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Experimental Investigation and Optimization of Solid Suspension in Non-Newtonian Liquids at High Solid Concentration

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ABSTRACT

This research deals with experimental work on solid suspension and dispersion in stirred tank reactors that operate with complex fluids. Only suspended speed (Njs) throughout the vessel was characterized using Gamma-Ray Densitometry. The outcomes of this study help to understand solid suspension mechanisms involving changes the rheology of the fluid and provide engineering data for designing stirred tanks. All experiments were based on classic radial and axial flow impellers, i.e., Rushton Turbine (RT) and Pitched Blade Turbine in down pumping mode (PBT-D). Three different liquids (water, water+CMC, and water+PAA) were employed in several concentrations. The CMC solution introduced as a pseudo plastic fluid and PAA solution was applied as a Herschel Bulkley fluid. The rheological properties of these fluids were characterized separately. According to the findings, the critical impeller speeds for solid suspension for non-Newtonian fluids were more eminent than those for water. Experiments were performed to characterize the effects of solid loading, impeller clearance and viscosity on N_{js}. Also the PSO method is employed to find suitable parameters of Zwietering's correlation for prediction of N_{js} in Non Newtonian fluids.

Keywords: Liquid-Solid; Stirred tank; Gamma-ray densitometry; non-Newtonian fluids; Just Suspended Speed; PSO.

NOMENCLATURE

CMC	Carboxymethyl Cellulose	S	zwietering correlation constant
D	impeller diameter	*T*	vessel diameter
dp	mean particle diameter	*X*	solid loading: Ms/Mt
g	gravity acceleration		
H	liquid height	μ	viscosity
I	count rate (count/sec)	ρ	density
N_{js}	impeller speed (1/sec) at just suspended condition of LS system	ε	solid hold-up or volume fraction
		$\dot{\gamma}$	shear rate
P	poly acrylic acid		

1. INTRODUCTION

A major design objective in liquid-solid mixing systems is to obtain a complete off-bottom suspension of solids to render their surface area available to the processing. The surface areas of the solids which are not suspended are not available for contacting. Once the solids are suspended, their entire surface area is exposed to transport and/or chemical reaction. The hydrodynamic condition of a liquid-solid system is known as the just suspended speed and the impeller's speed which can provide this condition is known as just suspended speed (Njs). Many researchers used Zwietering's (Zwietering 1958)criteria and correlation in different liquid-solid mixing systems (Nienow 1968; Baldi *et al*. 1978; Rao *et al*. 1988; Armenante and Nagamine 1998) and reported that this equation could predict Njs accurately in turbulent condition (Nienow 1968).

$$N_{js} = S(\frac{g\Delta\rho}{\rho_l})^{0.45} d_p^{0.2} X^{0.13} \upsilon^{0.1} D^{0.88} \tag{1}$$

Table 1 Summary of studies that has been done with viscous and non-Newtonian fluids

Ref.	*Summary of work*
(Zwietering 1958)	Liquid phase were water, acetone, carbon tetrachloride, a solution of potassium carbonate in water, and light oil. The range of viscosity was between 1-9.3 cP widespread amount of experiments lead to equation 1.
(Hirsekorn and Miller 1953)	Worked in laminar flow regime using viscous fluids from 8 to 80 Pa s. They showed that solid particle can be suspended in laminar flow regime.
(WU *et al*. 2001)	Reported a reduction in Njs by increasing non-Newtonian viscosity, which was explained in terms of change of the ratio of the particle settling velocity over the tip speed caused by viscosity.
(Ibrahim and Nienow 2010)	N_{JSg} has been studied in water and in corn syrup of 0.01 and 0.1 Pas giving Reynolds numbers from the full turbulent region down to ~103.
(IBRAHIM and NIENOW 1999)	Particle suspensions in Newtonian fluids of viscosities from 0.01 to 1 Pa.s have been Studied. At high viscosity, there was less random particle movement across the base prior to suspension. On the other hand, once the agitation speed, N, was high enough to achieve suspension, i.e., N = Njs , particles remained longer in suspension after a reduction to N <Njs , though eventually with little or no hysteresis.

Which parameters S, g, ρ, dp, X, $\boldsymbol{\nu}$ and D shows Zwietering correlation constant, Gravity acceleration, Mean particle diameter, Solid loading, dynamic viscosity and Impeller diameter, respectively. Different experimental methods were used along with various criteria to characterize Njs in liquid-solid and gas-liquid-solid systems. Details of these schemes, accuracy and limitation were previously discussed comprehensively (Kasat and Pandit 2005; Jafari *et al*. 2010). By applying these techniques, researchers defined how Njs might change if the physical properties of liquid and solid or design of mixing system changed (Jafari *et al*. 2010). Since most of the solid suspension applications are in the turbulent flow regime, a few studies have been done considering transition and laminar flow regime and general effect of liquid viscosity has been broadly neglected. In Table 1 summarized the investigations on the effect of viscosity were. Due to the importance of mentioned subject, this study is conducted. The principal aim of this paper is to investigate the effect of viscosity, solid loading, impeller clearance and types of impeller on Njs in the stirred tank reactor for Non-Newtonian fluids.

2. MATERIALS AND METHODS

Water was used as a base liquid phase. To change viscosity and rheology of the liquid phase different amount of carboxy methyl cellulose (CMC) and polyacrylic acid (PAA) was dissolved in water. CMC and PAA solution rheology was characterized with a rheometer (Physica-MCR 301) at room temperature (20 °C). Viscosity of different liquid phase used in this study is shown in table 2. The uncertainty of viscosity is less than 5 percent that is calculated by repetition of measurement.

Table 2 Viscosity of different liquid phase used in this study

Solution	Viscosity (pa.s)
Water + 0.1 %wt CMC	0.003
Water + 0.1%wt PAA	0.002
Water + 0.3%wt CMC	0.012
Water + 0.3%wt PAA	0.004
Water + 1%wt CMC	0.14

The shear rate in an agitated medium was related to impeller speed by (Metzner and Otto 1957). In this study all the experiments were carried out at room temperature (20 °C) and agitation speed was in the range 5-20 s−1 (N=300–1200 RPM) which corresponds to $\dot{\gamma}$av =57.5-300 s−1. Sand was used as solid phase (density of 2650 kg/m3). Particle size distribution of sand (dp=240 μm) was measured by the Horiba laser scattering particle size distribution analyzer (model: LA-950).

Experiments were conducted in a transparent polycarbonate agitated cylindrical vessel with standard baffles, an open top and a flat bottom. Two different impellers were tested, mounted on a central shaft, namely a six blade Rushton turbine (RT) 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 Table3. The operating slurry height was set equal to the vessel diameter. Figure 1 shows the position of gamma source and detector in densitometry experiments.

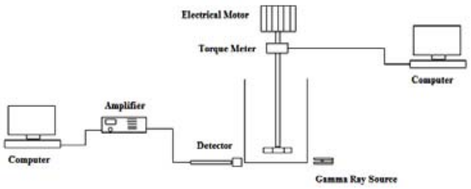

Fig. 1. Schematic of set up.

Table 3 Design details of mechanically agitated vessel

Parameter	Value
vessel diameter	0.2(m)
H/T	1
Baffle width	T/10
No. baffles	4
Material	Plexiglass
Geometry	Cylindrical with flat bottom
Impellers	PBT-D (4 blade), D=T/3 RT (6 blade), D=T/3

At the first step, each solution was made with the warm water (T~60°C) in the vessel. After liquid cooled down to room temperature solid particles were added to the liquid. The shaft was driven by a DC motor (Type 42A5FEPM, 130 V, 1.8 A, 0.25 HP, Bodine Electric Company). The gamma ray densitometry technique was used for characterizing Njs. (Details can be found in (Chaouki *et al*. 1997; Esmaeili *et al*. 2008; Jafari *et al*. 2010). A source of gamma ray consisting of a 2 cm glass bead filled with scandium oxide was activated 200 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 culminated by lead support. It passed through a 2 mm hole in the protective 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). At each impeller speed after system reached the steady state form and mixing condition fully developed gamma ray intensity were recorded for 5 min with sampling time of 400ms of different radial positions.

$$\varepsilon=\varepsilon_0\,\frac{\ln\left(\dfrac{I_{liquid-solid\,(N\succ 0)}}{I_{wateronly}}\right)}{\ln\left(\dfrac{I_{liquid-solid\,(N=0)}}{I_{wateronly}}\right)} \tag{2}$$

Solid concentration was calculated by the equation 2and it was plotted vs impeller speed for each case. To assure the repeatability of results each experiment at least repeated for three times and values reported here (and error) corresponds to average values.

Experiments were done in 108 different cases and each one repeated three times. The results presented here are the collection of all data.

The PSO method is used to find suitable parameters of Zwietering's correlation for prediction of Njs in Non Newtonian fluids. Particle Swarm Optimization (PSO) which introduced by Kennedy and Eberhart (Kennedy and Eberhart 1995) in 1995, is a form of swarm intelligence which mimics biological behaviour of folks of birds or a school of fish. When a swarm looks for food, its individuals spread in the environment and look for food independently from each other. When an individual finds a food source, announces the location of the food to other individuals so they can move toward it. PSO tries to simulate this behavior to solve optimization problems.

In PSO every solution for the optimization problem is an individual in the search space which is called “particle”. PSO starts with a population of randomly initialized particles and tries to produce better solutions in each generation. Every particle has a velocity vector which states the next location of the particle. In each generation, velocity vector of a particle is updated according to three vectors. First one is its current value, the second one is the best position that the particle has found so far (pbest) and the third one is the best position of the entire population that has been reached till current generation (gbest). After finding the pbest and gbest, the position of every particle is updated as

$$\begin{aligned}&\upsilon^{ij}(t+1)=w(t)\upsilon^{ij}(t)+c_1 r_1(x^{j}_{pbest}-x^{ij})+c_2 r_2(x^{j}_{gbest}-x^{ij})\\&x^{ij}(t+1)=x^{ij}(t)+\upsilon^{ij}(t+1)\end{aligned} \tag{3}$$

Where t is generation number,w(t) is a coefficient called inertia factor, r_1 and r_2 are random numbers uniformly driven from interval [0 1], c_1 and c_2 are positive constants called self-recognition constant and social constant respectively, x^{ij} is j-th dimension of i-th particle, x^{j}_{gbest} is j-th dimension of p_{best} and x^{j}_{gbest} is j-th dimension of gbest.

For increasing exploration, in each iteration, mutation operator for some individual could be practiced. Mutation changes a little portion of the particle randomly.

3. RESULTS AND DISCUSSIONS

Non-Newtonian fluids with the range of viscosity from 1 mPas (water) to 140 mPas were employed as well as two types of impellers at different clearances and different solid loading (10% wt to 50% wt). Figure 2 shows typical results of densitometry technique. When impeller was not rotating solid particles were resting at the bottom of the vessel. By increasing impeller speed solid particle was picked up from stagnant solid volume and dispersed in the vessel.

X and Y-axis show the impeller speed and amount of energy that detector absorbs respectively. In low impeller speed, the particles settle down in bottom of the vessel because of the low drag force. It means impeller does not have enough energy to suspend the solid particles, so the density of solid particles is high in the bottom of the vessel and they absorb most of energy that comes from tracer (region 1). The particles start to suspend in the liquid by increasing the impeller speed and the amount of solid particles decreases by impeller speed increment in bottom of the vessel that allows gamma ray energy goes to the detector more, so the count recording in detector increases according the impeller speed increment (region 2). The income energy will be constant when all the solid particles suspend (Njs) in the liquid and the concentration of solid does not change by increasing the impeller speed (region3), so the plateau region will occur.

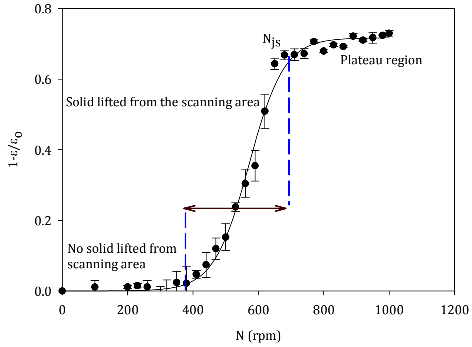

Fig. 2. Typical densitometry results, Variation of average solid hold-up by increasing impeller speed at the bottom of the vessel. Impeller: RT, X: 20%, dp: 240 μm, C/T: 0.33.

By using PSO method, parameters of Zwietering's correlation for prediction of N_{js} of Non Newtonian fluids are calculated and have been shown in table 4.

$$N_{js} = S(\frac{C}{T})^{\Delta} \upsilon^{\alpha} \left[\frac{g_c(\rho_s - \rho_l)}{\rho_l} \right]^{\beta} d_p^{\gamma} D^{\delta} X^{\theta} \tag{4}$$

Table 4 Zwitering parameters for Non Newtonian fluids

	PBT-D CMC	RT CMC	PBT-D PAA	RT PAA	PBT-D CMC
S	0.6894	1.3971	0.6078	0.8401	0.6894
Δ	0.1601	0.1331	0.1783	0.1047	0.1601
α	0.1762	0.1429	0.1363	0.0510	0.1762
β	-0.7172	-0.1784	-0.1577	-0.7445	-0.7172
γ	-0.5368	-0.4398	-0.2600	-0.4356	-0.5368
δ	-0.7545	-0.1278	-0.7553	-0.4636	-0.7545
θ	0.1142	0.0937	0.2451	0.1378	0.1142

Figure 3 illustrates the effect of viscosity on Njs. Increasing liquid viscosity increased Njs. Viscosity increment, or any change in liquid rheology (from Newtonian to non-Newtonian) can significantly change flow pattern and regime in a stirred tank from turbulent (Re>104) to transition (10<Re<104) to laminar (Re<10). Effect of viscosity on Njs mostly expressed as empirical correlations similar to equation 1. Increasing the viscosity of the liquid may not significantly affect Njs since most applications of solid suspension are in the turbulent regime. This also can be concluded from the low exponent of ν in Njs correlations (equation 1). However, at high viscosity of the fluid or whereas 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 a 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. This phenomenon was observed during tests with 1%wt CMC solution and solid loading higher than 10%wt where apparent viscosity was 140 mPaS. When impeller was placed at (C/T) higher than 0.15 flow generated from impeller could not transfer enough momentum to solid particles and pick them up from vessel base. Increasing impeller speed only produced enough liquid pumping that could lift solid particles from the surface of settled solid bed. Lifted solid particles only could be dispersed throughout the small volume of the vessel. Three distinct zones were observed, i.e. 1) settled solid bed at the bottom; 2) partially suspended solid particles in impeller region and 3) solid lean region mostly at the top of the vessel. Change of Njs could have been particularly connected with reductions in impeller pumping capacity as the fluid became more viscous (WU *et al*. 2001). The event was really complex because although the flow needed to suspend particles was lowered, the velocity at which they settled also reduced. At higher viscosity it was observed that when particles got suspended, they did not settle at all (or took very long time to settle). Increasing agitation in this condition may not have helped neither improved solid suspension nor solid dispersion. Reduction in Njs by increasing non-Newtonian viscosity also could be explained in terms of the ratio change of the particle settling velocity over the impeller agitation velocity caused by viscosity (WU *et al*. 2001).

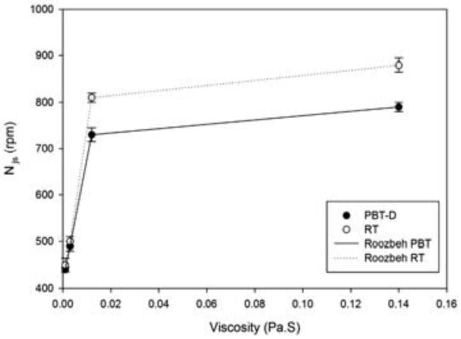

Fig. 3. Effect of viscosity on Njs at solid loading 10% and clearance 0.1 for different concentrations of CMC solution.

Most of the researchers observed no quantifiable effect on Njs with increasing kinematic viscosity for a low viscous medium. There are few studies that have been done for viscous or non-Newtonian fluids (Hirsekorn and Miller 1953; Flugg *et al*. 1977; Ibrahim and Nienow 1994; Kushalkar and Pangarkar 1995; Kawase *et al*. 1997). As Ibrahim and Nienow (Ibrahim and Nienow 1994; IBRAHIM and NIENOW 1999; Ibrahim and Nienow 2010)concluded, for low viscosity fluids (μ<100 cp) the Zwietering correlation had about a 10% uncertainty, while for more viscous systems (μ<1000 cp) the errors were greater (up to 90%). Figure 3 shows, Roozbeh's equation can predict Njs for Non Newtonian in both axial and radial flow patterns.

Figures 4 and 6 shows effect of solid concentration on Njs for two different type of impellers (Rushton & RPT-D) with two different non-Newtonian fluids (Herschel Bulkely & Pseudo plastic).

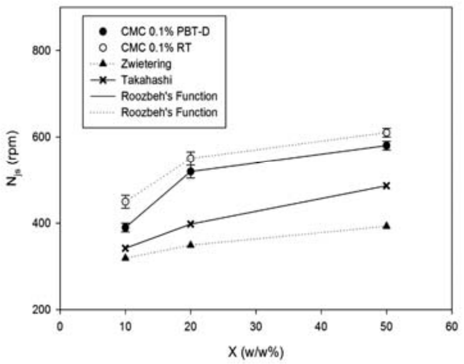

Fig. 4. Comparing Effects of Solid loading on Njs , Clearance 0.1.

Upon increasing solid loading, more power was required to suspend large portions of solid because the global density of fluid increases. Presence of solid particles can change the flow pattern of the fluid. At lower solid loading, this effect is negligible, but at high solid loading, it may become more significant. At high solid loading, with viscous liquid, the viscosity increase may become sufficiently high to move the process into the laminar regime. Depending on the size of the solid particles the system may become shear thickening and power input may significantly increase. Solid pickup from the vessel base can be expressed as a combination of burst of the eddies which can pickup and suspend solid particles and flow pattern of liquid that can push solid particles to the wall and lift them from there. Increasing solid concentration and viscosity of fluid, either together or individually, can decrease the energy of eddies and damp fluid momentum to suspend solid particles. By increasing solid loading energy of impeller will be spent to keep particles in suspension condition rather than accelerating fluid to suspend more particles. Figure 4 shows, Roozbeh's equation can predict Njs for Non Newtonian in both axial and radial flow patterns because the classical models did not care about rheological properties of base fluid. As observed, at lower viscosity (μ<10 mPas), impeller has been able to provide enough momentum to reach to just suspending conditions, even with high solid loading (X>40% wt). However, when fluid became more viscous, just suspending condition couldn't be achieved at standard condition. Hindered settling phenomena can occur in agitated tanks, which expected to make solid suspension much easier to accomplish. In the hindered settling state, solids do not settle as readily as if they were in free fall. Other surrounding particles and viscous nature of fluid hinder free fall.

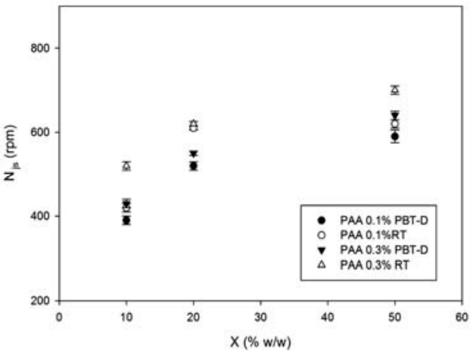

Fig. 5. Effect of Solid loading on Njs , Clearance 0.1.

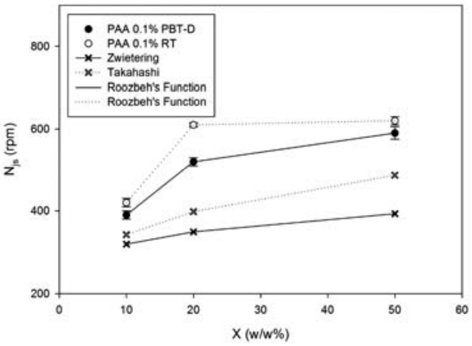

Fig. 6. Comparing Effects of Solid loading on Njs , Clearance 0.1.

McDonough (1992)considered that solids finer than 200-mesh and in concentration greater 40% by weight exhibited hindered settling. Hindered settling may help to keep solid particles in suspension, but do not serve to achieve just suspending condition. In viscous fluid, while solid particles reach the top region of the vessel where the bulk flow pattern of liquid is not strong enough to keep solid particles suspended it was observed that solid particles were moving down close to the vessel wall and in some points they were stagnant. As illustrated in Figure 4 and 6, Zwieteringequation was not able to predict Njs at viscous conditions with high solid loading. Inaccuracy went as high as 90 %. Figure 6 shows, Roozbeh's equation can predict Njs for Non Newtonian in both axial and radial flow patterns.

The degree of solid suspension in stirred tanks 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 examined a variety of impellers for solid suspension. The choice of a given impeller to achieve maximum solid suspension with a minimum power requirement is the key to the technical and economic viability of the process. NJS 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. Two types of impellers have been studied in this article: Rushton Turbine (RT), Pitched Blade Turbine in down-pumping mode (PBT-D). Axial flow impellers (like PBT-D) were more favorable for liquid-solid mixing processes since they could provide a better quality of solid suspension at lower impeller speed compared to radial flow impellers (Armenante and Nagamine 1998; Atieme-Obeng *et al*. 2004).

There are many characterization studies regarding the flow pattern of radial and axial flow impellers (for example (Kresta and Wood 1993; Armenante and Chou 1996; Kumaresan and Joshi 2006)). The radial flow generated with radial flow impeller first hits the wall and change direction, making motion upward and downward (Kresta *et al*. 2001; Kumaresan and Joshi 2006). Downward jet hits the 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. 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 (Kresta and Wood 1993; Jaworski *et al*. 1996; Zhou and Kresta 1996; Schäfer *et al*. 1998). Walling jet generated by axial flow impeller at the wall is stronger than the one generated by a radial flow impeller. The flow pattern of axial flow impellers facilitates suspension in comparison to radial flow impellers.

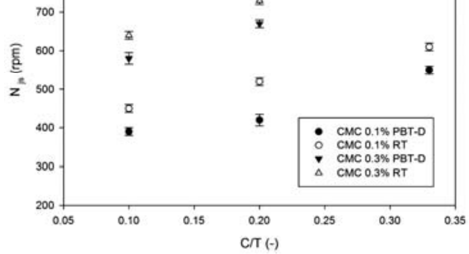

Fig. 7. Effect of Impeller type and clearance at solid loading 10% of CMC solution in different concentration.

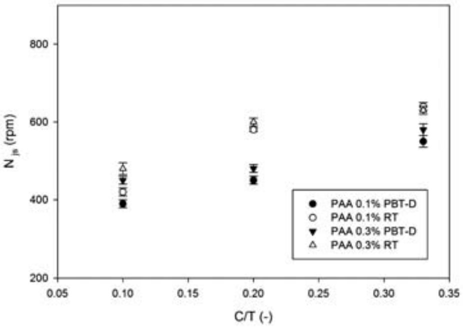

Fig. 8. Effect of clearance, Solid loading 10% for PAA solution in different concentration.

Increasing fluid viscosity could have changed flow pattern generated by an impeller. For axial flow impellers flow pattern should have hit the bottom first, then deflected toward vessel wall. At high viscosity and high clearance fluid deflection happened sooner as flow generated from impeller hit the wall rather than vessel base and impeller might have acted as a radial flow impeller. Ibrahim and Nienow (1994) found that the performance of axial flow impellers were more affected by viscosity than radial flow impellers because the flow changed from axial to radial. Also by increasing liquid viscosity maximum velocity of the wall jet generated by axial flow impeller decreased, which would have caused increased Njs and reducing cloud height. During experiments, it was noticed that even at just suspending condition, solid particles only could have been lifted to the certain height of the vessel which decreased by increasing solid loading and fluid viscosity. This could have been explained based on viscosity effect on wall jet velocity, which decreased significantly by increasing fluid viscosity.

The effect of the impeller clearance on the just-suspended speed is illustrated in Figure 7. Experimental results indicated that the clearance had a real effect on solid suspension. Critical impeller speed for off-bottom suspension, increased as the clearance was more. Energy transfer from the impeller to the particles was maximized in configurations where the impeller operated close to the tank base (Armenante and Nagamine 1998). When the impeller was placed near to the vessel base, the particles trapped at the tail end of the vessel underneath the impeller were initially driven toward the boxes. This centre-to-corner motion faced minimal resistance while accumulating sufficient momentum to lift into suspension after sliding to the junction of wall and vessel base. The variation of NJS as a function of impeller clearance was investigated in details by Sharma and Shaikh (Sharma and Shaikh 2003). In viscous system Njs increased linearly by increasing impeller clearance, which was different from what were observed in the case of water. While operating with high viscous fluids if impeller placed at high clearance (C/T>0.2) solid suspension may not happen at all. In this condition stream line initiated from impeller are not able to attain to the solid basis to lift solid particles or their velocity is low that strong wall jets cannot be brought forth. While operating in laminar flow conditions, classical impellers cannot be enforced. In this condition special design may be employed to be able to achieve solid suspension. Solid flow patter, scale-up and quality of solid dispersion can be considered as critical subjects to be able to fill the holes in the body of knowledge.

4. CONCLUSION

The technique of gamma-ray densitometry was chosen to overcome limitations of the conventional schemes for characterizing the suspended speed in liquid-solid mixing systems. Experimental data showed that the viscous Non-Newtonian fluid had a different flow pattern in mixing systems that had the common correlation failed in predicting their behaviours and correlations for predicting Njs did not have general validity. For axial flow impellers, the flow pattern should hit the butt first and then deflected toward the vessel wall. At high viscosity and clearance, the fluid did not have enough momentum to hit the bottom so deflection happened sooner, and impeller might have acted as a radial flow impeller. Likewise, higher solid loading caused an increase in required Njs. Upon boosting solid loading, more power was needed to set aside large portions of the solid, because the mean density of the system increased. Results show that the proposed equations can predict Njs of the Non Newtonian fluid in both Rheological properties. The errors of these correlations are less than 6%. Besides, these results show Rheological properties change all parameters of the Zwietering correlation.

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