AIR INGRESS TO NITROGEN INERTED CHUTES

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Nitrogen purging of chutes used for adding powders to vessels can avoid the potential problems of igniting a flammable atmosphere within the vessel. Under steady nitrogen flow conditions, an oxygen profile is established within the chute. This is disturbed by the addition of powders, with fine powders being more disruptive than coarse powders. Plug flow adds far more oxygen to the vessel than poured powders. An equation is given for determining the flow and timing requirements for various powder addition regimes.

Keywords: Nitrogen, Inerting, Powder additions,

INTRODUCTION

The addition of powders to vessels down long chutes has the potential to generate electrostatic charges on the powder. Where the powder is ignition sensitive or there is a flammable solvent atmosphere within the vessel, there is the potential for ignition to occur. It is generally recognised[1] that for chutes up to 3 metres in length where the powder is not highly resistive, the risk of an incendive spark ignition is acceptably low. This was based on existing available evidence for chutes up to 3 metres in length, used to add powders to a vessel from the floor above. However, with increases in the size of process vessels, the floor-to-floor distance has increased, and chutes are now considerably longer than 3 metres. Consequently, it has been customary to reduce the oxygen content of the atmosphere within the vessel by the use of nitrogen to render it non-flammable.

Although nitrogen can remove the oxygen in the vessel, the interstices of the powder contain oxygen, and hence the addition of powder down a chute is liable to draw air into the vessel, thus destroying the inert gas blanket. The use of a continuous purge up the chute can be used to counter this, but little practical information is available on the rates of nitrogen needed to remove oxygen. There is a subsequent problem in that a flow of nitrogen up the chute at the same time as powder is tipped down the chute can lead to dust emission with the attendant hygiene issues.

The initial and subsequently modified work of Husa[2,3] indicated a logarithmic profile of oxygen concentration with distance from the open end of a vent pipe, under steady flow conditions. Under cross-wind conditions, Husa suggests increasing the flow by 60% to compensate for air ingress due to vortices. Bryce and Fryer-Taylor[4] consider the effect of vortices on the entrainment of air, and show that the oxygen concentration is non-uniform across the diameter of the pipe. The theoretical calculation of a suitable flow for removing oxygen carried in with a powder was used as an initial basis for the experimental work. From that initial work, the experiments continued to determine safe addition regimes for conditions where no nitrogen flow was used during the period that the powder was actually being poured down the chute.

THEORY

To determine the flow of nitrogen needed to remove any air entrained with solids requires certain assumptions to be made. These assumptions are pessimistic, but were made to ensure that under worst-case conditions, the oxygen level in a vessel would not rise above a certain value to ensure that any solvent atmosphere within the vessel would be non-flammable by virtue of being below the minimum oxygen concentration for combustion. This is typically 10% for common solvents, so a maximum oxygen level of 5% v/v was selected to allow a safety margin.

The other assumptions were that:

- The chute is full of nitrogen before adding the powder
- The powder falls as a solid, gas-tight plug down the chute
- The plug of solid draws air into the chute behind it
- The nitrogen purge removes the air from the chute
- Perfect backmixing is assumed within the empty chute
- End effects are ignored
- The time taken for the powder to fall down the chute is neglected when calculating the total cycle time of the addition

In order to determine the time for purging out the chute, a differential equation can be set up. Taking the volume of the chute to be V, the concentration of oxygen throughout the chute as c, and the nitrogen flow through the chute as Q, then the quantity of oxygen leaving the chute in a time increment δt is:

$$Q\,c\,\delta t$$

and the change in the total quantity of oxygen in the chute is:

$$-V\,\delta c$$

As any air leaving the chute corresponds to a reduction of concentration within the chute, then these two are equal. Therefore:

$$Q\,c\,\delta t = -V\delta c$$

and as δ approaches zero and the equation is rearranged:

$$dt = \frac{-V}{Q\,c}\,dc \qquad (1)$$

Integrating with respect to c between the initial and final concentrations, c_i and c_f, gives:

$$t = \frac{-V}{Q}\int_i^f \frac{1}{c}\,dc$$

$$t = \frac{-V}{Q}\left[\ln c\right]_i^f$$

$$t = \frac{-V}{Q}\left[\ln c_f - \ln c_i\right]$$

$$t = \frac{-V}{Q}\ln\left(\frac{c_f}{c_i}\right) \qquad (2)$$

Substituting in suitable values based on Plant experience of 10 m^3hr^{-1} up a 250 mm chute 4 metres long gives the following time to reduce the concentration from 21% oxygen to 1% oxygen:

$$t = \frac{-0.196}{10} \ln\left[\frac{1}{21}\right] = -0.0196 \times -3.0445 = 0.0597 \text{ hrs} = 215 \text{ sec} \quad (3)$$

If it is assumed that no backmixing occurs, ie the flow is perfect plug flow, then the time taken would simply be:

$$t = \frac{V}{Q} = \frac{0.196}{10} = 0.0196 \text{ hrs} = 71 \text{ sec} \quad (4)$$

EXPERIMENTAL

EXPERIMENTAL TEST RIG

All of the solids tipping experiments were carried out in the open air using a vertical 4 metre long by 267 mm actual bore (10" nominal bore) charge chute. A 200 litre steel drum was placed at the bottom of the chute to catch the solids, and the joint between the chute and the drum was sealed to prevent the ingress of air. A tun dish was placed at the top of the chute to facilitate the pouring of the solids.

The oxygen concentration within the chute was measured using Schools Type Oxygen Probes and Model OT4 Oxygen Meters, both supplied by Walden Precision Apparatus Ltd., of Cambridgeshire, UK. The probes could be inserted into the chute at any of nine pre-determined locations along the length of the chute. Semi-cylindrical inserts were provided at each probe position to protect the probes from damage caused by the falling solids. The outputs from the oxygen meters were logged on a Personal Computer using the Orchestrator Data Logging package. The oxygen measuring system was calibrated across the range 0 to 20.8% v/v oxygen before each experiment. The upper value was calibrated in fresh air and the lower value was calibrated using oxygen-free nitrogen. The response time of the probe/meter system across the full calibrated range was typically 1-2 seconds.

The nitrogen used in the experiments was Oxygen-Free grade supplied by BOC and the flow rate to the chute was measured using a Model 826-CE-NX-OV1-PV1-V1 mass flow meter by Sierra Instruments Inc. of Monterey, USA. The flow-meter was pre-calibrated using nitrogen by the manufacturer. As for the oxygen meters, the flow meter output was logged using the Orchestrator Data Logging package. The nitrogen supply was connected via a pressure regulator and the flow meter to either the bottom of the drum or the bottom of the chute. The nitrogen was directed to the required location by ball valves.

The wind speed was measured at the top of the charge chute using a Model TA400 hot-wire anemometer from Airflow Instruments, of High Wycombe, UK. Although the maximum limit of this meter was 2 m s^{-1}, this is satisfactory for measuring the range of air velocities typically occurring in a solids tipping booth. The experimental rig is shown in Figure 1.

EXPERIMENTAL PROCEDURE

Once the oxygen probes were calibrated, they were inserted into the chute at the required locations. The wind speed across the top of the chute was measured and the general atmospheric conditions were noted.

The drum was then purged with nitrogen until the oxygen concentration profile within the chute reached equilibrium, or steady state, conditions. The nitrogen flow was then diverted to the bottom of the chute and a short time was allowed to elapse to ensure that the oxygen concentration was still in equilibrium. The equilibrium concentration profile was then determined.

Aliquots of the powders were then added whilst the nitrogen flow was maintained, to determine the quantity of oxygen that would be carried down to the base of the chute.

Subsequent experiments were undertaken where the same equilibrium oxygen profile was established, and the nitrogen flow was then stopped. After a selected time delay, solids were tipped down the chute from bags or containers. In some of the experiments, further solids were added without re-purging the chute. This was done not only to try to gain an understanding of the mechanisms involved, but also in order to address a specific practical problem of avoiding dust being carried out of the chute by the nitrogen flow. The nitrogen flow rate and the oxygen concentrations at various positions in the chute were logged throughout the duration of each experiment.

In order to investigate the role of the physical form of the solid, three different types of solid were used in the experiments: dried peas, common salt and wood shavings (purchased as commercial pet bedding). Selected physical properties of these materials are given below:

Material	Bulk density kg m^{-3}	Void fraction	Particle size data	Comment
Dried peas	786	0.43	Mean diameter = 7.6 mm; σ = 1.1 mm	Free-flowing
Salt	1282	0.43	Mean diameter = 330μm 96% within range 150-600μm	Free-flowing
Wood shavings	104	0.79	60% coarse flakes; 40% less than 1680μm	Unpredictable - can be free-flowing or very cohesive.

Table 1

RESULTS

PURGING OUT THE AIR

The theoretical times to purge the chute alone vary from 71 seconds using Equation (4) for pure plug flow to 215 seconds assuming perfect backmixing calculated from Equation (2). Clearly perfect plug flow would be unlikely to occur in practice. Similarly, although the cross wind at the top of the chute would contribute to excellent mixing at the top of the chute, it would be unlikely to mix the entire chute. Thus the time would be expected to be somewhere between the two times.

Experimental data presented in Figure 2 shows that the actual time taken to purge the lower 3.05 metres of the chute, from 21% to 1% oxygen measured at 0.95 metres from the open end, is actually 75 seconds. The calculated time according to Equation (2) is 187 seconds, and according to Equation (4), 61 seconds. This is about 25% longer than the time for perfect plug flow, but only 40% of the perfect back-mixing time. Thus it can be seen that the perfect back-mixing equation is pessimistic, and therefore a purge-time calculated from Equation (2) is always safe. If the plug-flow case is taken, then a safety factor should be applied.

OXYGEN PROFILE UNDER STEADY STATE WITH A NITROGEN FLOW

While the nitrogen purge was applied to the chute, an "equilibrium" oxygen profile was established, which depended upon the nitrogen purge rate and the atmospheric conditions prevailing at the time. The oxygen profile was of a logarithmic nature. This is illustrated in Figure 3. However, both the mean and transient variability in the oxygen concentration were found to depend upon the wind conditions, particularly within the top metre of the chute. The transient wind effect was seen to move the oxygen concentration, at a depth of 0.3 m, by as much as ±5% (absolute) around the mean value during particularly windy conditions. This is shown in Figure 4.

DIFFUSION OF AIR INTO A NITROGEN-FILLED CHUTE WITH NO NITROGEN FLOW

When the nitrogen purge was stopped, the oxygen concentration was seen to increase in the chute as the air diffused into it. The rate of oxygen ingress depended upon the wind conditions and so was somewhat variable; particularly within about 1 metre of the top. In one example, the same oxygen profile observed in the chute after 15 seconds in windy conditions was the same as that seen after 1 minute in calmer conditions. However, with wind speeds typical of those experienced in a laminar flow booth above a chute to a vessel (i.e. less than about 1 m s^{-1}) the change in oxygen profile within the chute due to air ingress was reasonably consistent. A regression analysis of the air ingress versus depth for the 10" nominal bore chute gave a power law dependence of the form:

$$T = k\, S^{1.9} \qquad (5)$$

where T is the time taken for the concentration to reach a particular oxygen concentration, k is a constant, and S is the distance from the end. The variation of oxygen content under conditions of no nitrogen flow versus time is shown in Figure 5.

ADDITION OF SOLIDS WHILST NITROGEN IS FL OWING

The effect of solids addition was investigated on the previously determined steady state concentration profile. When the solids were added, it was found that oxygen was carried to the base of the chute, but the nitrogen flow quickly removed the oxygen, and the conditions at the base of the chute quickly returned to their original state. The flow rate used for Equation (3) was used for the experiments, and the results indicate that the measured oxygen concentration at the base of the chute rose, and then decayed exponentially. The frequency of addition was selected on the basis of a minimum practical addition rate that could be achieved repeatedly, and was selected as one bag per minute. In comparison, the theoretical time required for a plug-flow purge to return the oxygen level to the starting conditions would be:

$$t = \frac{V}{Q} = \frac{0.196}{10} = 0.0196 \text{ hrs} = 71 \text{ sec} \qquad (6)$$

The graph in Figure 6 shows that the oxygen level at the base of the chute rose by about 1% for each bag addition, but that adding at a rate of one bag per minute did not allow adequate time for the nitrogen to remove all the oxygen brought in with the powder and return to the equilibrium oxygen content. After the last addition, it appears that the oxygen level did not return to the base level even after a prolonged period, suggesting that merely purging the chute, may not adequately remove oxygen which had been carried through to the drum beneath, and this was diffusing back into the chute.

ADDITION OF SOLID MATERIALS WITHOUT NITROGEN FLOW

When the solid material is tipped down the chute, the material disturbs the oxygen profile within the chute. Overall, the total quantity of oxygen, including that originally in the chute and that added with the powder, is constant, but the profile is disturbed. The low oxygen concentration at the bottom and high concentration at the top tend to equate when the solids are added, tending to a more uniform concentration throughout the whole chute. This is shown in Figure 7. An alternative way of showing this effect is to plot the oxygen profile within the chute at a number of stages: during purging; after air has diffused into the chute; and immediately after the addition of the solids. An example of this, for the addition of salt, is shown in Figure 8.

The average oxygen concentration in the chute (calculated as the total volume of oxygen as a percentage of the total chute volume) increased with time, once the purge had stopped.

It was found that the changes in oxygen profile in the chute depended upon the type of solid tipped as well as the quantity of solid. The tipping of salt, a free flowing granular solid, gave reproducible changes in oxygen profile between experiments. The peas also gave reproducible changes in profile, though there was some variation in oxygen level, thought to be due to a poorer mixing effect.

The wood shavings did not give very reproducible changes in oxygen profile and this appeared to be linked to the way in which the material flowed down the chute. When the material was poured down the chute in a continuous stream, the oxygen concentration at the bottom of the chute only increased to 2% v/v compared to 4% v/v when the shavings fell in plug flow.

DISCUSSION

PURGING OUT THE AIR

The purging of the chute tends more to plug flow than perfect back-mixing, which is to be expected. Typically, the total purge time is about 1.3 times that taken for perfect plug flow, showing that some axial mixing is taking place. Under plug flow conditions, some axial mixing would be expected, but for such a short length to diameter ratio it would be unlikely to be accurately predictable using the method Taylor[5] because of the vortices at the open end. Thus assuming perfect backmixing occurs would ensure that the purge time was always adequate to remove oxygen, but would incur a time penalty. However, from the practical result, it can be concluded that using the perfect back-mixing model is too conservative, and a practical time of about twice the theoretical plug-flow purge time would be acceptable in practice.

OXYGEN PROFILE UNDER STEADY STATE WITH A NITROGEN FLOW

The steady state work determined the oxygen profile down the chute as being a logarithmic function of the distance from the open end. This dependency is in broad agreement with Husa's later work, which also found a logarithmic dependency. There was a large and rapid variation of oxygen concentration within the top metre of the chute, which is effectively noise associated with the cross-wind, illustrated in Figure 4. This is discussed in greater detail by Bryce and Fryer-Taylor, who show vortices induced by the cross-wind to be the cause. Further down the chute, the variation was smaller, and less "noisy", showing that the progress of air down the chute was more diffusion controlled rather than controlled by the mixing at the open end. Thus Husa's later work appears not to take account of the potential for pockets of oxygen-rich gas to occur further down the chute than would appear from assuming that the

oxygen profile is uniform. However, at long distances (ie greater than about 15 diameters), the effect of vortices at the open end appears to have a minimal effect.

The purge flow-rate used to generate Figure 2 was substituted into Husa's later equation. It was found that Husa's later equation for windy conditions, gave reasonably similar results to the actual data from calm conditions. This is shown in Figure 3. However, the equilibrium concentration at the bottom of the chute at this purge rate can be predicted with reasonable accuracy from Husa's later work.

DIFFUSION OF AIR INTO A NITROGEN-FILLED CHUTE WITH NO NITROGEN FLOW

As would be expected, air slowly diffused into the chute once the nitrogen purge had been stopped. The steady state concentration can be considered to be a continuous purge at a rate exactly equal to the rate of inward diffusion of the oxygen. Since the steady state concentration profile is logarithmic, it would be understandable to expect that the ingress of oxygen were the reverse, ie exponential, but in practice the data acquired suggests a power relationship with an exponent of about 1.9. This is shown in Figure 5. The coefficient of fit for the regression analysis is not particularly good, but there is such limited data that no firm conclusion as to the general behaviour of the system could be determined. Tite[6] et al have presented data for air ingress into pipes pre-purged with natural gas, and their data indicates a similar power law relationship for rate of air ingress. However, no further comparisons could be drawn as their arrangement was too dissimilar. Further work is in hand to investigate this on smaller pipe sizes to attempt to obtain a generic solution to the inward diffusion of air into inerted pipes.

ADDITION OF SOLIDS WHILST NITROGEN IS FL OWING

The addition of the solids to the chute disturbs the steady state oxygen concentration profile, such that the oxygen concentration at the top of the chute momentarily decreases, whereas the concentration at the bottom increases. This increase is larger than can be accounted for by the oxygen within the interstices of the powder. This was not removed adequately even after a prolonged purge, indicating that oxygen was being carried down the chute and into the drum at the base. Thus there is a steady, but small accumulation at the base of the chute as further bags are added. This would slowly mix with the gas at the base of the chute, indicating a low permanent concentration.

The continuous purging of the vessel ullage space can present practical problems where the vessel is at a higher temperature than the chute, as vapours leaving with the gas purge can condense within the chute, causing the powder to adhere to the walls of the chute. This can be avoided by injecting the purge at the base of the chute instead. However, this work has shown that when injecting the purge at the base of the chute, a small quantity of oxygen would be carried through to the vessel with each aliquot of powder. This oxygen may accumulate to a hazardous level for large powder additions to a vessel with a small ullage space.

The time required to purge out any oxygen in the period between addition of the bags is difficult to determine. This is because the effect of the volume of the drum at the base of the chute was not investigated in this work. However, the use of the theoretical perfect back-mixing calculation in Equation (2) for a reduction to 5% oxygen from 21% after the addition of each aliquot of powder would appear to provide a suitable time for ensuring adequate purging.

ADDITION OF SOLID MATERIALS WITHOUT NITROGEN FLOW

The intention behind this addition method was to minimise the hygiene problems associated with the use of a continuous flow of gas up the chute which tends to form external dust clouds of finely divided material. Adding the solids to the chute without the nitrogen flow had a similar, but more pronounced, effect on the oxygen concentration profile as when the nitrogen purge was flowing continuously. Once the chute had been inerted to a steady oxygen profile, the cessation of purging results in a slow continuous ingress of air. This effectively increases the total oxygen within the chute, so that when solids were added after a delay from stopping the nitrogen flow, the oxygen concentration at the base of the chute was higher than it would be if the powder were added immediately after stopping the purge. This because the addition of solids after a delay serves to transfer more oxygen to the base of the chute and the drum beneath. Since the concentration of oxygen at the top of the chute is much higher, the concentration at the base of the chute after being mixed by the solids also becomes higher. This was unexpected as it was originally though that the oxygen in the solids would be purged out as it fell through the stationary gas in the chute. The use of such a discontinuous purge (ie stopping the purge during the addition of powder and re-starting afterwards) may not be adequately safe, as it would be prone to operator error. This is particularly hazardous where the purge has been stopped and there is an excessive delay before the powder addition is made.

A comparison was made of the effect of powder characteristics on disturbing the oxygen concentration profile, by using two materials other than salt. The characteristics of the powders show that for the finer powder (ie salt), the mixing effect is greater. This is not unexpected, as the interfacial surface area is far greater for small particles than the same total volume of large particles. The effect of plug flow with the wood shavings suggests that more oxygen reaches the bottom of the chute under plug flow than under poured flow. This was considered to be caused by the large quantity of air already within the top of the chute being pushed down the chute and mixed in with the nitrogen at the base of the chute. It was not possible to confirm the plug flow carrying air down with the salt or dried peas, as the design of the equipment did not permit a large enough quantity of solid to be used in a single addition. Also, both the peas and the salt were extremely free-flowing. The use of other materials was considered, but many were rejected on toxicity or flammability grounds, and lack of time prevented a wider experimental programme being pursued.

CONCLUSIONS

1. The information provided by Husa's later paper for the purging requirements of stacks under windy conditions has been found to be adequate to determine the minimum purge rate for short charge chutes under calm conditions.
2. The nitrogen flow purges oxygen from the chute in a manner which is between that for plug flow and that for perfect back-mixing.
3. Assuming perfect back-mixing will ensure safe purging times but in practice, where few additions are made, the time between additions can be significantly reduced.
4. Air ingress to a pre-inerted chute with no continuous purge appears to follow a power law, but further work is required to confirm this.
5. Under steady flow conditions, an oxygen concentration profile forms within the chute, and this is disturbed by the addition of powders.
6. The physical form of the powder has an effect on the disturbance of the oxygen profile, but insufficient data is available to be able to predict it.

7. Where plug flow occurs, the oxygen passed to the vessel can double, and an appropriate factor should be applied to the purge regime to compensate.
8. The use of a purge fed to the base of a chute rather than to a vessel ullage space, can allow some oxygen to accumulate within the vessel.
9. Where the purge is added to the vessel, the flow and time required between aliquots of powder can be calculated from the basic perfect backmixing calculation in Equation (2), or from plug flow providing a suitable safety factor is used.

ACKNOWLEDGEMENTS

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Figure 1

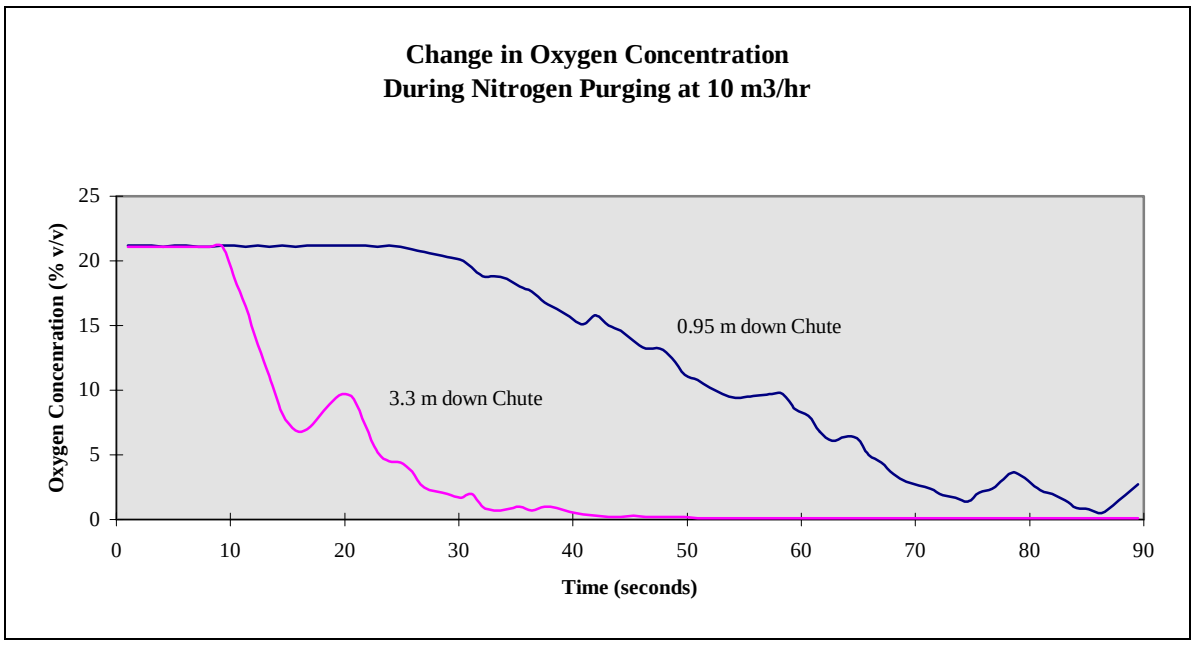

Figure 2

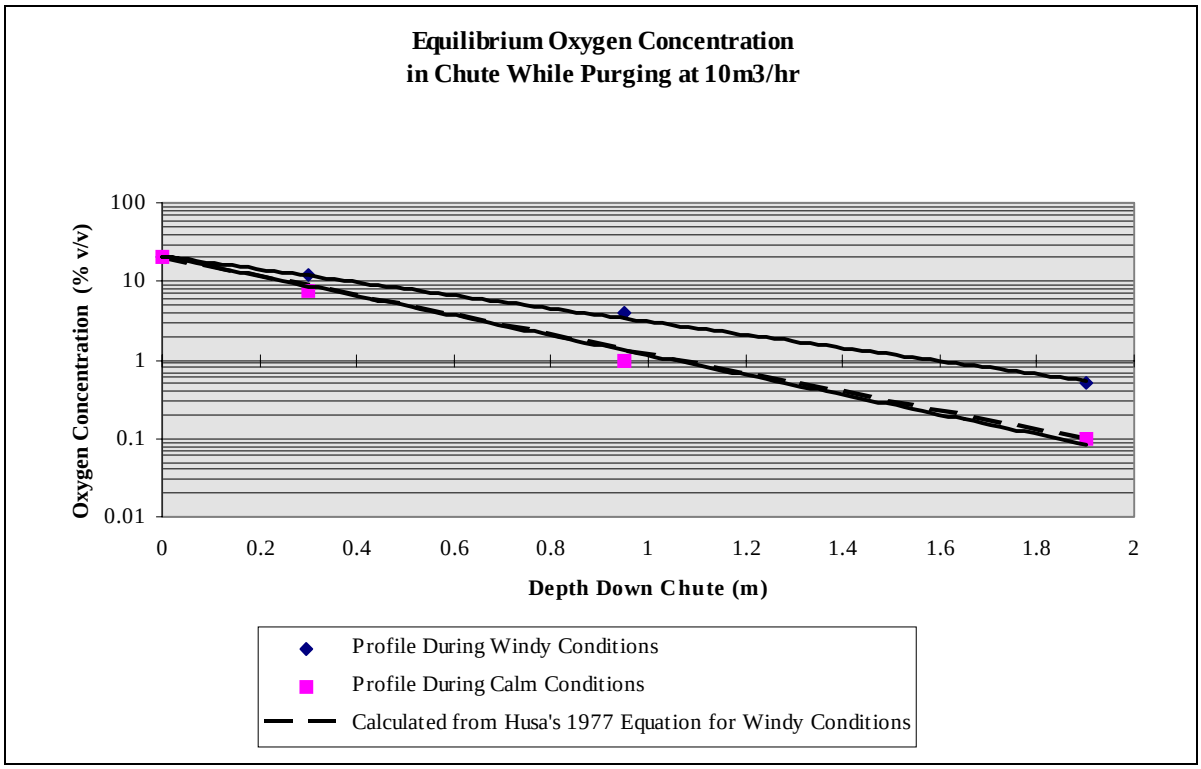

Figure 3

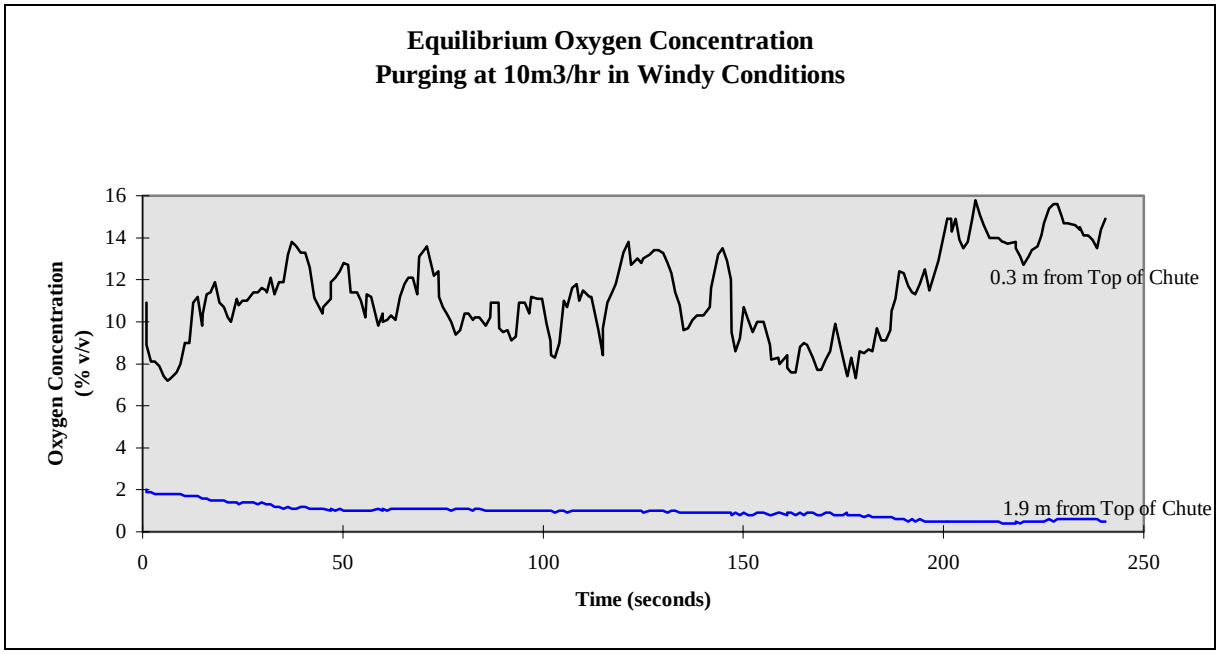

Figure 4

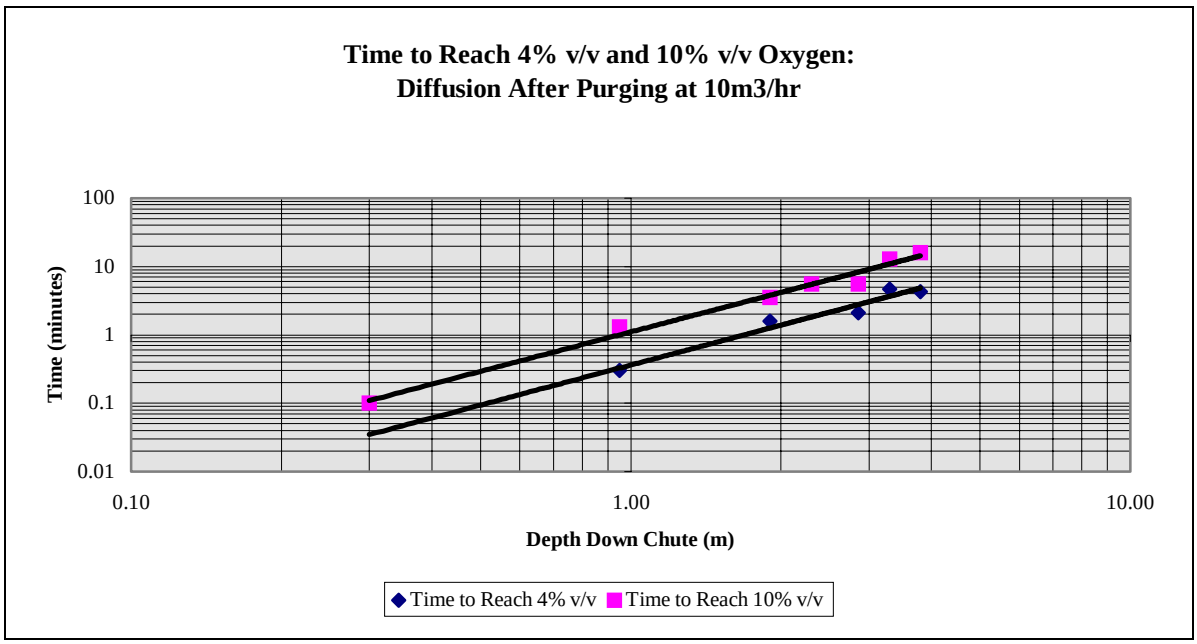

Figure 5

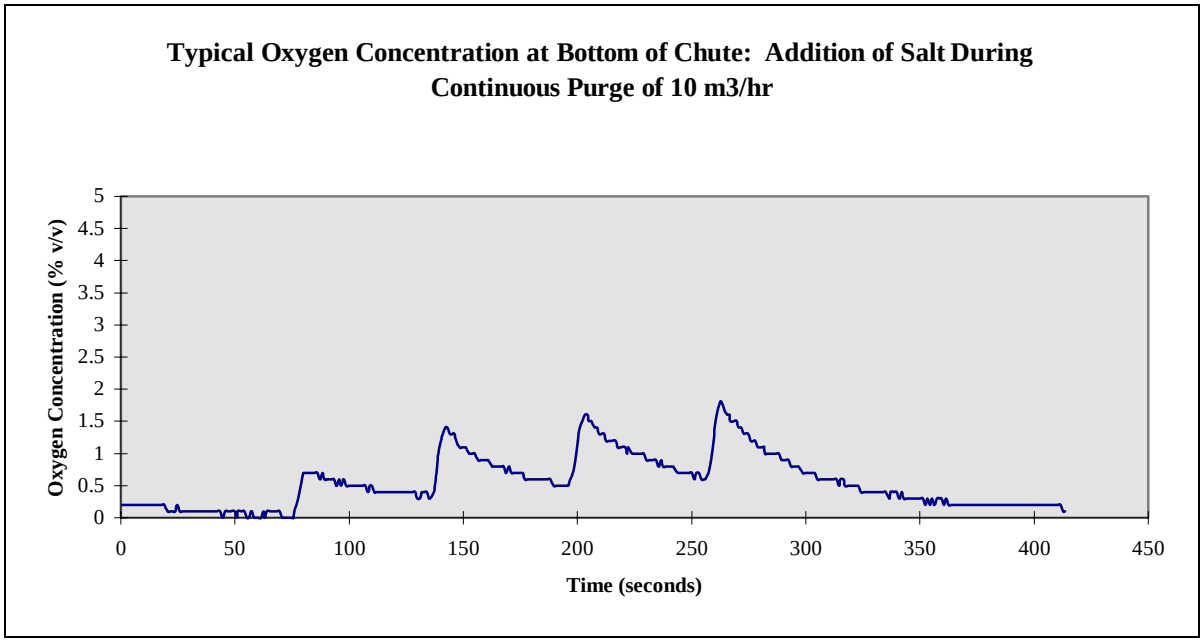

Figure 6

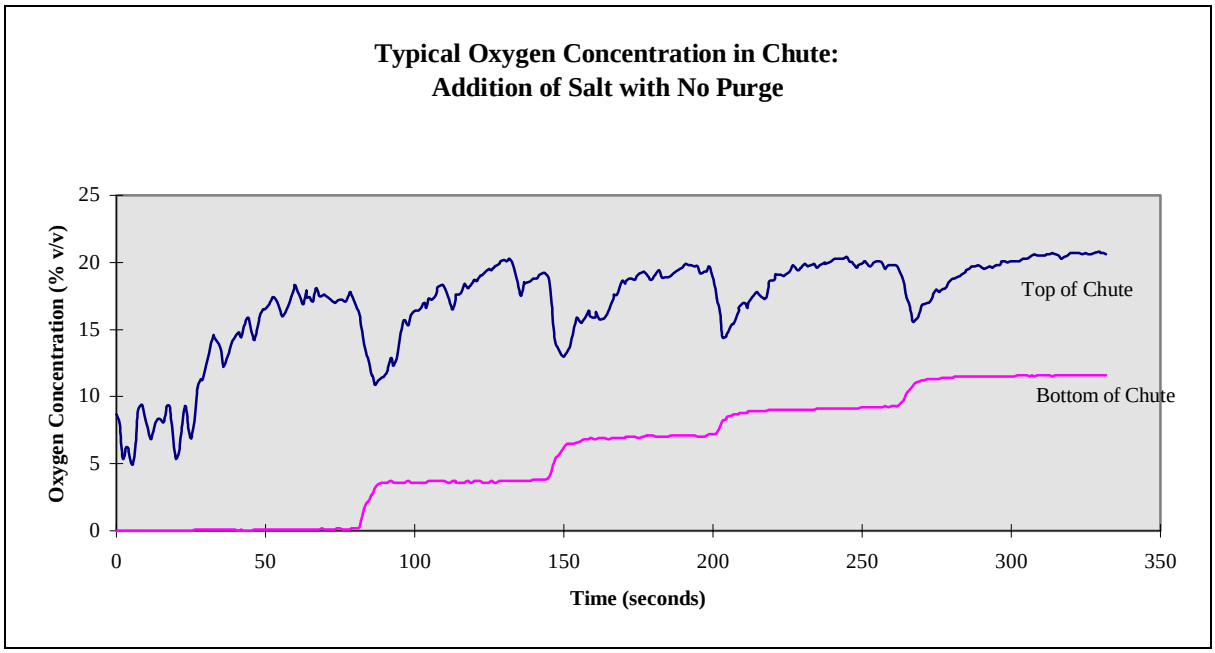

Figure 7

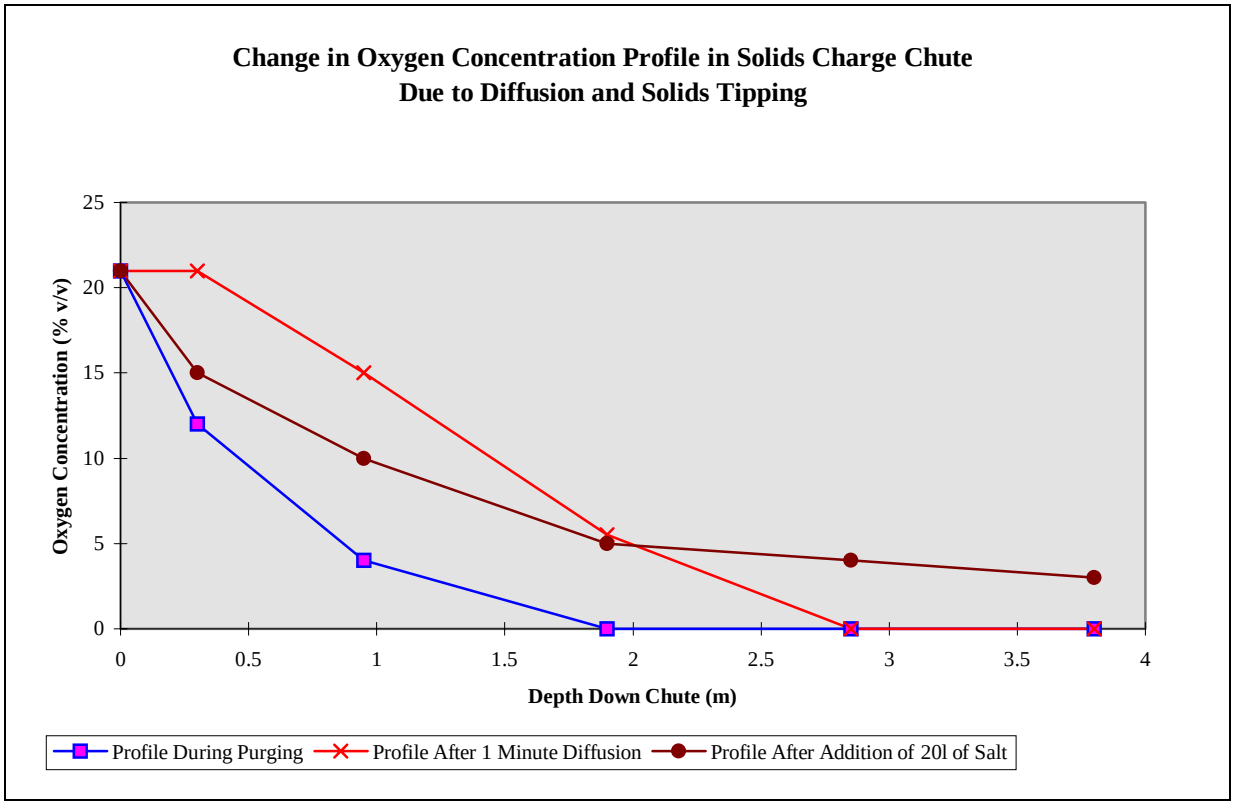
Change in Oxygen Concentration Profile in Solids Charge Chute
Due to Diffusion and Solids Tipping
Oxygen Concentration (% v/v)
25
20
15
10
5
0
0
0.5
1
1.5
2
2.5
3
3.5
4
Depth Down Chute (m)
Profile During Purging
Profile After 1 Minute Diffusion
Profile After Addition of 20l of Salt

Figure 8