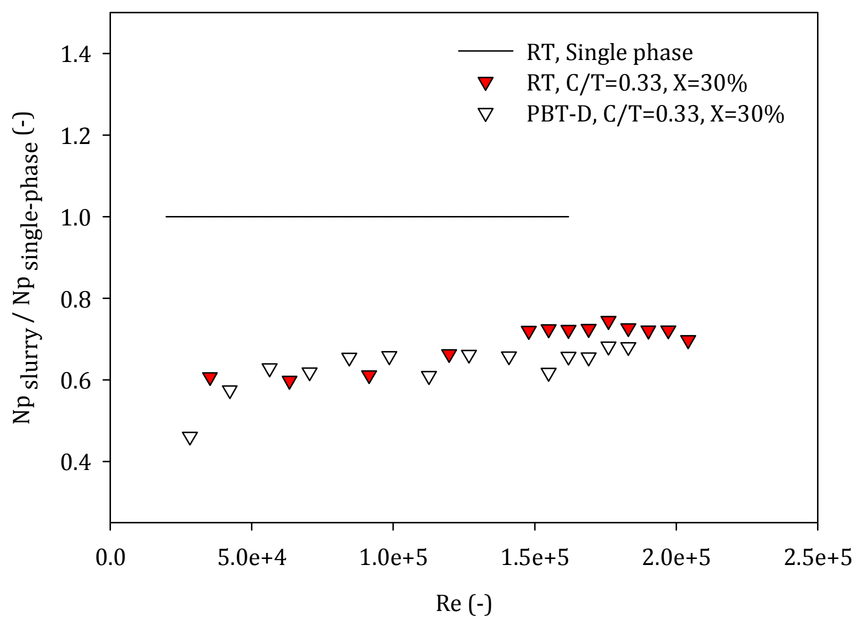
CBT, Single phase
CBT, C/T=0.25, X=30%
CBT, C/T=0.25, X=50%
CBT, C/T=0.33, X=30%
CBT, C/T=0.33, X=50%
CBT, C/T=0.4, X=50%
Np slurry / Np single-phase (-)
Re (-)
1.4
1.2
1.0
0.8
0.6
0.4
0
2e+4
4e+4
6e+4
8e+4
1e+5

(b)



PBT-D, Single phase
PBT-D, C/T=0.25, X=30%
PBT-D, C/T=0.25, X=50%
PBT-D, C/T=0.33, X=30%
PBT-D, C/T=0.33, X=50%
PBT-D, C/T=0.4, X=30%
PBT-D, C/T=0.4, X=50%
Np slurry / Np single-phase (-)
Re (-)
1.4
1.2
1.0
0.8
0.6
0.4
0
2e+4
4e+4
6e+4
8e+4
1e+5

(C)



(d)

Fig. 6-9. Power number ratio, a) RT, b) CBT, c) PBT-D, d) large vessel

While solid particles become suspended, more solid particles enter the suspension and energy dissipation at the liquid-solid interface increases. The density of the slurry also increases and, consequently, the Np_{slurry} increases. A further increase in impeller speed reduces the solid concentration in the impeller area and the power number increases slightly or reaches a constant value.

As reported by Angst and Kraume (Angst and Kraume 2006) at lower impeller speed Np_{slurry} decreases by increasing impeller speed. This was probably caused by a reduced bottom clearance of the impeller due to settled solid particles resting at the bottom of the vessel. With increasing impeller speed the particle layer thickness is reduced and, therefore, the power number decreases by decreasing the bottom clearance of the stirrer. This cannot, however, explain the high values of the power number at very low impeller speeds. This effect is more pronounced when the impeller is placed at a lower clearance and solid concentration is higher. When particles start to become suspended, the increased density in the vicinity of the impeller leads to an increased power number.

The explanation of Angst and Kraume (Angst and Kraume 2006) could be applied to describe what has been observed here. Known phenomena related to the effect of solid particles on power consumption can be listed as the following. Nienow (Nienow 1992) mentioned that the power number of the slurry system could be reduced due to centrifugal effects by decreasing the solid fraction between the blades of the stirrer to a degree equal to the one of a single-phase system. On the other hand, the influence of the solid particles on the turbulence of the continuous phase could have an impact on power consumption. Turbulence of the continuous phase is reduced due to the presence of solid particles (Graham 2000). Since the increase in shear stress in a turbulent fluid is comparable to a fluid with higher viscosity (Lee and Börner 1987), a reduction in turbulence would lead to smaller shear stress and to reduced power consumption.

In addition, increasing the solid content in the agitated vessel may reduce the velocity of the secondary loop and circulation number (Kohnen and Bohnet 2001). Angst and Kraume (Angst and Kraume 2006) explained the reduction of the power number for the axial flow impeller using the third phenomena. The same explanation could be applied here. By analogy to centrifugal pumps, if the flow rate of the centrifugal pump is reduced (by a reduction of the inlet or outlet cross-section) a drop in power consumption will occur, because a small amount of fluid is accelerated.

6.2.4.8 Evaluation of the scale-up procedure

Scale up is a major challenge for liquid-solid mixing systems. It is a significant step in equipment design from conditions established in laboratory to industrial size. The required agitation speed in a large vessel to achieve the same level of suspension as in a smaller one can be estimated by different theoretical and empirical approaches. Common scale-up rules are presented as follows:

$$ND^n = constant \qquad \text{Eq. 6-2}$$

The most common criterion for scale-up is to maintain the power per unit volume (specific power) constant between the different scales (n=0.67). Different values have been reported for n, which are listed in Table 6-5. Differences in scale-up procedures do not appear to be large, but they could have a major effect on energy consumption, selectivity, and yield in large scale operations.

In this section we intend to compare different scale-up procedures and identify which one can satisfy process the requirements. Four different scale-up procedures have been evaluated, namely constant power per unit volume, constant tip speed, the Montante et al. procedure (Montante et al. 2007) and the Buurman method (Buurman et al. 1986). The solid concentration in the large vessel (T=0.4 m) has been measured by means of the fiber optic probe described previously. Both setups are geometrically similar and measurements were made only at C/T=0.33. The positions of the measurement points also have been chosen so as to conserve geometrical similarity.

As illustrated in Fig. 6-10 different scale-up approaches result in different solid concentration profiles. None of the scale-up approaches evaluated here can provide the same solid concentration profile as in the small vessel. However, the deviation from the solid concentration profile in small vessel is less, if constant power per unit volume or the Buurman method is applied.

In addition, RSD in both scales clearly shows differences between the scale-up procedures. The largest RSD difference was observed when the tip speed is kept similar. In fact, the use of a constant tip speed as scale-up criterion for solid dispersion underestimates the power requirement at large scale. The Buurman method (Buurman et al. 1986) slightly underestimates the power requirements and provides the most similar RSD to that of the small vessel. It can be concluded that the procedure proposed by Buurman et al. (Buurman et al. 1985,1986) could satisfy the requirement for operating on a large scale and achieving the same homogeneity as on a laboratory scale.

Table 6-5, Different scale-up rules for solid dispersion in agitated vessels

Reference	Scale-up rule (value of n)	Remarks
(Bourne and Hungerbuehler 1980)	1	Tip speed constant can satisfy required homogeneity in continuous crystallizer
(Buurman et al. 1985)	0.78	Described solid distribution quality in terms of the height of the homogenous zone
(Rieger et al. 1988)	0.67 (normal operation) 1 (highly homogenous)	Described solid dispersion by turbulence theory
(Magelli et al. 1990, Magelli et al. 1991)	0.93	Empirical study on multi-impeller systems
(Montante et al. 2003)	0.93	Empirical study on multi-impeller systems
(Mak 1992, Mak and Ruszkowski 1990)	0.67	Empirical study on single impeller system
(Angst and Kraume 2006)	0.52 – 0.86	Empirical study on single impeller system
(Ochieng and Lewis 2006)	1	CFD simulations in diluted suspension
(Montante et al. 2007)	0.96	Used Turbulent intermittency concept

Table 6-6, RSD in a small and large vessel for different scale-up procedures, impeller: RT, X=30% and C/T=0.33

Small Vessel	Large vessel			
	Tip Speed Constant	Montante (2007)	Buurmann (1986)	Constant Power Per Unit Volume
0.14	0.23	0.21	0.155	0.16

However, deviation in the solid concentration profile could have an effect on product selectivity and yield on a large scale compared to the one at laboratory scale.

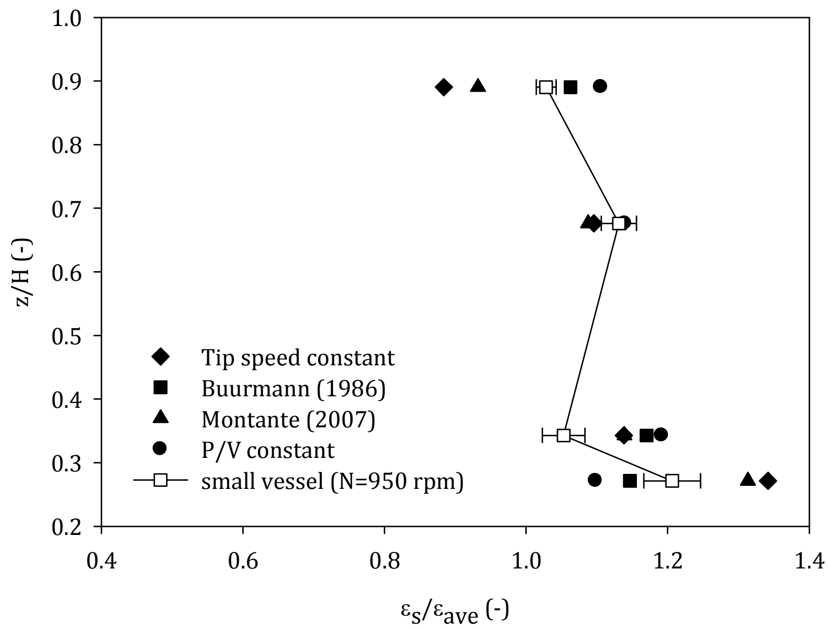


Fig. 6-10. Solid concentration profile in small vessel (N=950 rpm) and large vessel with different scale-up approaches, impeller: RT, X=30% and C/T=0.33

6.2.5. Conclusion

The solid concentration profile was measured in a concentrated liquid-solid suspension using an optical fiber probe. The effect of impeller type and clearance were investigated as well as solid loading. Solid concentration distribution varies for axial and radial flow impellers. At the bottom of the vessel solid concentration is higher close to the wall for axial flow impellers compared to radial flow impellers. Solid loading has an effect on the solid concentration profile. Under extreme conditions a layer of solid lean region is generated at the top of the vessel. Its volume deepens on impeller clearance and speed. It disappears at high impeller speeds. Modeling procedures were evaluated for predicting the solid concentration profile. Although sedimentation dispersion models are useful and accurate for predicting solid concentration in dilute conditions, they fail to make the same prediction under concentrated conditions. In addition, power measurements have been made to determine how the impeller power number may change in a liquid-solid mixing system. The presence of solid reduces the power number and the same trend was observed for radial and axial impellers. This reduction could be related to the changing flow pattern at the bottom of the vessel, which eliminated the baffle effect and the damping of turbulence for impeller speeds higher than N_{js}. However, there are discrepancies between results presented here and some published works. The scale-up procedure to achieve the same quality of suspension on a large scale was evaluated and it was shown that most of the scale-up procedures underestimated the power requirement to provide the same homogeneity on a large scale. The lowest differences between RSD in small and large vessels are for the method proposed by Buurman (Buurman et al. 1985,1986).

Acknowledgement

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Nomenclature

C	*Impeller clearance (m), or solid concentration (kg/m^3)*
D	*Impeller diameter (m)*
d_p	*Mean particle diameter (m)*
H	*Liquid height (m)*
n	*Parameter equation 6-2*
N	*Impeller speed (1/sec)*
N_p	*Power number (-)*
P	*Power consumption (watt)*
r	*Local radial position (m)*

R	*Vessel radius (m)*
Re	*Reynolds Number ($\rho ND^2/\mu$)*
RSD	*Relative standard deviation (-)*
T	*Vessel diameter (m)*
U_{core}	*Core velocity of the jet (m/s)*
V	*Vessel volume (m^3)*
X	*Solid loading Ms/Mt×100 (% wt/wt-)*
z	*Local axial position (m)*

Greek letters

ρ	density (kg/m^3)
ε_s	Solid hold-up (-)
$\varepsilon_{s,0}$	Solid hold-up for fully settled bed (-)

Subscribes

ave	*average*
i	*Sampling point counter*
imp	*Impeller*
js	*Just suspended*
l	*Liquid*
s	*solid*

Abbreviations

A-310	*Hydrofoil impeller, Lightnin A-310*
CBT	*Concave Blade Turbine*
PBT-D (n)	*Pitched blade turbine down pumping flow (number of blades)*
RT (n)	*Rushton turbine (number of blades)*

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CHAPTER 7: Effect of Scale up Procedures on local gas-liquid mass transfer coefficient in stirred tank reactors

7.1. Introduction

In the process industries, many chemical transformations involve multi-phase reactor technology. Successful process design requires the ability to predict how reactants (molecules) come together and how reactor performance (yield and selectivity) differs from the lab scale. Many parameters are central to the design and scale-up of multi-phase reactors, such as inter-phase mass and heat transfer coefficients, flow regime, solid, bubble or droplet size and their distribution. Mass transfer from a gas into liquid (or slurry) phase is a widely spread phenomena in chemical engineering in order to provide reactants to the continuous phase (liquid or slurry). It has crucial importance in systems involving chemical synthesis (oxidation or hydrogenation), ore leaching (gold cyanidation), waste water treatment and aerobic fermentation.

Usually the mass transfer rate is described as proportional to the concentration gradient, where proportionality is given by the volumetric gas transfer coefficient (k_La). This parameter must be known in order to carry out design and scale-up of gas-liquid (GL) and gas-liquid-solid (GLS) contactors. This is a very complex task, especially in systems with chemical reactions, viscous media and in concentrated slurries (at high concentration of solid). In such processes, a mechanically agitated vessel (stirred tank reactor) is a common contactor in which gas is distributed in the liquid, as bubbles, by the proper combination of sparger and agitation system.

Characterization of hydrodynamic parameters (gas hold-up, bubble size, power consumption and gas-liquid mass transfer coefficient) has been studied significantly by researchers (Middleton and Smith 2004). The successful design and scale-up of such reactors require comprehensive knowledge about all those parameters, but, most importantly, they should predict how these quantities will change with the equipment scale. In the multiphase reactor, chemical phenomena (reaction kinetics) are usually independent of the vessel dimensions, while many physical phenomena are significantly affected by dimensional change. Intense mixing is easy to achieve in small scale reactors while in larger scale reactors reactants experienced different flow patterns, mixing conditions and turbulence structures. In most gas-liquid multiphase reactors non-homogenous distribution of dissolved gas is responsible for poorer performance in large scale operation and causes serious drawbacks at the industrial scale.

Garcia-Ochoa and Gomez (Garcia-Ochoa and Gomez 2004) presented a theoretical approach for predicting gas-liquid mass transfer coefficient and hold-up in gas-liquid stirred tank reactors. They have developed a method based on theoretical principles for determination of the volumetric mass transfer coefficient, k_La, in stirred tank reactors with Newtonian and non-Newtonian fluids. Their model is based on Higbie's penetration theory, which establishes a relationship between the mass transfer coefficient, k_L, and the contact time between two different phase elements. This exposure time can be estimated from turbulence isotropic of Kolmogorov's theory as the eddy length to fluctuation velocity ratio. Volumetric mass transfer coefficient values can be predicted with reasonable accuracy.

Martin et al. (Martín et al. 2008) have proposed a model to predict a mean k_La as a combination of two scales of mixing in stirred tank. Two main scales of mixing can be considered inside a stirred tank: macro-mixing and micro-mixing. Macro-mixing is related to the tank size circulation and is responsible for bubble motion, surface aeration and tank homogenization. Meanwhile, micro-mixing is related to the small liquid eddies, responsible for the concentration gradients surrounding the bubbles, and it prevails around the impeller. They have used experimental results and empirical equations to unveil the contribution of both mechanisms to the volumetric mass transfer coefficient, k_La.

Recently some studies have been done to characterize gas-liquid mass transfer distribution in the stirred tanks (Alves et al. 2004, Laakkonen et al. 2006, Laakkonen et al. 2007). However the effect of scale-up procedure on kLa distribution in the vessel has not been investigated.

The experimental approach has been considered in this study to describe how scale-up procedure affects the gas-liquid mass transfer coefficient (k_La) distribution. It is shown how gas hold-up distribution and fluid dynamic parameters change according to the scale-up procedure selected. k_La distribution has been predicted using the multi-block modeling approach.

7.2. Materials and Methods

Details of each experimental setup listed in table 7-1. Water was used as the liquid phase, air as the gas phase and sand (dp=277 μm, ρs=2650 kg/m^3) as the solid phase (in GLS systems). The operating liquid (slurry) height was set at a value equal to the vessel diameter. The shaft was driven with a DC motor (Type 42A5FEPM, 130 V, 1.8 A, 0.25 HP, Bodine Electric Company). Gas was fed to the system using a ring sparger with a diameter of D_s=0.75D (as recommended in (Middleton and Smith 2004)). It was located underneath the impeller.

Air was supplied through eight orifices facing up, each with 1 mm diameter. Air flow rate was measured and controlled with an accuracy of ±0.05 L/min using a mass flow meter (Aalborg, Model GFM371) with a flow range of 0-50 L/min. At the beginning of an experiment, oxygen present in the liquid was eliminated using nitrogen until $C_{O2} \approx 0.5$ mg/L. At this point the gas suddenly was changed to air. The time-dependent oxygen concentration was recorded after switching to air using a high precision dissolved oxygen (DO) probe (YSI model 58) and LabVIEW software. By recording the oxygen concentration versus time, measuring the saturated concentration of oxygen in the liquid phase and fitting experimental data to the mass balance equation for oxygen, the gas-liquid mass transfer coefficient can be determined.

Table 7-1: Design details of a mechanically agitated vessel

Parameter	Value
Small vessel diameter (m)	0.2
Large vessel diameter (m)	0.4
H/T	1
Baffle width	T/10
Number of baffles	4
Material of construction	Plexiglass
Sparger (both setups)	Ring sparger,
	8 hole, evenly distributed Ds=0.75D
Impeller position (both setups)	C=T/3
Geometry (both setups)	Cylindrical with flat bottom
Impellers (both setups)	PBT-D (4 blade), D=T/3
	CBT (6 blade), D=T/3
	RT (6 blade), D=T/3

Due to the strong dependency of oxygen concentration at the equilibrium with temperature, measured k_La was corrected for 20°C. This method has been used for characterizing k_La in both small and large vessels (details can be found in (Sardeing et al. 2004)).

Fiber optics has been used for characterizing gas or solid hold-up and bubble size which, had one light emitter and two light receivers (do=1 mm). When gas bubbles pass in front of the probe they reflect light. Reflected light converted to voltage and amplified by a particle velocity analyzer (PV4A, Institute of Chemical Metallurgy, Chinese Academy of Science) and raw signals were acquired by PV4A software. The signals were processed by home-made codes to calculate the gas hold-up and bubble size. Measurements were carried out for 4 radial positions and 4 axial positions in both vessels.

7.3. Results and Discussions

7.3.1 Gas liquid mass transfer

Mass transfer results are presented in Figure 7-1 (a & b) and 7-2. Results also have been compared with literature data. In order to assess the surface aeration effect on recorded data, primary tests have been done without sparging gas in the system. It was found that the mass transfer due to surface aeration was not greater than 6% of the mass transfer coefficient measured with sparging and, therefore, considered as negligible. As expected, k_La increases by increasing the gas flow rate due to increasing gas hold-up.

Also, it increases by increasing impeller speed since bubble breakage is enhanced. Thus, the surface area available for mass transfer increases. At the same impeller speed RT provides higher k_La, however, this conclusion does not consider additional power dissipated in the system by RT. In Figure 7-1 (a & b), experimental data are also compared with empirical correlations to show the accuracy of measurements.

As it can be seen in Figures 7-2, k_La for gas-liquid–solid is lower than that of gas-liquid. There are three main mechanisms by which solids are claimed to effect k_La:

- A viscosity effect due to turbulence damping by solid particles
- In heterogeneous regime, solid particles supplant small bubbles in dense-phase. This must affect gas-liquid mass transfer.
- An interface effect, for very tiny active particles acting at the interface to enhance k_La

7.3.2 Evaluation of scale-up procedures

Experiments have first been carried out in the small vessel at different impeller speeds under fully turbulent conditions (Re>10,000) and for two gas flow rates (vs-0.001 m/s and vs=0.003 m/s). For the large vessel operating conditions were determined by following different scale-up procedures. For this purpose, two operating conditions must be defined, i.e., gas flow rate and impeller speed.

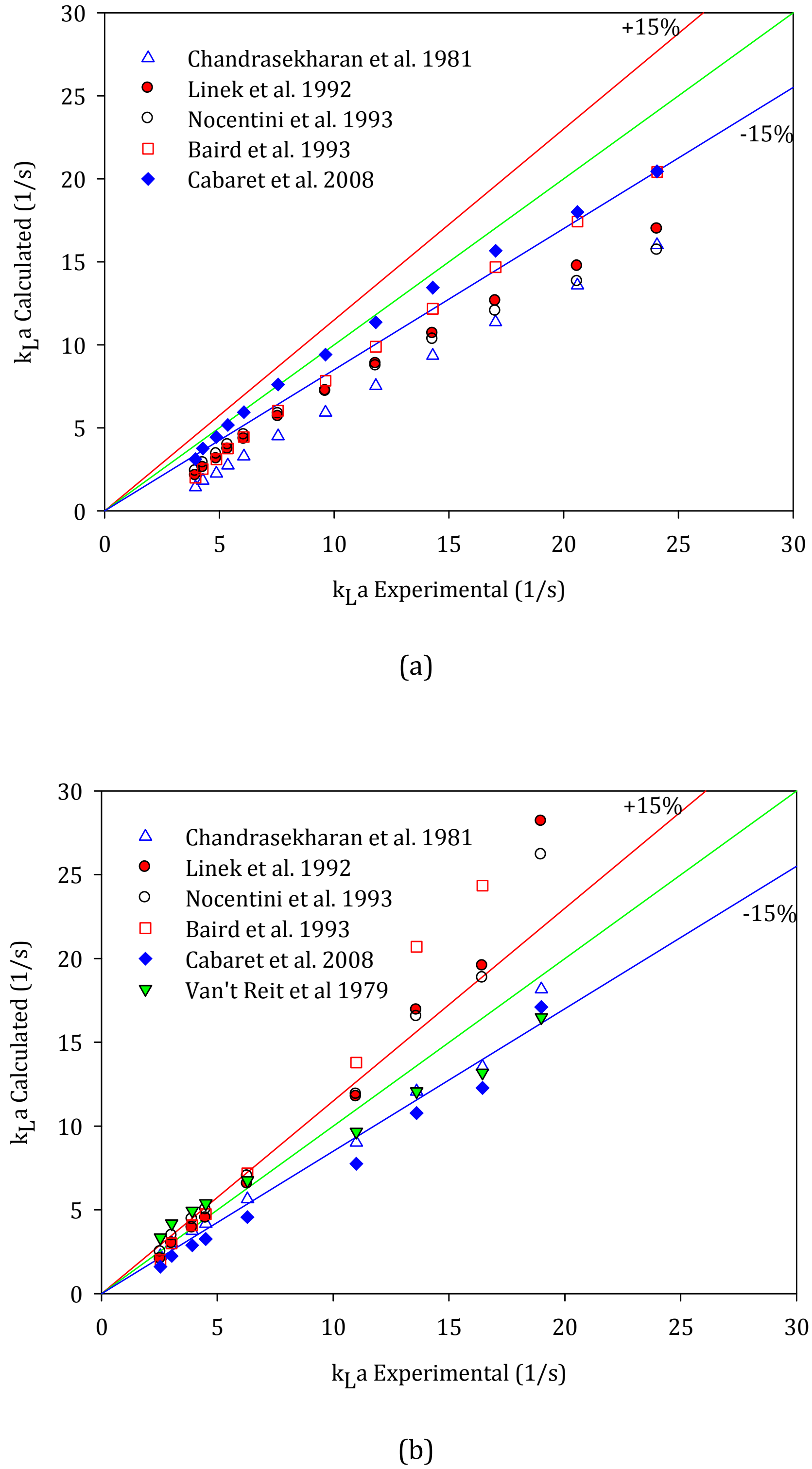


Figure 7-1 Sample k_La results for small (a) and large vessels (b) compared with empirical correlations (Baird et al. 1993, Cabaret et al. 2008, Linek et al. 1992, Nocentini et al. 1993)

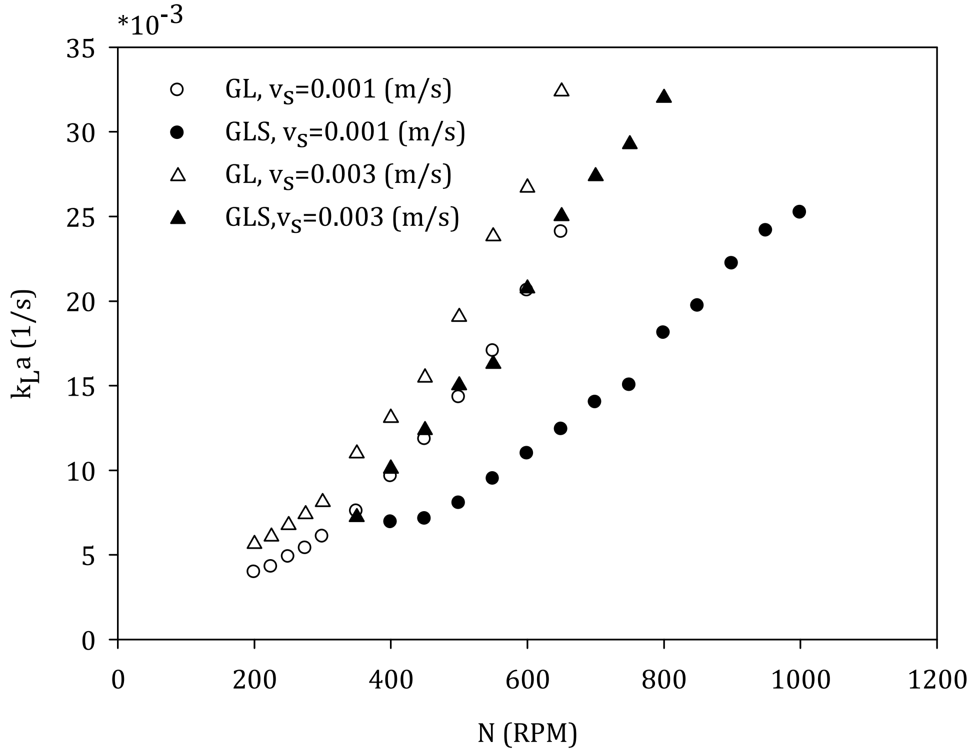


Figure 7-2 k_La variation by increasing impeller speed for RT, in GL and GLS systems (X=50% wt/wt) and for different gas flow rates

For gas-dispersed agitated vessels different scale-up concepts are available (Middleton and Smith 2004). Three standard methods are available for calculating gas flow rate during scale-up, i.e., constant flow number, constant volume of gas per volume of liquid per minute (vvm), and constant gas superficial velocity. For impeller speed six standard methods are available, i.e., equal specific energy dissipation rate, equal impeller tip speed, constant mixing time, constant Reynolds number, constant Froude number and constant gas transfer rate. For the gas flow rate constant, superficial gas velocity has been chosen and for impeller speed all six methods have been examined. Comparison of k_La in small and large vessels is illustrated in Figure 7-3. It represents the variation in k_La when the agitated vessel was scaled-up based on different scale-up methods. All methods show a decrease of k_La by increasing the scale of the vessel. Differences become more significant when the agitated vessel operates at a higher impeller speed, which is related to operation in different flow regimes.

While the small vessel is operating in a vortex cavity regime with recirculation, the large vessel is still in vortex cavity with no recirculation regime. In some cases the difference is more significant, like scaling up based on constant impeller Reynolds number. The decrease in measured k_La also can be explained by the distribution of k_La in the agitated vessel. In the small vessel mixing is very intense so the k_La is almost homogenously distributed, but in large vessel this is not the case. Local gas-liquid mass transfer coefficient is a strong function of local gas hold-up and local fluid dynamic parameters, like turbulence intensity.

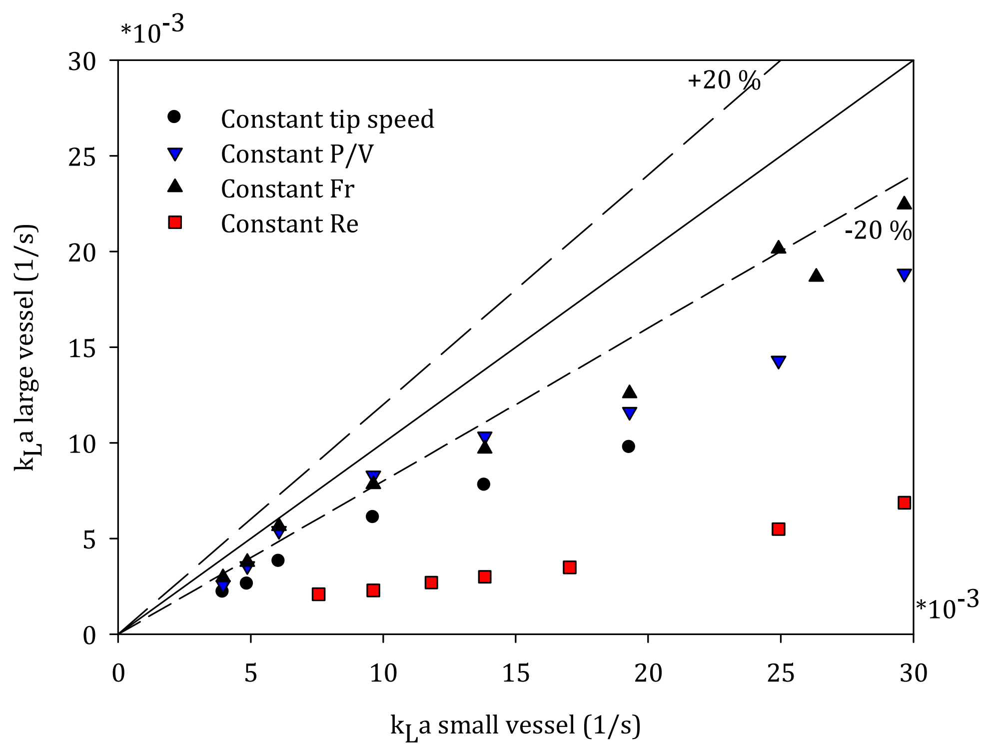


Figure 7-3 Comparing gas-liquid mass transfer in small and large vessels with different scale-up procedures

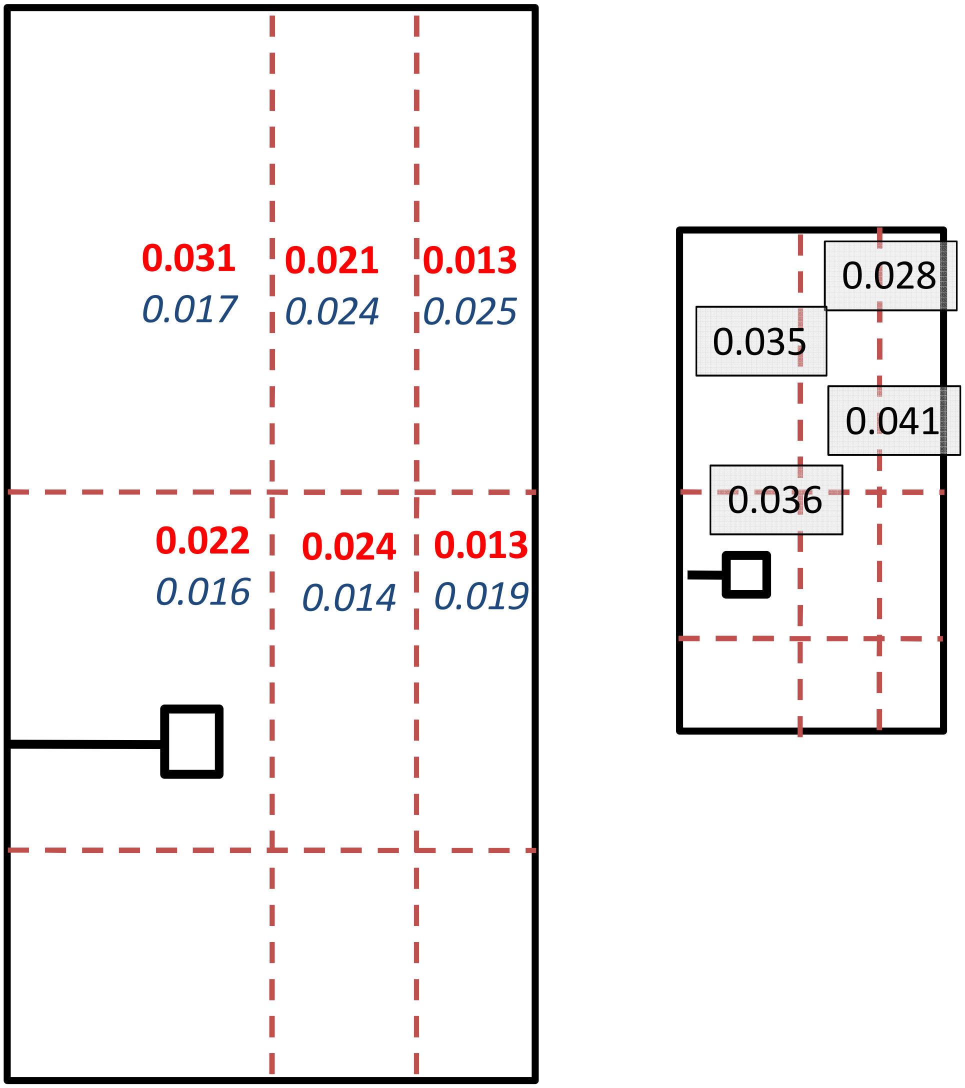


Figure 7-4.a, gas hold-up distribution variation by two different scale-up method, (left: large vessel, right: small vessel). Bold numbers: scale-up based on constant tip speeds, italic numbers: scale-up based on constant power per unit volume,

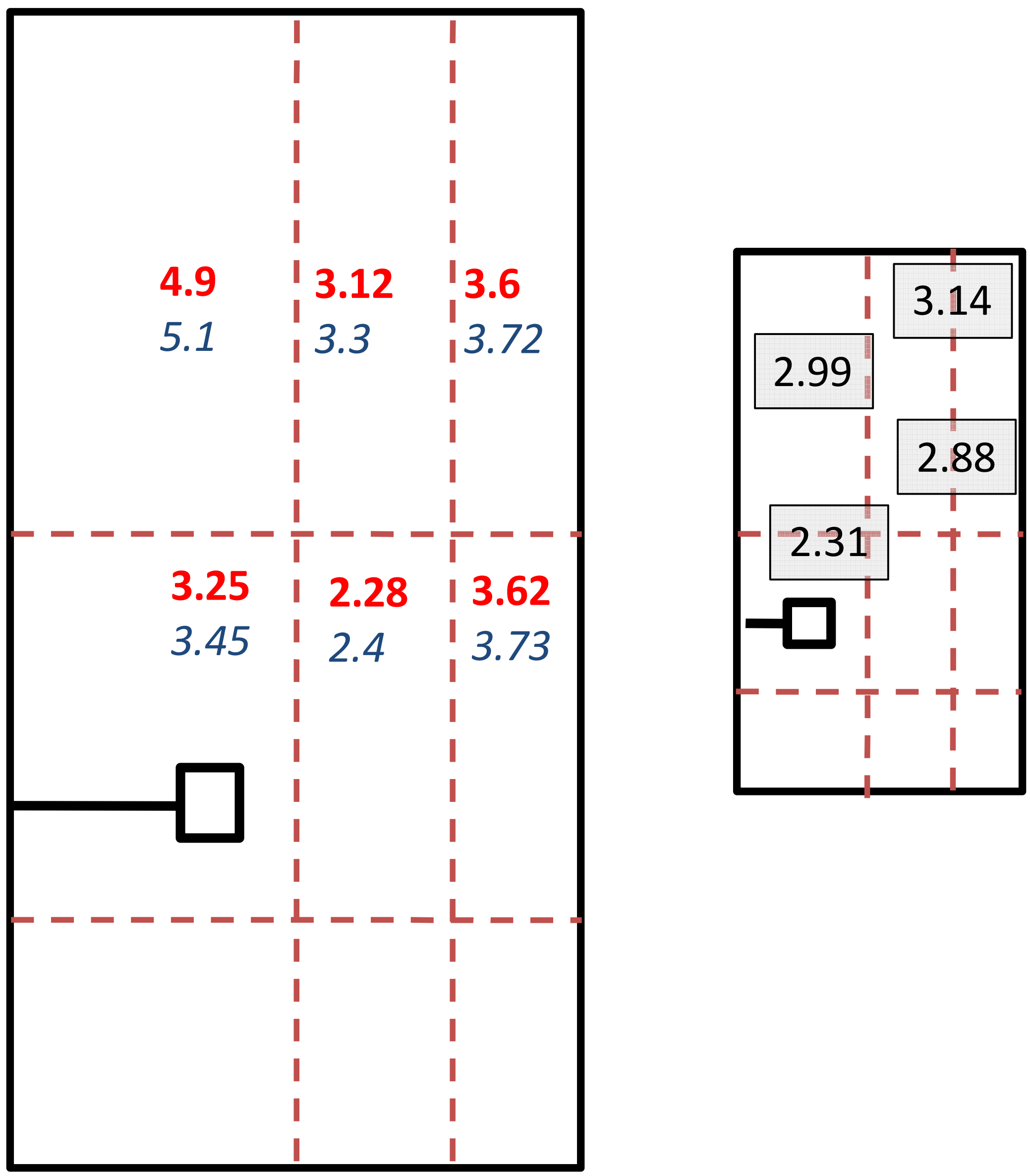
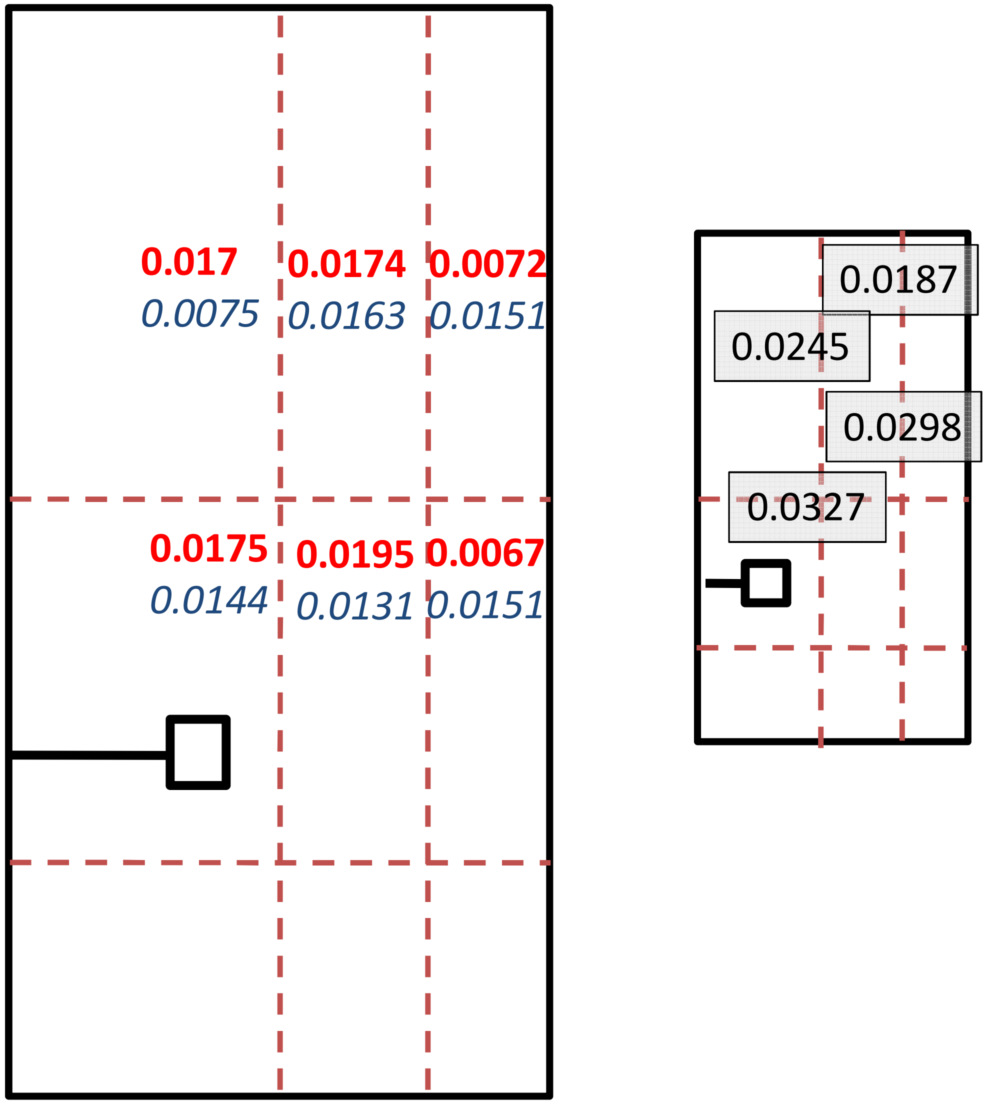


Figure 7-4.b, bubble size distribution variation by two different scale-up method, (left: large vessel, right: small vessel). Bold numbers: scale-up based on constant tip speeds, italic numbers: scale-up based on constant power per unit volume,



(C)

Figure 7-4.c, k_La distribution variation by two different scale-up method, (left: large vessel, right: small vessel). Bold numbers: scale-up based on constant tip speeds, italic numbers: scale-up based on constant power per unit volume

For a better understanding of the effect of scale-up procedures on k_La gas hold-up distribution and fluid dynamics, parameters have been characterized in both small and large vessels. Gas hold-up and bubble size were measure by means of fiber optic probes and local k_La calculated by equation 7-1 and 7-2.

$$\varepsilon_{ave} = \frac{P_g}{\rho_l(\pi/4)D^2H} \qquad \text{Equation 7-1}$$

$$k_L.a = 2\sqrt{\frac{D_{ab}}{\pi}}.(\frac{\varepsilon_{ave}\rho_l}{\mu})^{1/4}.(\frac{6\varphi}{d_b}) \qquad \text{Equation 7-2}$$

The difference in hold-up distribution is more obvious. Although scale-up based on the same specific power shows the same hold-up distribution in both small and large vessels, for other procedure the difference is more significant.

7.3.3. Contribution of scale of mixing

Inside agitated vessels different scales of mixing are present, i.e., macro-mixing or micro-mixing. Also, there are regions where one of the scales of mixing prevails. Many studies have been done to identify those regions with different techniques, i.e., the fast chemical reaction method or material dispersion characterization (for example: (Martín et al. 2008)). Micro-mixing prevails near the impeller where the velocity gradient surrounding bubbles is defined by small eddies. It is widely accepted that the gas flow rate or stirring improves the macro-mixing in the tank, however, this fact does not guarantee improvement in micro-mixing. For proper design and scale-up of gas-sparged agitated vessels it is helpful to determine the contribution of each scale of mixing on k_La. Details of theoretical models can be found elsewhere (Martín et al. 2008). Higbie's theory provides an expression for the liquid film coefficient (Equation 7-3). According to it, k_L depends on the turbulence intensity expressed as dissipated energy as long as the surface removal is quicker than in the case of bubbles rising under potential flow,

$$k_L = 2\sqrt{\frac{D_{ab}}{\pi.t}} \qquad \text{Equation 7-3}$$

Kawase and Moo-young (Kawase and Moo-Young 1988) proposed that the contact time between phases, t, could be considered as the ratio between the length of turbulence (η) and the turbulent velocity (u) defined by the Kolmogorov's theory of isotropic turbulence. So equation 7-3 can be written as

$$k_L = 2\sqrt{\frac{D_{ab}}{\pi}}.(\frac{\varepsilon_{ave}\rho}{\mu})^{1/4} \qquad \text{Equation 7-4}$$

In order to predict k_La, not only the liquid phase resistance to the mass transfer is needed, but also the contact area between phases. The specific area is calculated using the empirical equation of Claderbank's (Calderbank 1958).

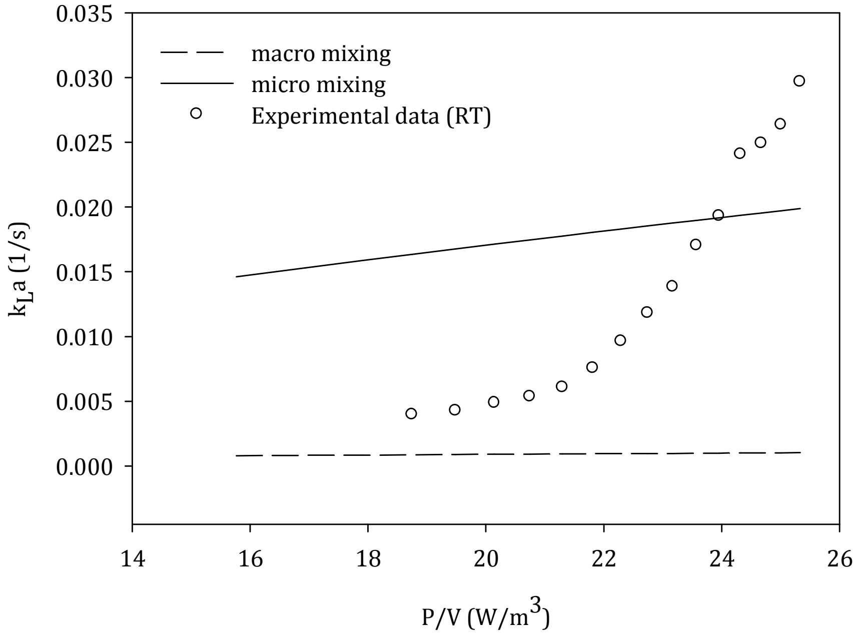
$$a = 1.44\left[\frac{(\frac{Pg}{V})^{0.4}\rho^{0.2}}{\sigma^{0.6}}\right]\left(\frac{vs}{U_\infty}\right)^{0.5} \qquad \text{Equation 7-5}$$

For macro-mixing the same correlations can be used, but micro-mixing contact time should be replaced by macro-mixing "mixing time".

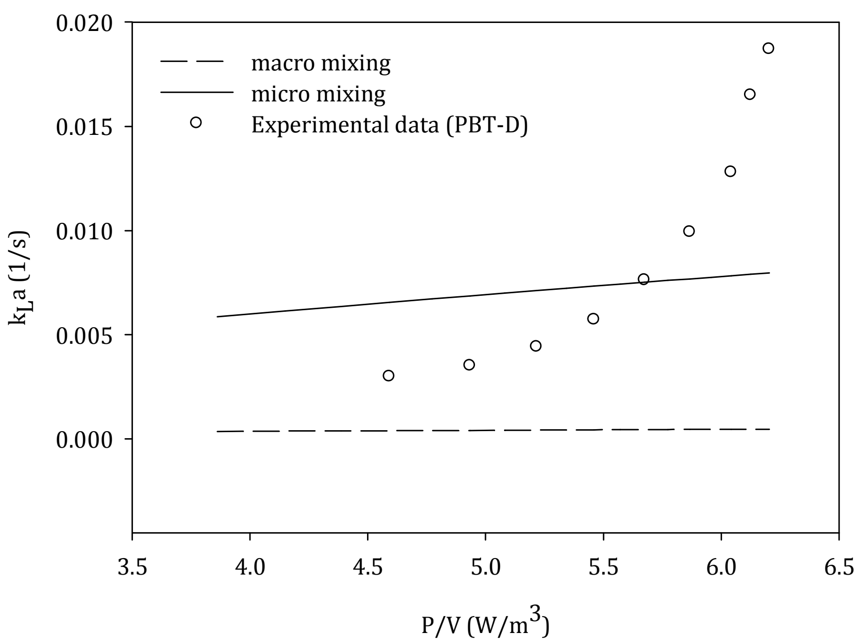
$$\theta = 5.9\left[\frac{T}{D}\right]^{-1/3}\varepsilon^{-1/3}D^{2/3} \qquad \text{Equation 7-6}$$

Figure 7-5 (a, b & c) illustrates the effect of impeller type on the contribution of micro-mixing and macro-mixing on k_La in a GL system. The impeller configuration determines the fraction of the tank where either macro- or micro-mixing prevails. Experimental data usually placed between micro and macro scale of mixing.

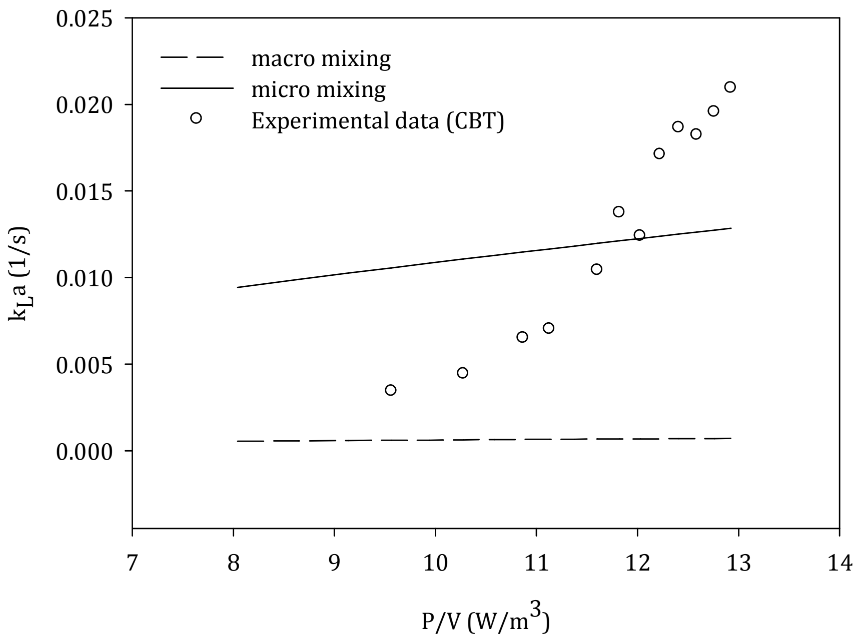
In most operating conditions macro mixing prevails, but by increasing impeller speed to very high values (>600 rpm) micro mixing prevails in the system. Since experiments have been carried out in a small scale vessel, this can be acceptable. At a high impeller speed, in a small vessel, mixing intensity is very high and the degree of segregation is very low. As a result, the entire vessel may experience high intensity turbulence, which increases the degree of micro mixing.


0.035
0.030
0.025
0.020
0.015
0.010
0.005
0.000
macro mixing
micro mixing
Experimental data (RT)
k_La (1/s)
14
16
18
20
22
24
26
P/V (W/m^3)

(a)


0.020
0.015
0.010
0.005
0.000
macro mixing
micro mixing
Experimental data (PBT-D)
k_La (1/s)
3.5
4.0
4.5
5.0
5.5
6.0
6.5
P/V (W/m^3)

(b)



(c)

Figure 7-5, Contribution of scale of mixing for different impellers, a) RT, b)PBT-D, c) CBT

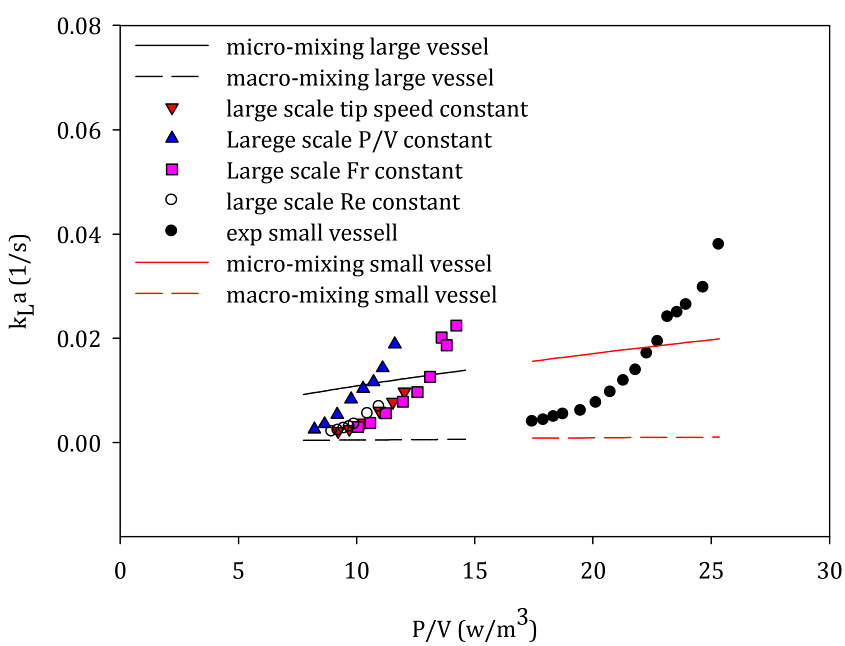


Figure 7-6, Contribution of scale of mixing and scale-up procedure

Scale procedures were evaluated in the previous section. In this section we intend to determine how macro-mixing and micro-mixing may change for different scale–up procedures. It is obvious from Figure 7-6 how the contribution of scale of mixing changes for different scale-up procedures. If we have another look at Figure 7-3 we will find out that the P/V constant and Fr constant are proper scale-up procedures while the Re constant cannot provide the appropriate conditions on a large scale. Comparing results illustrated in Figure 7-4 with those presented in Figure 7-6 indicated that the variation of k_La during scale-up on a large scale is caused by changes in the volume of the vessel where micro-mixing is prevailed. Such conditions could guarantee that the contribution of micro-mixing and macro-mixing remains unchanged, successful scale-up could be achieved.

7.4. Conclusion

Non-homogeneity of the gas-liquid mass transfer coefficient in a gas-liquid mechanically agitated vessel has been studied. Experiments have been done on two different scales and operating conditions of a large vessel were calculated following six different scale-up procedures. It has been shown that radial flow impellers can provide better gas dispersion compared to axial flow impellers. The presence of solid, especially at high content, significantly decreases the gas-liquid mass transfer coefficient. Non-homogeneity in the vessel and the effect of scale-up procedure on it were investigated. It has been shown that by following proper scale-up procedure kLa in large and small vessels is not distributed the same. The effect of scale-up and the contribution of scale of mixing for each procedure were also investigated and it was concluded that the best scale-up procedure is the one which can provide the same level of micro- and macro-mixing contribution on a large scale.

Nomenclature

a	Specific area (1/m)
C	Impeller clearance (m)

C_{O2}	Oxygen concentration in liquid phase (mg/l)
do	Orifice diameter (m)
d_b	Bubble size (m)
d_p	Average particle size (m)
D_s	Sparger diameter (m)
D	Impeller diameter (m)
D_{ab}	Diffusivity (m^2/s)
H	Liquid height (m)
Fr	Impeller Froude number N^2D/g
k_L	Mass transfer coefficient in liquid phase (m/s)
k_La	Gas-liquid mass transfer coefficient (1/s)
N	Impeller speed (RPM)
P	Power dissipation (W)
P_g	Aerated power input (W)
Re	Impeller Reynolds number $\rho ND^2/\mu$
t	Time (s)
T	Vessel diameter (m)
v or v_s	Gas superficial velocity (m/s)
V	Volume (m^3)

Greek letters

ρl	liquid density(kg/m^3)
ρs	Solid density(kg/m^3)
ε_{ave}	Specific power (w/kg)
μ	Liquid viscosity (Pa.s)
φ	Gas volume fraction (-)
σ	Surface tension (N.m)
θ	Mixing time (s)

Abbreviations

RT	Rushtun turbine
PBT-D	Pitched blade turbine in down-pumping mode
CBT	Concave blade turbine

CHAPTER 8

GENERAL DISCUSSION

The successful design and operation of LS and GLS mechanically agitated vessels require the accurate determination of which level of solid suspension and gas dispersion are essential for the process at hand. Engineers and scientists must define geometrical and operating conditions for specific medium (specified physical properties) in such a way that provides the optimum level of solid suspension and gas dispersion. This requires comprehensive knowledge about how the state of solid suspension may be affected by changing physical, operational, and geometrical parameters, how the quality of gas dispersion may change and how scale-up could affect process performance. Also, accurate empirical correlations or theoretical concepts are necessary to fulfill that objective. Failure to design the agitated vessel to achieve optimum conditions and maintain the system at these conditions during operation may cause significant drawbacks concerning product quality (selectivity and yield) and cost.

This dissertation intends to provide that background for scientists and engineers. It critically surveys most of the published work in this field and makes specific recommendations for the appropriate conditions that provide the successful operation of agitated vessels. It also identified certain deficiencies in the data reported in the literature and tried to fill in those gaps.

This thesis includes a comprehensive review of solid suspension, which presents a critical survey of the experimental works reported in the literature along with a detailed explanation of different experimental methods for characterizing the state of suspension and theoretical concepts for predicting that. It also provides comprehensive knowledge about how the state of solid suspension may be affected by changing physical, operational, and geometrical parameters. Such knowledge is vital for the successful design and operation of LS and GLS mechanically agitated vessels and to prevent significant drawbacks concerning product quality (selectivity and yield) and costs.

It also reports the limitations of applying conventional measurement techniques for the accurate characterization of N_{js} at high solid concentration. Consequently, the Gamma-Ray Densitometry technique for characterizing critical impeller speed for just off-bottom suspension (N_{js}) was introduced. The new method can overcome the limitation of previous experimental techniques. The application of Gamma-Ray Densitometry presents an effective and feasible approach for the accurate characterization of N_{js} when visual observation is not possible or other methods are not applicable (or accurate). The theoretical concept of this method is explained and experimental validation is presented to confirm the accuracy of the Gamma-Ray Densitometry technique. The effects of impeller clearance, scale, type, and solid loading on N_{js} for several impellers are discussed. It was clearly observed that, based on impeller clearance, axial and radial impellers operate differently. All impellers are efficient at low clearance. However, there exists a critical clearance where the flow pattern of the axial flow impeller changes and they subsequently cause the same effect as radial flow impellers. It was also shown that correlations for predicting N_{js} do not have universal validity. A new robust correlation for predicting N_{js} was proposed based on Gamma ray densitometry results. Finally, a theoretical approach was proposed based on the analogy between solid suspension in agitated vessels and the incipient movement of solid particles in pipes. This model shows good agreement with experimental data collected from literature. However, model accuracy could be improved by local solid-liquid characterization close to the bottom of the vessel. Future work involves characterization of the local hydrodynamic and fluid dynamic parameters of the liquid-solid mixing system by means of reliable methods to provide better understanding about suspension mechanism.

This dissertation also investigates the solid concentration profile at concentrated liquid-solid suspension by using optical fibers. The effect of impeller type and clearance were investigated as well as solid loading. Solid concentration distribution varies for axial and radial flow impellers. At the bottom of the vessel solid concentration is higher close to the wall for axial flow impellers compared to radial flow impellers. Solid loading has an effect on the solid concentration profile. Under extreme conditions a layer of solid lean region is generated at the top of the vessel. Its height deepens on impeller clearance and speed. It disappears at high impeller speeds. Modeling procedures were evaluated for predicting the solid concentration profile. Although sedimentation dispersion models are useful and accurate for predating solid concentration in dilute conditions, it fails to make the same prediction under concentrated conditions. In addition, power measurements have been taken to determine how the impeller power number may change in a liquid-solid mixing system. The presence of solid reduces the power number and the same trend was observed for radial and axial impellers. This reduction could be related to the changing flow pattern at the bottom of the vessel, which eliminated the baffle effect and the damping of turbulence for an impeller speed higher than Njs. However, there are discrepancies between results presented here and some published works. Scale-up procedures to achieve the same quality of suspension on a large scale were evaluated and it was shown that most of the scale-up procedures underestimated the energy requirement to provide the same homogeneity on a large scale. The lowest differences between RSD in small and large vessels is for the method proposed by Buurman (Buurman et al. 1985,1986). Results of this kind are considerable since experimental work on concentrated liquid-solid suspension was rarely published before. These results offer new information helpful to the understanding of the solid–liquid mixing processes.

Another part of this document is dedicated to gas dispersion in agitated vessels. A significant problem encountered in the scale-up of agitated vessels (especially for multi-phase applications) is the lack of knowledge about the effect of scale-up procedures on the fluid dynamics and hydrodynamics of the reactor. In most gas-liquid multiphase reactors non-homogenous distribution of dissolved gas is responsible for different performances on a large scale operation. The experimental approach has been considered in this study to describe how scale-up procedure affects the gas-liquid mass transfer coefficient (k_La) distribution. The hydrodynamics of the gas sparged-agitated vessel (gas-liquid and gas-liquid-solid) has been characterized in a small vessel by employing the dynamic adsorption desorption technique and fiber optics. Experiments have been repeated for a large vessel. The operating conditions of a large vessel were calculated by following different scale-up procedures. It is shown how gas hold-up distribution and fluid dynamic parameters change according to the scale-up procedure selected. Finally, k_La distribution has been predicted by using the multi-block modeling approach. In addition, the contribution of scale of mixing in k_La was explained and new scale-up procedures were proposed. The non-homogeneity of the gas-liquid mass transfer coefficient in a gas-liquid mechanically agitated vessel has been studied. Experiments have been done on two different scales and the operating conditions of a large vessel were calculated following six different scale-up procedures. K_La distribution is more homogenous if systems are scaled-up based on constant mixing time while the worst case scenario is scaling up based on a constant Reynolds number.

CONCLUSIONS AND RECOMMENDATIONS

In this dissertation solid suspension and gas dispersion in a mechanically agitated vessel were studied and the following conclusions can be drawn from the work.

1. Although extensive experimental and numerical studies have been done on solid suspension and gas dispersion in a mechanically agitated vessel, the successful design, operation, and scale-up of such system is still state-of-the-art.
2. Empirical correlation or theoretical methods cannot be generalized and there are very few empirical equations will global validity.
3. The reason for such uncertainty comes from the subjectivity of experimental techniques.
4. To overcome the limitations of experimental techniques and eliminate the subjectivity of methods a new gamma ray densitometry technique was proposed, which is able to provide accurate experimental data under dense conditions.
5. Empirical correlations were modified to be able to have precise predictions of Njs.
6. It was shown that the axial flow impeller at high clearance will loose its ability to suspend solid particles.
7. Characterization of solid concentration distribution in the vessel indicates the present solid concentration gradient, which is influenced by operating conditions and geometrical parameters.
8. k_La decreases in the presence of solid and its non-homogeneity in an agitated vessel could cause drawbacks on a large scale.
9. Taking into consideration the contribution of micro- and macro-mixing could help to achieve the proper scale-up procedure.

Further investigations could include the following:

- This study can be extended by considering other type of impeller specially hydrofoils.
- Characterizing Njs (Njsg) in viscous or non-Newtonian fluid is very challenging. The gamma ray densitometry can be used to attain accurate data tin those systems
- Detail information on solid motion in agitated vessel will help to extend the current knowledge about solid suspension and dispersion mechanism
- Gamma ray densitometry can help to achieve better understanding on fillet structure, deformation and generation in agitated vessel. That type of information will be valuable to achieve proper design.
- Solid dispersion and its mechanism in viscous and non-Newtonian fluids could attract a lot of interest. Detail information of solid concentration profile, scale effect and solid motion in those systems will extent the level knowledge to reliable design and optimum operating conditions.
- Characterizing dynamic behaviour of solid particles in LS and GLS agitated systems by BRPT also will provide valuable information on solid dispersion and suspension mechanism.
- Characterizing the distribution of k_La in large vessel experimentally and numerically could be subject for further studies.
- Current level of knowledge on gas dispersion in large-scale GL and GLS agitated vessel is very limited. Such information will help to overcome scale-up issues.

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