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Determination of Minimum Agitation Speed for Complete Solid Suspension Using Four Electrode Conductivity Method

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Abstract. In many solid liquid operations, it is required to keep all the solids in suspension to avoid localised reactions, prevent the solid build-up at a particular location on the base of the vessel or just avoid clogging the outlet port. In operations like dissolution of a solid in a liquid, the employment of agitation speed over the just suspension speed (Njs) does not necessarily increase the effective surface area of the solids and hence increase mixing efficiency. Many workers have proposed the correlations for the Njs but it is widely accepted that the phenomenon is best represented by Zweitering correlation. The discrepancies in the correlations are probably due to lack of accurate Njs measurement instruments. In this paper, a sensor based on the four electrode conductivity measurement has been proposed to directly measure the Njs. This sensor relies on the change in the electrical resistivity as the solids settle over the electrodes. A relationship to determine the Zweitering constant has been investigated for the Rushton impeller using this technique. A 5-10% discrepancy in Njs values is normally found between the sensor prediction and the visual determination.

Keywords: Minimum agitation speed, Njs, Solid liquid mixing, Conductivity

INTRODUCTION

The idea of just suspension speed (Njs) was first introduced by Zweitering (1958) to measure the minimum agitation speed required to keep all the solid particles in suspension in a stirred vessel. At Njs, all the solids are completely suspended and no particle rests on the bottom of the vessel for more than 1-2 s (Zweitering, 1958). Many workers (Baldi (1978), Nienow (1968), Wichterle (1988)) have proposed the correlations for the Njs but it is widely accepted that the phenomenon is best represented by Zweitering correlation.

The discrepancies in the correlations are probably due to lack of accurate Njs measurement instruments. Zweitering (1958) used visual methods which were simple but subjective. The actual value of the Njs varies among researchers who studied the same particle system. Zweitering (1958) gave a value of 10% error for his experiments relying on visual method. The Njs measurements become complicated for the systems whose suspensions are usually opaque even at lower concentrations and make it harder to visually measure the Njs.

Mak (1992) used Ultrasonic Doppler Flow meter (UDF) placed on the bottom of the vessel to directly measure the Njs. The probe measures the frequency shift of an ultrasonic signal reflected from the particle discontinuities in the flowing fluid. The maximum measurement error in Njs as recorded by Mak for UDF was about 10%.

Measuring the local solid concentration is also one of the popular methods to determine the Njs. Recently, Ricard et al (2005) used Electrical Resistance Tomography (ERT) with a linear sensor to measure the average resistance in the bulk of the mixing vessel to predict the Njs. They noted down the impeller speed at which the measured bulk solid concentration becomes constant. It was assumed that when the solid concentration in the bulk remains constant, all the solids have been suspended and no more solids are remaining on the bottom of the vessel. Ricard reported the maximum measurement error to be 10%. The use of a linear ERT probe may not give the exact impeller speed at which the particles have ‘just’ started to suspend (as per Zweitering’s definition) but gives a fairly good idea of the Njs. Similar Njs experiments of measuring the local solid concentration by photometric method (Fajner et al, 1986) were also reported in the literature.

Zweitering (1958) used dimensional analysis to determine the correlation for the Njs and used his experimental data to determine the exponents of various suspension parameters (Eqn 1).
Zweitering presented the plots of correlation parameter ‘s’ against T/D at various impeller clearances. ‘s’ is a constant (for particular impeller and T/D) in the correlation and is supposed to take into account the impeller clearance and the characteristic flow profile generated by the impeller (Eqn 2).

\[ s = \int \left( \frac{T}{D} \right) \mathrm{d} \]

For all impellers, but for 6-blade fan turbine he studied, the constant s decreases with decreasing clearance ratios. But for the 6-blade fan turbine impeller, the s parameter actually remains constant through a range of impeller clearances. This observation of Zweitering is rather surprising as many researchers like Armenante et al (1998) and Nienow (1968) (including present authors) have observed that the particles are suspended at lower impeller speed at lower clearances even for the 6-blade turbine than opined by Zweitering.

So, Armenante et al (1998) proposed the modified Zweitering correlation, in effect regressed their data in the Zweitering correlation using s as a function of T/D and T/C (Eqn 3)

\[ s = a \left( \frac{T}{D} \right)^b \exp \left( c \frac{C_b}{T} \right) \]

C_b in eqn 3, is the clearance of the impeller from bottom of the vessel to the impeller bottom. The modified Zweitering as given by Armenante et al is given in Eqn 4 and 5.

\[ N_s = \left[ 2.10 \left( \frac{T}{D} \right)^{1.18} \exp \left( 0.24 \frac{C_b}{T} \right) \right]^{0.1} \left( \frac{\mu_f}{\rho_f} \right)^{0.1} \left( \frac{d^{0.2} X^{0.13} \rho_f - \rho_f^2}{D^{0.85}} \right)^{0.45} \text{ for } C/T > 1/5 \]

These empirical models present possible ways to estimate N_s based many known process conditions that may be difficult in practice. In this paper, we propose the use of a 4-electrode conductivity sensor to directly measure the N_s of the solid particles.

**METHODS AND MATERIALS**

We propose to use a 4-electrode conductivity sensor to measure the N_s. The 4-electrode sensor was essentially a conductivity measuring instrument employing one pair of electrodes to inject the current (source) and the other electrode pair to measure voltage (signal). The sensor was placed at the base of the vessel. The agitation speed was reduced gradually in steps and the change in voltage due to the solid settling was measured. The N_s was calculated based on the voltage profile.

The electrode construction can be referred from other articles (Wang 1995, 2005). Each electrode used in the experiment was 2 x 2 cm² in dimension and was 1 cm apart from the adjacent electrode. An additional reference ground electrode was also provided and the dimensions were the same as that of the measuring electrodes. The two sets of 4-electrode sensors with the ground electrode were mounted on a flexible non-conductive rubber base of 5 mm thickness. The flexible rubber base was to ensure that the electrodes were placed firmly on the curvature of the base of the vessel. The whole arrangement was fixed to the base of the vessel (See figure 1).

The data collection from the sensor was automated by a data acquisition system (ITS P2000) and the AC (9.6 kHz) current injection and voltage measurement were controlled by the DAS. The optimum current injection of 5 mA was selected with the insurance of no signal saturation at any processing condition after carrying out various optimising studies on current injection quantities. In the experiments reported, particles used were white, spherical and had a diameter of 3 mm and a density of 1051 kg/m³ which were custom made for the settling experiments. The mixing vessel was made of Perspex material with a dished bottom and with 4 standard baffles (width=T/10). The vessel contents were agitated with an IKA power vise® mixer which was capable of communicating with a computer via RS232 port. The mixer was programmed to ramp down the agitation speed by 5 rpm. The contents of the vessel
were stirred with an impeller (6-blade Rushton and A315 impellers) arranged in the centre of the vessel. The diameter of Rushton impeller used was 0.5T and that of A315 was 0.42 T. The experimental arrangement is shown in the figure 2.

FIGURE 1. The construction and measurement configuration of the two pair of 4-electrode sensors for measurement of the N_{js}, (a) shows the schematic diagram of the sensors, (b) an enlarged photograph of the sensors (c) the sensor is placed on the bottom of the vessel where the voltage measurement electrodes are in the centre of the base of the vessel.

FIGURE 2. The experimental set-up showing the arrangement of N_{js} sensor on the base of the vessel.

The determination of the N_{js} was made from the 4-electrode sensor measurement as well as visual observation by two other researchers by increasing and decreasing the stirrer velocity by 5 rpm at regular intervals.
RESULTS AND DISCUSSIONS

When the impeller speed is close to the Njs, the location of the earliest particle settling depends on the characteristic flow profile established at the base of the vessel and also depends on the particle properties (Mak, 1992). The Rushton impeller, being a radial type, tends to make a particle heap at the centre of the base of the vessel; hence the four electrodes are arranged in the centre of the vessel in the present study.

Figure 3 shows the evolution of the voltage as the agitation speed is increased. At very low impeller speeds (relative to Njs), the solids are at rest at the bottom of the vessel. At this stage, the particles are very densely packed on the electrodes and the voltage recorded is very high. As the impeller speed is gradually increased and the speed begins to approach the actual Njs, the particles are in partial suspension and there is exponential decrease in the voltage as the resistance of the fluid nearest to the sensor decreases. When the impeller speed reaches Njs, the particles are in complete suspension and off the bottom offering little resistance between the electrodes (the voltage recorded at this stage is very less as there are very less particles resting on the electrodes).

An exponential first order decay curve is fitted to the experimental data (figure 3). A cut-off point of 5% of the maximum voltage at fully settled condition is considered to accommodate the change in the sensor voltage due to the presence of the particles closest to them during the impeller speeds just before reaching Njs. After the cut-off point, the particles are considered completely suspended. This 5% cut-off line, though arbitrary, is necessary to compensate the resistance offered by the particles closest to the electrodes. The Njs measurements made by conventional visual observations were also found to be in agreement with this experimental data analysis.

![Figure 3](image-url)  
**Figure 3.** The experimental data is fitted with an exponential decay curve and the N_{js} is at the intersection of the 5% cut-off line and the decay curve. The recording of the voltage (at impeller speeds >Njs) shows a linear relationship with the bulk solid concentration. The voltage curve shows exponential trend when the N_{js} is being reached.

The particle settling curves are obtained as the normalised voltage vs. impeller speed and can be described by the correlation

\[ y = A e^{-N_{js}/B} \]  

or can be rearranged as

\[ N_{js} = -B \ln \left( \frac{y}{A} \right) \]  

The \( y \) value is the chosen cut-off value of 0.05 (i.e. 5%) depending on the sensitivity of the sensor to particle concentration. The constants \( A \) and \( B \) are regressed with the experimental data (eqn 7 and 8) and are described as a function of impeller clearance and concentration.

\[ A = \left( 12.81 \frac{T}{C} - 16.95 \right) \chi^{14.09c - 17.9} \]  

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\[ B = 46.02e^{-0.39T/C} X^{0.24} Y^{-0.70} \]  

(8)

Hence, we are able to propose a new correlation to present the relationship between Ns and process conditions based on 4-electrode measurement, which is given by eqn 9. For the convenience of the discussion, we called it as the conductivity correlation (CC)

\[ N_s = -46.02e^{-0.39T/C} X^{0.24} Y^{-0.70} \ln \left( \frac{12.81T/C - 16.95}{X^{14.05} - 1.5X} \right) \]  

(9)

**Determination of s for Rushton turbine**

Zweitering constant, s (eqn 1), is considered a constant which is a function of impeller clearance and impeller diameter during the calculation of Ns. The plots of s vs. T/D and s vs. C/T show the relationship between the two functions (figure 4).

\[ s = 1.74 \left( \frac{T}{D} \right)^{1.09} \exp \left( 0.41 \frac{C}{T} \right) \]  

(10)

The regression of s vs. T/D and s vs. C/T data to determine the dependency is obtained from Armenante (eqn 3) and is given in eqn 11.

\[ N_s = \left[ 1.74 \left( \frac{T}{D} \right)^{1.09} \exp \left( 0.41 \frac{C}{T} \right) \right] \left[ \frac{\mu_f}{\rho_f} \right]^{0.41} \left[ \frac{d^2 X^{0.11}}{D^{0.05}} \right] \left[ \frac{\rho_p - \rho_f}{\rho_f} \right]^{-0.41} \]  

(11)

Various authors have contributed to the modification of the Zweitering relationship and the correlation obtained in the eqn 12 can be compared with other researchers in table 1.

| TABLE 1: Comparison of constants in the function of s on T/D and C/T by various authors |
|---|---|---|
| Armenante (1998) | 2.10 | 1.18 | 0.24 |
| This work | 1.74 | 1.09 | 0.41 |
Comparison between different correlation methods

The comparison was made between \( N_{js} \) predicted using the new conductivity correlation (CC) derived with the 4-electrode sensor method (Eqn 11) and the \( N_{js} \) estimated by Armenante correlation (AC) (modified Zweitering’s correlation, Eqn 4) in Table 2. Table 2 shows the results of CC match the trend observed by Armenante. The maximum error of 18.6% in the measurements is reflected at concentration 1% w/w and at clearance \( T/2 \). The average deviation of all the measurements is found to be in an acceptable range of below 6%. As can be observed in the table, the \( N_{js} \) speed is indeed less than those predicted by Zweitering at small impeller clearances.

**TABLE 2**: Rushton 6 blade impeller, diameter 0.5 \( T \)

<table>
<thead>
<tr>
<th>Concentration (%w/w)</th>
<th>Clearance</th>
<th>( N_{js} ) (RPM), CC</th>
<th>( N_{js} ) (RPM), AC</th>
<th>Deviation %</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.00</td>
<td>T/1.5</td>
<td>98</td>
<td>95</td>
<td>-3.1</td>
</tr>
<tr>
<td>2.50</td>
<td>T/1.5</td>
<td>118</td>
<td>107.5</td>
<td>-9.7</td>
</tr>
<tr>
<td>4.68</td>
<td>T/1.5</td>
<td>121</td>
<td>116.6</td>
<td>-3.7</td>
</tr>
<tr>
<td>1.00</td>
<td>T/2</td>
<td>108</td>
<td>91</td>
<td>-18.6</td>
</tr>
<tr>
<td>2.50</td>
<td>T/2</td>
<td>112</td>
<td>103.5</td>
<td>-8.2</td>
</tr>
<tr>
<td>4.68</td>
<td>T/2</td>
<td>113</td>
<td>112</td>
<td>-0.0</td>
</tr>
<tr>
<td>1.00</td>
<td>T/3</td>
<td>87</td>
<td>88</td>
<td>0.0</td>
</tr>
<tr>
<td>2.50</td>
<td>T/3</td>
<td>94</td>
<td>99</td>
<td>4.0</td>
</tr>
<tr>
<td>4.68</td>
<td>T/3</td>
<td>101</td>
<td>107.7</td>
<td>6.2</td>
</tr>
</tbody>
</table>

Average deviation 5.94

Similar procedure as described above was adopted to measure the \( N_{js} \) of the same particle system with A315 impeller. The results were compared with the data published by Harrop et al (HC) (Harrop et al 1997). The table 3 shows that there is about 10% deviation between the HC and CC. Harrop et al addressed about 7.9% deviation in their measurement of \( s \) for A315 impeller and the authors’ data fairly agree with their data.

**TABLE 3**: \( N_{js} \) for A315 impeller, \( D/T = 0.42 \), impeller clearance at \( C = T/4 \)

<table>
<thead>
<tr>
<th>Concentration (%w/w)</th>
<th>( N_{js} ) (RPM), CC</th>
<th>( N_{js} ) (RPM), HC</th>
<th>Deviation (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.00</td>
<td>100</td>
<td>110</td>
<td>9.0</td>
</tr>
<tr>
<td>2.50</td>
<td>135</td>
<td>124</td>
<td>-8.0</td>
</tr>
<tr>
<td>4.68</td>
<td>145</td>
<td>135</td>
<td>-7.4</td>
</tr>
</tbody>
</table>

Average deviation 8.13

Zweitering predicts a constant \( N_{js} \) over a range of impeller clearances, which is not what is observed by this and other authors. The reason for Zweitering to predict so could be because the difference between \( N_{js} \) at different clearances is too close to be observed by visual means.

**CONCLUSION**

A flexible 4-electrode conductivity sensor has been developed to directly measure the \( N_{js} \) in the solid-liquid mixing systems. The experimental results have shown that the sensor measurements and the visual measurements of \( N_{js} \) differ by 5-10%.

As the \( N_{js} \) is highly dependent on the size and shape of the particles the direct measurement sensor can be used to measure the ‘characteristic’ \( N_{js} \) with all the internals inside the mixing vessel and in a non-standardised mixing vessel. It can also be used in optically opaque mixtures and as a feedback control to monitor the varying requirements of \( N_{js} \).

The relationship between the Zweitering constant \( s \) and other variables like \( T/D \) and \( T/C \) give an insight into the dependency of \( N_{js} \) on impeller clearance and diameter. A very low impeller clearance is to be avoided if impeller is
bare metal and is not coated with non-conductive material (like powder coating) as it might affect the measurements (even though the impeller effect is subtracted during the reference measurements of the sensor).

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NOMENCLATURE

\( a, A \): Constant
\( b, B \): Constant
\( s \): Zweitering Constant
\( \mu_j \): Viscosity of the liquid (Ns/m³)
\( N_{JS} \): Minimum agitation speed for complete suspension (rps)
\( C \): Impeller clearance from the centreline of the impeller to base of the vessel (m)
\( C_b \): Impeller clearance from the bottom of the impeller to base of the vessel (m)
\( H \): Height of the liquid level in the vessel from the bottom of the vessel (m)
\( T \): Diameter of the vessel (= \( H \))
\( d \): Diameter of the particle (m)
\( D \): Diameter of the impeller (m)
\( \rho_p \) : Density of the particles (Kg/m\(^3\))

\( \rho_f \) : Density of the liquid (Kg/m\(^3\))

\( y \) : Cut-off point for the normalised voltage in settling plots on y-axis

\( X \) : Concentration of solids (%w/w)