

# Enhanced Fluidization of Nanoparticle $\text{TiSiO}_4$ with the Assist of Stirrer

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**Abstract:** Some experimental observations on the fluidization characteristics of nanoparticles in the form of agglomerates with assistance of stirrer are presented. The nano agglomerates consist of,  $\text{TiSiO}_4$ , with a primary particle size of  $<50$  nm, and air used as fluid media. Fluidization of ultrafine solid particles is widely used in a variety of industrial applications because of its unusual capability of continuous powder handling, good mixing, large gas-solid contact area, and very high rates of heat and mass transfer. As the particle size decreases the cohesive forces between particles increase. Therefore, fluidization of ultrafine particles becomes much more difficult as compared to the larger size particles and it needs efficient method. In the present work, the Richardson-Zaki equation is used, and a proposed criterion for agglomerate Zaki constant ( $n$ ) is presented in order to predict the effect of the increase of assisted stirrer on the bed. The fluidization behavior of the nanoparticle, including the fluidization regime, and the bed expansion, Voidage are also investigated. The results showed that smoother fluidization was observed with increasing agitation speed, because the agglomeration and channeling were reduced by the mechanical agitation. It was found that with the assist of stirrer, by increasing in the speed of stirrer initial bed Voidage increased.

**Keywords:** Fluidization, nanoparticles, bed expansion, stirring.

## I. INTRODUCTION

When a liquid or gas is passed at very low velocity up through a bed of solid particles, the particles do not move. If the fluid velocity is steadily increased, the pressure drop and the drag on individual particles increases and eventually the particles start to move and become suspended in the fluid, the terms fluidization and fluidized bed are used to describe the condition of fully suspended particles. Since the suspension behaves as a dense fluid, if the bed is tilted the top surface remains horizontal and the large objects will either float or sink in the bed depending on their density relative to their suspension. The fluidized solids can be drained from the bed through pipes and valves just as a liquid can and this fluidity is one of the main advantages of fluidization for handling of solids.

A fluidized bed is formed when a quantity of a solid particulate substance (usually present in a holding vessel) is placed under appropriate conditions to cause the solid/fluid mixture to behave as a fluid. This is usually achieved by the introduction of pressurized fluid through the particulate medium. This results in the medium then

having many properties and characteristics of normal fluids; such as the ability to free-flow under gravity, or to be pumped using fluid type technologies. The resulting phenomenon is called fluidization.

Gas fluidization of powders has usually been restricted to Geldart (1973) group A and B powders. Finer particles in the group C range ( $<30$   $\mu\text{m}$ ) are difficult to fluidize because of the strong interparticle (cohesive) forces between them. A number of studies (Chaouki et al., 1985; Iwadata and Horio, 1998; Morooka et al., 1988; Pacek and Nienow, 1990; Wang, Y., et al., 2002; Wang, Z., et al., 1998; Zhou and Li, 1999) have been conducted to investigate the fluidization of ultrafine particles in conventional fluidized beds. Some of these investigators used nanoparticles, which are at the extreme end of Geldart group C powders. It has been observed that group C particles including nanoparticles can sometimes be fluidized in the form of agglomerates, and has been called "agglomerating fluidization" by Iwadata and Horio (1998). However, the formation of agglomerates and agglomerating fluidization occur at a superficial velocity well above the theoretical minimum fluidization velocity. The work reported in the literature thus far includes experiments and theoretical modeling of hydrodynamic behavior and agglomerate size.

## II. EXPERIMENTAL

A schematic diagram of the gas fluidization system is shown in the figure 1. The system consists of a fluidized bed of fine particles, a flow meter and manometer to measure the pressure gradients. The fluidized bed is a vertical transparent column with a distributor at the bottom consisting of a ceramic plate about 2mm thick with a pore size of  $20\mu\text{m}$ . The column is a glass tube with an inner diameter of 34mm and an outer diameter of 50mm and a height of about one meter. To generate a uniform gas field, glass beads are placed in a chamber placed below the distributor and above the gas inlet to form a packed bed about 50 mm high. This is tightly attached to the column with the help of gasket, so that there is no leakage of air.

A wet filter is placed at the gas outlet to entrap any fine particle agglomerates. The filter may be dried later to obtain the fine particles back. During fluidization process a stirrer (a rod promoter) was hanged from the top of the

fluidized column to vibrate the bed as shown in. The stirrer was connected to a motor and speed of rotation was

varied by a Variac.

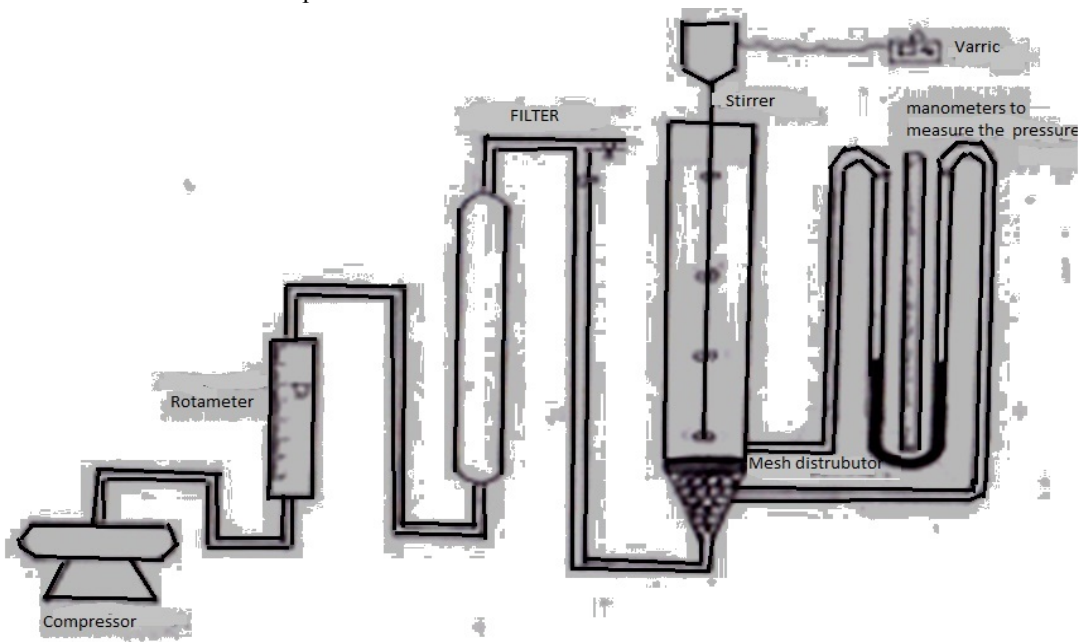


Figure 1 Schematic diagram experimental Set-up

The compressor is connected to one end of the flow meter and the other end of the flow meter is connected to the bottom of the chamber containing the glass beads. The glass beads help in generating a uniform gas field. On the top of the vertical glass column, an ultrafine mesh or a wet filter is placed. Pressure taps can be seen above and below the distributor, which is placed just above the chamber containing the glass beads, to measure the bed pressure drop. The pressure taps are connected to a U tube manometer through which the bed pressure drop is calculated. Pressure taps are present at the middle and top of the column.

Before the experiment is begun, the sand particles are sieved through an appropriate mesh and their size and particle diameter is determined. Sieving also serves to remove any large agglomerates formed during packing, storage and transportation. However, because of fragmentation and re-agglomeration during fluidization, some agglomerates in the bed might exceed the sieve openings. Because of the surface treatment by the manufacturer, the fine particles may either be hydrophilic or hydrophobic. To minimize the effect of humidity on the fine particle fluidization, pure air from a compressed air tank is used as the fluidizing gas.

The gas flow rate is measured and adjusted by using U-Tube manometer. The bed pressure drop is measured between the two pressure taps, one located at the top of the column near the flow exit and the other slightly above the distributor, so that it is not necessary to measure the pressure drop across the distributor. U tube manometers

are used to measure pressure drops. Pressure taps also placed at elevated height on the column to measure the pressure gradients. A stirrer of one meter length along with stirring leaves placed at equal lengths is introduced into the glass column to ensure uniform mixing. Particles are placed in the vertical column via opening at the top. Wet filter/ ultrafine mesh is fitted at the top and the particles are allowed to settle. The initial height of the bed is noted down. Compressed gas, set at a certain flow rate is allowed to move through the vertical column via the chamber of glass beads, for uniform distribution, and move through the pores of the distributor and hence bed expansion takes place. Values are noted down for both increase and decrease in the superficial velocity for both expansion of the bed and pressure drop respectively.

### III. RESULTS AND DISCUSSION

It was observed experimentally that mechanical agitator like stirrer helped breakup the channeling and spouting in a bed of nanosized powders. Previous studies on micron-sized powder fluidization (Qun Yu, Rajesh N. Dave, and Chao Zhu, 2005) and previous studies on nanoparticle fluidization (Jose Manuel Valverde and Antonio Castellanos, 2006) have shown that formation of agglomerates and their fluidization could be achieved at gas velocities considerably smaller than that based on the primary nanoparticle size) were adequate in our experiments, given that stirrer provided sufficient energy to the system to overcome interparticle forces and form stable agglomerates.

Assisted Stirrer fluidization:

We have found that, even when using the same nanoparticles, if we select agglomerates of same sizes, the bed will show very different fluidization behavior. For example, the Titanium silicon oxide agglomerates fluidize smoothly with large bed expansion (APF) at a low superficial gas velocity. Here, we define the superficial velocity as the gas superficial velocity without stirrer beyond which the bed pressure drop is no longer dependent on the gas velocity and becomes constant, and a relatively large bed expansion (typically 2 or more times the initial bed height) occurs.

Typical fluidization behavior of  $TiSiO_4$  nanoparticle agglomerates with and without the external assisted stirrer excitation is shown in Figure 2. Without the external assisted stirrer excitation, at a superficial gas velocity of

0.65 cm/s (Figure 2a), the nanoparticle agglomerates are first lifted as a plug and then the plug disintegrates to form stable channels through which the gas passes; the bed expands slightly with an uneven surface and the pressure drop is much less than the bed weight, indicating that the nanoagglomerate bed is not fluidized. However, if a sufficiently strong assisted stirrer field is applied, the nano particles are set in motion (translation and rotation) and the nanoparticle agglomerates are fragmented into smaller agglomerates because of collisions with the magnets, the vessel wall, and the distributor. After a few minutes, the channels disappear, and the bed begins to expand slowly and uniformly until it reaches its full expansion, of up to two times the initial bed height, and at the same time, the pressure drop reading is very close to the weight of the bed, indicating fluidization of the entire bed. A homogeneous fluidization state is established, and the surface is very smooth and even.

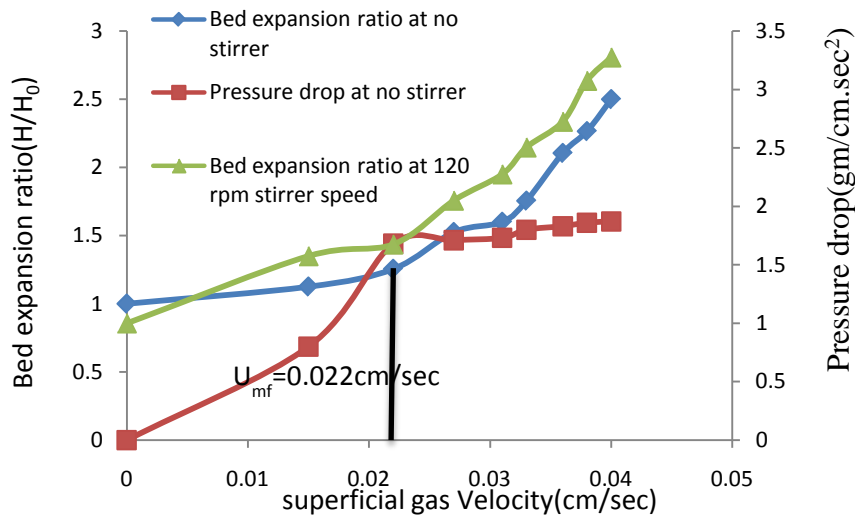


Figure 2. Bed expansion ratio and pressure drop for  $TiSiO_4$  with 120rpm and without Assisted stirrer.

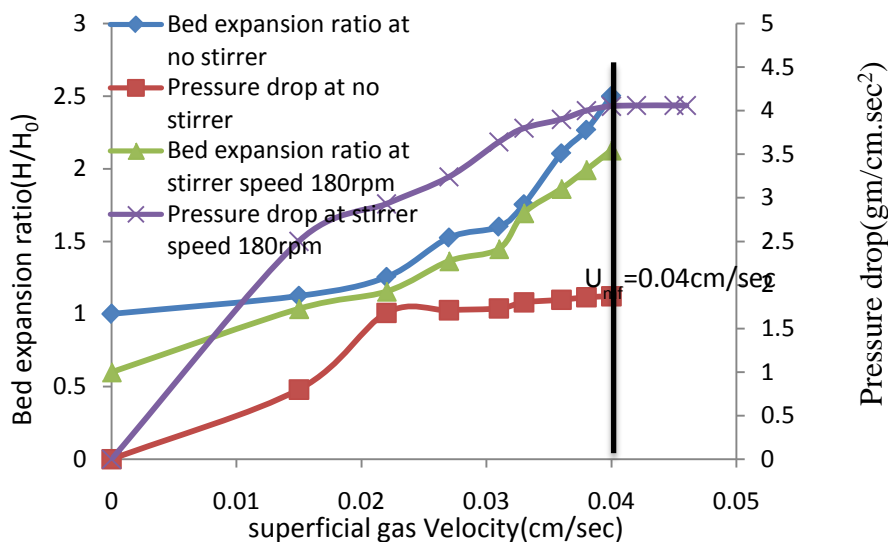


Figure 3. Bed expansion ratio and pressure drop for  $TiSiO_4$  with at 180rpm and without Assisted stirrer.

After the experiment, the powder is poured out, and from visual observation, most of the original large hard agglomerates are gone and the average agglomerate size appears very much smaller. The pressure drop normalized with the bed weight per unit area and the bed expansion ratio as a function of superficial gas velocity through the bed is shown in Figure 2, with and without assisted stirrer. It is clear from the figure that the stirrer excitation causes the bed to expand almost immediately as the velocity is increased and the bed fluidizes at a velocity more than one order of magnitude lower than that without stirrer assistance.

After separation from the particles, the nanoparticle agglomerates are recharged back into the column, and a second fluidization experiment with stirrer assistance is conducted using these agglomerates. Figure 3 is a comparison of the fluidization characteristics of the TiSiO<sub>4</sub> mixture, before and after Stirrer assistance processing. A significant reduction in the minimum fluidization velocity from 0.022 cm/s to 0.04 cm/s is observed, indicating that previous fluidization with Stirrer assistance causes the agglomerates to be fragmented into smaller ones and the average agglomerates size is reduced. However, the minimum fluidization velocity of these smaller agglomerates is still about an order of magnitude larger than the minimum fluidization velocity observed when the stirrer assistance is increased.

Mathematical model for bed expansion: The different models for correlation of bed expansion with superficial fluid velocity can be classified into three main groups. The first group is based on correlations giving the dependence between  $\frac{U}{U_i}$  and  $\epsilon$ . The Richardson and Zaki model is most popular in this group. Among all the correlations the Richardson and Zaki model is probably the most popular

one due to its simplicity and its good agreement with experimental data. It is based on the following equations.

$$\frac{U}{U_i} = \epsilon^n$$

Where the exponent n can be determined from

the correlations:

$$n = (4.4 + 18 \frac{d}{D}) \text{Re}_t^{-0.1}$$

for  $\text{Re}_t < 200$

$$n = 4.4 \text{Re}_t^{0.1}$$

for  $200 < \text{Re}_t < 500$  and

$$n = 2.4$$

for  $\text{Re}_t > 500$

$U_i$  The superficial fluid velocity at  $\epsilon=1$ , can be calculated from the following equation

$$U_i = U_t * 10^{-(d/D)}$$

Where  $U_t$  is the particle terminal velocity as given by the following equation

$$U_t = \sqrt{\frac{4(\rho_l - \rho_p)gd}{3\rho_l C_D}}$$

Where  $C_D$  is the drag coefficient

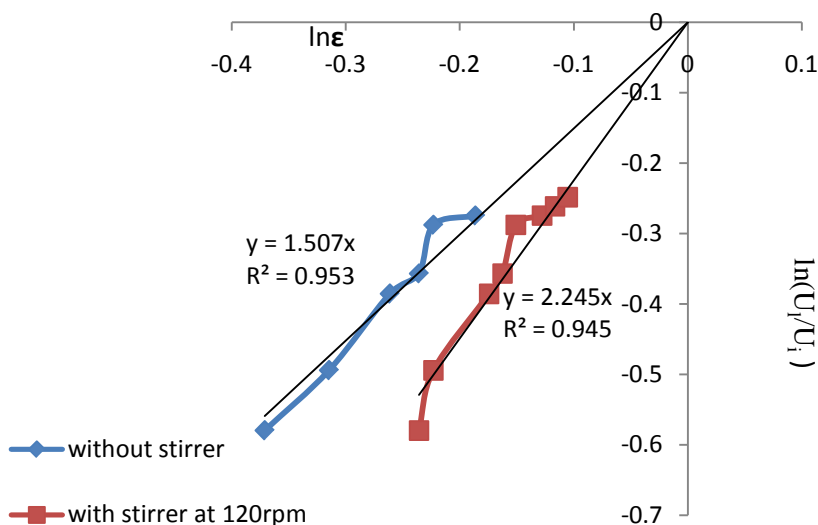


Fig 4: Effect of stirrer on Titanium silicon oxide bed expansion at without stirrer and with stirrer at 120 rpm speed

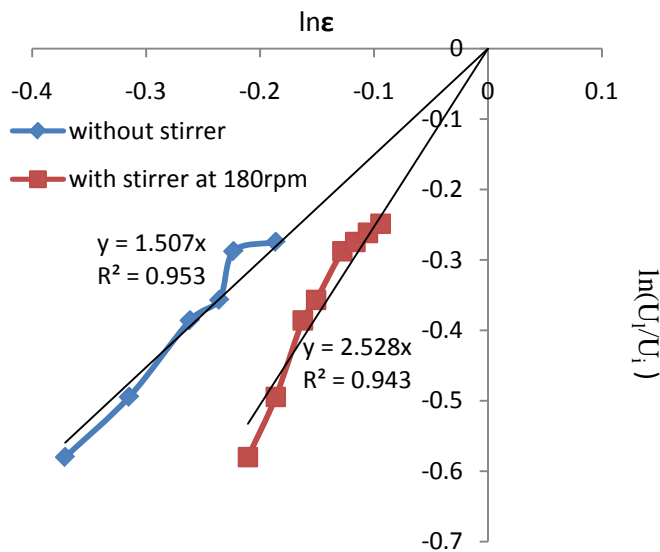


Fig 5: Effect of stirrer on Titanium silicon oxide bed expansion at without stirrer and with stirrer at 180 rpm speed

Effect of assisted stirrer on Mathematical model bed expansion:

The bed expansion was depends upon the superficial velocity of the air .The bed will be expanded by increasing the superficial velocity of the air.Here the bed expanded very high because here rotating stirrer was used as external force. From fig 4, the Richardson Zaki constant (n) was small in without stirrer because there external force not used so bed expansion slow but when the experimental procedure conducted with assisted stirrer at 120rpm was used as external force the bed expansion was increased larger than the without stirrer and the Zaki constant increased from 1.5 to 2.24. Also From fig 5, the Richardson Zaki constant (n) was small in without stirrer

because there external force not used so bed expansion slow but when the experimental procedure conducted with assisted stirrer at 180rpm was used as external force the bed expansion was increased larger than the without stirrer and the Zaki constant increased from 1.5 to 2.52

Effect of stirrer on bed Voidage:

It is observed that the bed Voidage of different nanoparticles size is increased with the increase in superficial gas velocity( $U_0$ ) and for the fluidized bed and as also increase with speed of the stirrer and thus indicating the different fluidization regimes in the column. Highly porous particles show particulate and bubbling fluidization behavior.

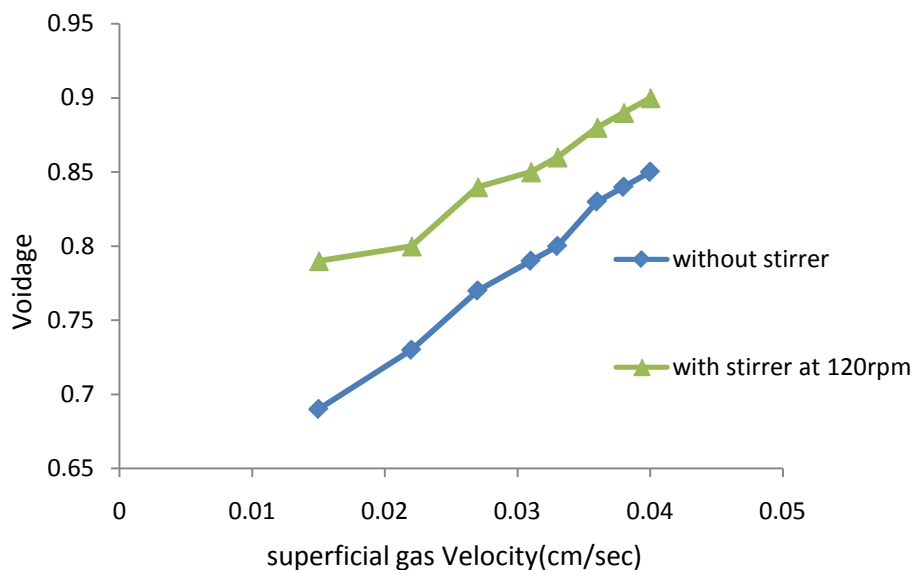


Fig 6: effect assisted stirrer on Titanium silicon oxide Voidage at without stirrer and with stirrer at 120rpm

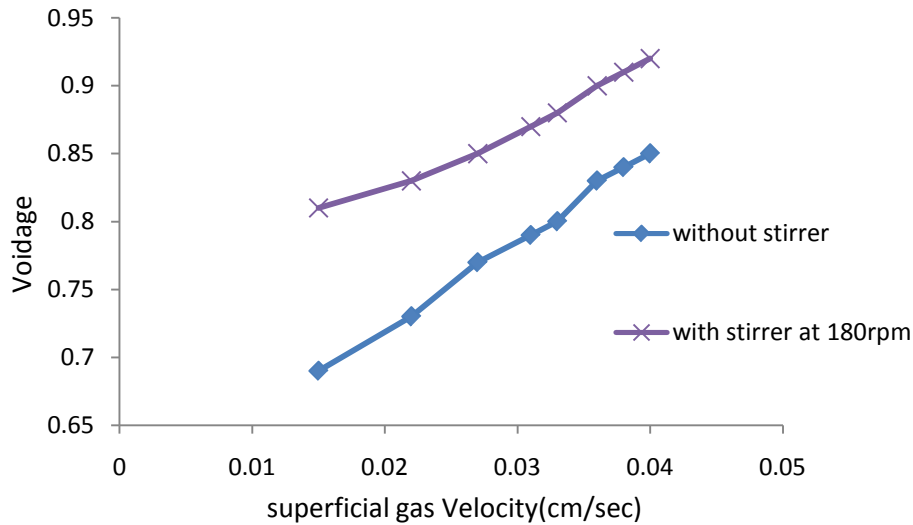


Fig 7: effect assisted stirrer on Titanium silicon oxide Voidage at without stirrer and with stirrer at 180rpm

The given correlation is used to determine the Voidage

$$m = \frac{H - H_{mf}}{H_{mf}} = \frac{1 - \epsilon_{mf}}{1 - \epsilon} - 1$$

Where  $H$  = Expanded bed height(  $cm$  ),  $H_{mf}$  = Initial bed height(  $cm$  )

$m$  = mass of nano materials(  $gm$  )

$$\epsilon = \text{Bed Voidage} = 1 - \frac{m}{\rho_p H}$$

$\epsilon_{mf}$  = Bed Voidage at initial bed height

By using the stirrer the bed Voidage increased than the without stirrer .From the fig.6 at without stirrer the initial bed Voidage 0.67,and when the experimental procedure was conducted with assisted stirrer at 120rpm the initial bed Voidage 0.78

From the fig.7 at without stirrer the initial bed Voidage 0.67,and when the experimental procedure was conducted with assisted stirrer the initial bed Voidage 0.81

Table1. Minimum Fluidization Velocities for Tisio4Agglomerates, at different assisted stirrer speeds

Experimental conditions	Without stirrer	With stirrer at 60rpm	With stirrer at 120rpm	With stirrer at 180rpm
$U_{mf}$ (cm/sec)	0.02	0.03	0.033	0.04

Table2. Effect of assisted stirrer on Richardson zakiconstant(n) for Tisio4Agglomerates, at different assisted stirrer speeds

Material	Initial bed heights (cm)	Stirrer speeds	Experimental (n)	Calculated (n)
Titanium silicon oxide	2	Without stirrer	1.5	0.89
		60rpm	1.81	
		120rpm	2.24	
		180rpm	2.52	
	3.2	Without stirrer	1.55	0.89
		60rpm	1.98	
		120rpm	2.29	
		180rpm	2.52	
	4.6	Without stirrer	1.99	0.89
		60rpm	2.33	
		120rpm	2.51	
		180rpm	2.82	

Table3. Bed expansion ratios for Tisio4Agglomerates, at different assisted stirrer speeds At the superficial gas velocity  $U_{gas}=0.04$ cm/sec

Experimental conditions	Without stirrer	With stirrer at 60rpm	With stirrer at 120rpm	With stirrer at 180rpm
Bed expansion ratio	2.5	3.02	3.27	3.54

Table4. Initial bed Voidage for Tisio<sub>4</sub> Agglomerates, at different assisted stirrer speeds

Experimental conditions	Without stirrer	With stirrer at 60rpm	With stirrer at 120rpm	With stirrer at 180rpm
Initial bed Voidage	0.69	0.73	0.79	0.81

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#### IV. CONCLUSIONS

- It was observed from the experiments and calculations the Richardson zaki constant (n) was increased by increasing the stirrer agitations
- It was observed that the minimum fluidization velocity( $U_{mf}$ ) was increased by increasing the assisted stirrer speed
- The results showed that smoother fluidization was observed with increasing agitation speed, because the agglomeration and channeling were reduced by the mechanical agitation.
- It was found for APF nano particle that with the assist of stirrer,by increasing in the speed of stirrer initial bed Voidage increased. The preliminary study has shown that fluidization of nanoparticle can be easily and smoothly fluidized with the assistance of External force.
- Minimizing the energy consumption. Therefore an external arrangement for creating some force On outside of the column will be best option to improve the bed dynamics as well as economy of the process.
- The bed expansion ratio and bed Voidage for the fluidized bed are observed to increase with the increased speed of the stirrer(N) as the rotation of the stirrer prevents the bubble formation, reducing channeling and agglomeration.

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