Catalytic Oxychlorination Process Optimization for the Conversion of Ethylene to Ethylene Dichloride

Rehab M. El-Maghraby, Alaa El-Din A. Youssef, Abeer M. Shoaiab

Abstract: The catalytic oxychlorination of ethylene is widely used for the production of ethylene dichloride (EDC) which is an essential step for the production of poly-vinyl chloride (PVC). However, catalyst entrainment from the fluidization reactor has formed a problem and caused the loss of expensive catalyst powder with the outlet gas, hence less conversion in achieved. In this research we studied the catalytic oxychlorination of ethylene over the surface of gendart A powder with average particle size of 80 microns. Simulation was developed using Aspen plus and Mat Lab. It was validated against the industrial data and they were matching.

Various operating conditions and variables were studied. The effect of fluidized gas velocity, bubble diameter, temperature and pressure on solid catalyst entrainment rate. In addition, the copper content from the quencher bottom was analyzed. It was found that the entrainment rate increased with the increase in fluidized gas velocity, bubble diameter, reactor temperature and pressure. While on the other hand increasing orifice diameter decreased the amount of entrained catalyst within the fluidized bed.

It was found that the optimum superficial gas velocity is 2570.4 m³/hr and the optimum orifice diameter is 1 cm. At this condition the lowest rate of solids entrainment out of the bed will be reached. This will decrease the amount of lost catalyst and the process will be more economic with higher gas conversion.

Keywords: Oxychlorination Process, Ethylene Dichloride, Optimization, Fluidization, Catalyst Entrainment, Orifice Diameter.

I. INTRODUCTION

Poly vinyl chloride (PVC) is widely used synthetic plastic around the world [1] due to its chemical resistance, fire retardant and mechanical strength properties. PVC is mainly produced from vinyl chloride (VC) monomer, by thermal cracking of ethylene dichloride. Ethylene Dichloride (EDC) is mainly produced by two methods [2], either by the direct chlorination of ethylene with chlorine in the presence of FeCl₃ as a catalyst; or the oxychlorination of ethylene in the presence of CuCl₂ supported on alumina; which is our main focus in this work.

The oxychlorination process was discovered in the 1930s and was first commercialized in the late 1960s by the Pittsburg Plate Glass industries [3]. The aim of the oxychlorination process is to reuse the HCl formed during the thermal cracking (pyrolysis) of EDC, to further produce more EDC.

In the industrial oxychlorination reactor, gaseous ethylene, hydrogen chloride and oxygen react in the presence of cuprous chloride catalyst at temperature of 208 to 215 °C, and pressure of 2.8 to 3.2 bar, according to the reactor loading. The oxychlorination process of ethylene is performed in a fluidized bed reactor.

One of the main problems faced in the VC monomer production plant is the entrainment of the catalyst powder out of the fluidized bed reactor that results in catalyst losses by time. Hence, the operating cost increases and catalyst charge also increases in order to process the full load of feed streams. In addition to problems in handling HCl due to low conversion and corrosion problems in pipelines, static and rotating equipment, problem in operating higher flow rates of HCl. Moreover, the blockage of the bottom quencher as the powder forms a complex with the sodium carbonates and sodium bicarbonates, and repeating shutdowns of the plant to solve each of the above stated problems, hence, the VC monomer production is decreased and the target production of PVC is not achieved.

A model and simulation were developed by Morira and Pires [4] to study the effect of fluidized bed height, bubble diameter, residence time, cupric chloride weight in the catalyst and the emulsion phase temperature on the conversion of ethylene.

Some studies were conducted on the particles size of the fluidization catalyst [8-10]. Hatzantonis et. al. [5] predicted the particle size distribution in catalysed olefin polymerization fluidized bed reactor by using a model. Results showed exponential increase between the rate of particle agglomeration and the bed temperature. Also, elutriation rate is affected by the agglomeration rate which largely reduces the number of the small particle size. On the other hand, Grace and Sun [6] showed that catalyst particles (geldart A) should be prepared with a broad range of sizes because mono dispersed particles give poorer performance. The most effective particles are those which spend greater time in the dilute phase. Geldart [7] conducted experimental work on the effect of particle size and particle size distribution in a fluidized bed reactor. The experiments showed that the behaviour of many powders is independent of particle size and particle size distribution, and that mean bubble diameter is dependent only on the distributor, the distance above the distributor and the excess velocity.

While, Paiva et. al. [8] studied the influence of the distributor on the bottom zone of the fluidized bed approaching the transition from bubbling to fluidization. Results showed that the flow changes with the change in operating conditions.

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It is clear from the literature that no studies tested or investigated the effect of fluidized bed operating condition on the catalyst entrainment. In this work we will study the entrainment of solids (Geldart A) in a fluidized bed reactor and how the variation of the supercritical gas velocity, distributor orifice diameter, bed temperature and entering pressure of gas will affect entrainment. In addition, we will study the effect of varying the supercritical gas velocity on bubble diameter. The amount of copper content as an indication of solid entrainment using laboratory analysed samples will also be studied.

II. METHODOLOGY

In the present work we will focus on the oxychlorination reactor of ethylene at the Egyptian petrochemicals company, as shown in Figure 1.

![Image of oxychlorination process layout](image)

**Fig 1. Oxychlorination process layout for the production of ethylene dichloride**

The main equipment in the oxychlorination unit is the oxychlorination reactor which is a fluidized bed reactor containing a bed of catalyst, while the fluidization is kept by the recycle gas compressor discharge flow.

The feed streams are; HCl from the VC monomer purification unit, ethylene from Ethydco or SIDPEC company, and Oxygen from the air separation unit, or the pressure swing adsorption unit. These feed streams enter the reactor through a distributer and sparger, the feed gases continue through the bed of catalyst and the reaction occurs over three steps [9]. The overall oxychlorination reaction of ethylene [9] is shown in Eq. 1:

\[
C_2H_4(g) + 2 \text{HCl} (g) + \frac{1}{2}O_2(g) \rightarrow C_2H_4Cl_2(g) + \text{H}_2O (g), \quad \Delta H = -198 \frac{kJ}{mol} \quad (1)
\]

The oxychlorination reaction is exothermic. The reaction temperature is controlled by the coolant tubes to stop overheating and get the desired products. The catalyst powder in the size range of 40 – 125 µm is separated from the product gas stream by three stage cyclones.

A. Aspen Plus Simulation

Aspen plus Version 8.8 was used to simulate the fluidization process of the oxychlorination of ethylene to produce ethylene dichloride. In the process flow sheet, shown in Figure 2, the recycle gas is feed to the fluidized bed reactor at a flow rate of 2650 m³/hr. The height of catalyst in the reactor is 11 meter and its weight is 30 ton. Reaction was conducted at pressure of 3.2 bar and temperature of 215 °C. The produced gases pass through a cyclone to collect the solids entrained out of the bed the size range of 40 – 125 µm.

![Image of Aspen plus flow sheet](image)

**Fig 2. Aspen plus flow sheet of the fluidized bed reactor for the oxychlorination of ethylene**

Fluidization will begin at the gas velocity where the weight of solids is equal to the drag force exerted to lift the solid catalyst up. This is the minimum fluidization velocity, \( U_{mf} \) and is calculated using Eq. 2.

\[
U_{mf} = \frac{\phi (dp) ^2}{150 \mu} = \left( g (\rho_p - \rho_g) \right) \frac{\varepsilon_{mf}}{1 - \varepsilon_{mf}} \quad (2)
\]

The voids fraction or porosity at minimum fluidization, \( \varepsilon_{mf} \) can be calculated using Eq. 3, and \( \eta \) is dimensionless.

\[
\varepsilon_{mf} = 0.586 \phi^{-0.72} \left( \frac{\mu}{\rho_g \sigma_d p} \right)^{0.029} \eta^{0.021} \quad (3)
\]

The superficial gas velocity, \( U_o \) is calculated using Eq. 4:

\[
U_o = \frac{\left( \frac{U}{\varepsilon} \right)}{4} \quad (4)
\]

According to Davidson and Harrison correlation [11], bubble rise velocity, \( U_b \) was calculated according to Eq. 5.
\[ U_b = U_o - U_{mf} + (0.71)(g d_b)^{1/2} \] (5)

The relation between the effective bubble diameter, \( d_b \) and the height of the bed at fluidization conditions, \( h \) is shown in Eq. 6. This equation is considered in the calculations of the fluidized bed reactor parameters.

\[ \frac{d_{bm} - d_b}{d_{bm} - d_{s0}} = e^{-0.33 h} \sqrt{\frac{1}{U}} \] (6)

The volume fraction of bubbles, \( \delta \) representing the fraction of the total bed occupied by bubbles, Eq. 7. This does not include the wake (the region just below the bubble).

\[ \delta = \left( \frac{U_o - U_{mf}}{U_o - U_{mf}(1+\alpha)} \right) \] (7)

The weight of solids hold up, \( W_s \) in gm is represented by Eq. 8.

\[ W_s = A_c h \rho_p (1 - \delta)(1 - \epsilon_{mf}) \] (8)

**C. Solid entrainment in fluidized bed**

The entrainment in the fluidized bed [12] is investigated using a series of equations as indicated below. At the surface of the bed, the bubbles pressure is higher than bed surface pressure. Hence, the bubbles will burst when reaching the surface. This will cause the spraying of solids from the bubbles into the free board, is the zone above the fluidized bed. So, the solids eject out of the fluidized bed. The height above which the entrainment does not change appreciably, but still there are entrainment of solids out of the bed, is the transport disengaging height, TDH, and is calculated from the Froude number according to Eq. 9.

\[ \text{Froude number} ; \quad \frac{u_o^2}{g TDH} = 10^{-3} \] (9)

**III. RESULTS AND DISCUSSION**

Our study was developed using calculations using Mat-lab and Aspen Plus simulation. In addition, gas samples from the fluidized bed were analyzed in the laboratory for copper content. Different parameters were studied;

**A. Optimum gas flow rate and superficial gas velocity**

Results developed using Mat-lab are shown in Table 1 at pressure of 4.6 bar, temperature of 155 °C and recycle gas discharge flow of 2650 m³/hr. According to Wen and Chin Yung calculations [12] the value of terminal velocity, \( U_t \) is 23.05 cm/s. In addition, superficial gas velocity, \( U_s \) must not exceed the terminal velocity, \( U_t \). Hence, as shown in Table 1, the range of gas flow rate that will fulfill this superficial gas constrain is within the range of 710,000 to 720,000 cm³/s. Based on this a more detailed iteration was conducted using gas flow rate in the specified range and the corresponding superficial gas velocity was calculated, see Table 2. This allowed the identification of the optimum gas flow rate, \( Q \) which is 714,000 cm³/s or 2570.4 m³/hr, this corresponds to a superficial gas velocity, \( U_o \) of 23.02 cm/s.

\[ \frac{10^3 u_0^2}{g \rho_s} = \text{Const. in} \left( \frac{m^3}{kg} \right) \] (10)

\[ \frac{\epsilon_s}{U_o \rho_g} = \text{Const.} \] (11)

\[ \frac{(U_o-U_t)^2}{g \rho_g} = \text{Const.} \] (12)

Table 1. Mat lab used to study the effect of gas flow rate variation on fluidized bed parameters.

<table>
<thead>
<tr>
<th>( Q ) (cm³/s)</th>
<th>700,000</th>
<th>705,000</th>
<th>710,000</th>
<th>715,000</th>
<th>720,000</th>
<th>725,000</th>
<th>740,000</th>
<th>745,000</th>
<th>750,000</th>
<th>755,000</th>
</tr>
</thead>
<tbody>
<tr>
<td>( D_b ) (cm)</td>
<td>114.89</td>
<td>115.23</td>
<td>115.56</td>
<td>115.89</td>
<td>116.22</td>
<td>116.86</td>
<td>117.5</td>
<td>118.15</td>
<td>118.8</td>
<td>119.45</td>
</tr>
<tr>
<td>( \epsilon_{mf} )</td>
<td>0.5049</td>
<td>0.5049</td>
<td>0.5049</td>
<td>0.5049</td>
<td>0.5049</td>
<td>0.5049</td>
<td>0.5049</td>
<td>0.5049</td>
<td>0.5049</td>
<td>0.5049</td>
</tr>
<tr>
<td>( U_o ) (cm/s)</td>
<td>260.58</td>
<td>261.09</td>
<td>261.6</td>
<td>262.1</td>
<td>262.6</td>
<td>262</td>
<td>261.6</td>
<td>263.6</td>
<td>265.6</td>
<td>266.6</td>
</tr>
<tr>
<td>( \delta )</td>
<td>0.086</td>
<td>0.0867</td>
<td>0.0868</td>
<td>0.0869</td>
<td>0.087</td>
<td>0.087</td>
<td>0.0868</td>
<td>0.087</td>
<td>0.090</td>
<td>0.0907</td>
</tr>
<tr>
<td>( U_s ) (cm/s)</td>
<td>22.57</td>
<td>22.73</td>
<td>22.89</td>
<td>23.06</td>
<td>23.21</td>
<td>23.53</td>
<td>23.86</td>
<td>24.02</td>
<td>24.18</td>
<td>24.26</td>
</tr>
</tbody>
</table>

Table 2. Relation between gas flow rate and superficial gas velocity.

<table>
<thead>
<tr>
<th>( Q ) (cm³/s)</th>
<th>710,000</th>
<th>711,500</th>
<th>712,000</th>
<th>712,500</th>
<th>714,000</th>
<th>715,000</th>
<th>716,000</th>
<th>717,000</th>
<th>718,000</th>
<th>719,000</th>
<th>720,000</th>
</tr>
</thead>
<tbody>
<tr>
<td>( U_s ) (cm/s)</td>
<td>22.89</td>
<td>22.9</td>
<td>22.96</td>
<td>22.99</td>
<td>23.02</td>
<td>23.06</td>
<td>23.09</td>
<td>23.1</td>
<td>23.15</td>
<td>23.18</td>
<td>23.21</td>
</tr>
</tbody>
</table>
B. Effect of superficial gas velocity on bubble diameter

From Table 1 and Figure 3, by using Mat-lab it is clear that the bubble diameter increases with the increase in the gas velocity. As the bubble diameter increases, the mass transfer between the bubble phase and the emulsion phase increases which favor the transportation of bigger quantities of unreacted gases through the bed. According to the constrain on the superficial gas velocity as illustrated in the previous section, to keep an optimum superficial gas velocity of 23.02 cm/s or less, the bubble diameter must be less than 115.89 cm, see Table 2 and Figure 3. At such value an optimum conversion of the gases will be achieved with a terminal and superficial velocity value of less than 23.05.

![Fig 3. Relation between the bubble diameter inside the fluidized bed and the superficial gas velocity of the gas entering the reactor.](image)

C. Effect of changing distributor orifice diameter on the entrained solids

The effect of increasing the distributor orifice diameter on the mass of entrained solids was studied using Aspen Plus at a recycle gas discharge flow of 6800 Nm$^3$/hr at normal conditions of temperature and pressure of 25 °C and 1 atmosphere respectively. It was found that by increasing the orifice diameter, $D_o$, the mass of entrained solids out of the bed, M will decrease rapidly up to an orifice diameter of 10 mm where the amount of entrained solid reaches 8.9 kg. Any further increase of the orifice diameter above 1 cm. will affect the mass of entrained solids to a lesser extent, see Table 3 and Figure 4. Hence, it is clear that an optimum distributor orifice diameter of 1 cm is recommended to reach lowest possible mass of entrained solids out of the fluidized bed, while the current orifice diameter in the reactor is 0.7cm. This mean that using a different orifice diameter than the current one is expected to improve the performance of the unit under study.

In addition, according to Table 3, by increasing the orifice diameter of the distributor, we found that both bubble diameter and pressure drop through the orifice decrease.

![Fig 4. Relation between orifice diameter of the distributor versus entrained solids out of fluidized bed. Fluidized bed.](image)

D. Effect of superficial gas velocity on solid entrainment

According to the calculations listed in Table 4 and 5 based on Eq. 10 and 11 and by using the curve of Geldart A [13], the value of the entrained catalyst particles, $G_s*$ was determined.

The mass of solids in the product gas (entrained solid) according to the simulation developed using Aspen Plus is shown in Figure 5 against the change in the amount of superficial gas velocity at an optimum orifice diameter of 1 cm. It is clear that as the superficial gas velocity increases the entrained solids out of the bed increase, see Figure 5. Increasing the gas velocity means lower amount of catalyst in the fluidized bed and lower conversion of gases. But at an optimum superficial velocity of 23.05 cm/s, the entrained amount of solids, $G_s*$ will be less than 8.045 kg/m$^2$.s.

![Fig 5. Relation between superficial gas velocity and mass of entrained solids out of](image)

### Table 3. Distributor orifice diameter versus mass of entrained solids using Aspen Plus.

<table>
<thead>
<tr>
<th>$D_o$, mm</th>
<th>7</th>
<th>8</th>
<th>9</th>
<th>10</th>
<th>11</th>
<th>12</th>
<th>13</th>
</tr>
</thead>
<tbody>
<tr>
<td>$D_{b,max}$, cm</td>
<td>136</td>
<td>136</td>
<td>124</td>
<td>115</td>
<td>110</td>
<td>107</td>
<td>105</td>
</tr>
<tr>
<td>$\Delta p_{o}$, bar</td>
<td>1.135</td>
<td>0.666</td>
<td>0.415</td>
<td>0.272</td>
<td>0.18</td>
<td>0.131</td>
<td>0.095</td>
</tr>
<tr>
<td>M, kg/hr</td>
<td>15.07</td>
<td>11.26</td>
<td>9.716</td>
<td>8.939</td>
<td>8.503</td>
<td>8.239</td>
<td>8.07</td>
</tr>
<tr>
<td>TDH, 3.93</td>
<td>3.94</td>
<td>3.94</td>
<td>3.94</td>
<td>3.94</td>
<td>3.94</td>
<td>3.94</td>
<td>3.94</td>
</tr>
</tbody>
</table>
Table 4. Relation between gas flow rate, Q and transport disengaging height, TDS.

<table>
<thead>
<tr>
<th>Q (cm³/s)</th>
<th>700,000</th>
<th>705,000</th>
<th>710,000</th>
<th>715,000</th>
<th>720,000</th>
<th>730,000</th>
<th>740,000</th>
<th>745,000</th>
<th>750,000</th>
<th>755,000</th>
</tr>
</thead>
<tbody>
<tr>
<td>U₀ (cm/s)</td>
<td>22.57</td>
<td>22.73</td>
<td>22.89</td>
<td>23.06</td>
<td>23.21</td>
<td>23.535</td>
<td>23.86</td>
<td>24.02</td>
<td>24.18</td>
<td>24.26</td>
</tr>
<tr>
<td>Const.</td>
<td>45.9002</td>
<td>46.56</td>
<td>47.2210</td>
<td>47.89</td>
<td>48.5606</td>
<td>49.9188</td>
<td>51.2958</td>
<td>51.99</td>
<td>52.6916</td>
<td>53.66</td>
</tr>
<tr>
<td>TDS</td>
<td>519.9945</td>
<td>527.44</td>
<td>534.9576</td>
<td>542.5</td>
<td>550.1330</td>
<td>565.5206</td>
<td>581.1204</td>
<td>589.01</td>
<td>596.9325</td>
<td>604.82</td>
</tr>
</tbody>
</table>

Table 5. Entrainment flux, Gₛ* determined from Zens et. al. plot [13].

<table>
<thead>
<tr>
<th>Gₛ*, g/cm².s</th>
<th>5.5</th>
<th>5.75</th>
<th>6</th>
<th>6.75</th>
<th>6.5</th>
<th>7</th>
<th>7.5</th>
<th>7.75</th>
<th>8</th>
<th>8.5</th>
</tr>
</thead>
<tbody>
<tr>
<td>Gₛ*, kg/m².s</td>
<td>0.6811</td>
<td>0.712</td>
<td>0.743</td>
<td>0.7739</td>
<td>0.8049</td>
<td>0.8668</td>
<td>0.9288</td>
<td>0.9597</td>
<td>0.9907</td>
<td>1.0317</td>
</tr>
</tbody>
</table>

E. Effect of changing bed temperature on the entrained solids out of the bed

The relation between the mass of entrained solids and operating temperature is linear, see Figure 6. It was found that as the temperature increases, the entrained solids out of the bed increase.

![Fig 6. Relation between bed temperature and mass of entrained solids.](image)

F. Effect of changing entering gas pressure on the entrained solids

According to Figure 7 developed using data from Aspen Plus, the mass of entrained solids increases with the increase in the entering pressure. This may be directly related to the increasing density of the gas and thus a decrease in the terminal velocity which allows the catalyst entrainment to be elevated.

![Fig 7. Relation between pressure at the entrance zone of the bed and mass of entrained solids using aspen plus.](image)

G. Effect of the entering gas velocity on the copper content in the quencher bottom

The concentration of copper in the quencher bottom (the downstream of the reactor) is a direct indication of the entrained catalyst particles. From the laboratory analysis as shown in Figure 8, by observing the sample analysis of the quencher bottom for about 6 months and recording the recycle gas velocity. It was found that at a velocity of 6800 Nm³/h, the lowest concentration of copper in the sample is observed, which is a direct indication of the catalyst presence. When this quantity of entrained catalyst is as low as possible the chance of forming a complex with the carbonate and bicarbonate salts is kept to minimum and the problem of the blocking of quencher bottom flow is hindered.

![Fig 8. Relation between copper content concentration at the quencher bottom and the entering gas velocity.](image)

IV. CONCLUSION

In this study the oxychlorination process for the production of ethylene dichloride was simulated using Aspen Plus and equation was solved using Mat-lab. Copper content in the exiting gas was analyzed in the laboratory. It was found that:

- The entrainment of the catalyst particles affects the performance of the fluidized bed.
- Increasing the fluidization gas flow rate increases the quantity of solids entrained out of the bed and decreases the bubbles diameter in the bed.
Catalytic Oxychlorination Process Optimization for the Conversion of Ethylene to Ethylene Dichloride

- The change in the temperature and pressure of the fluidized bed affects the quantity of entrained solids out of the bed. The amount of entrained catalyst is increased by increasing bed temperature according to the simulation conducted using Aspen Plus.
- It is recommended to operate the reactor at a flow rate of 714,000 cm$^3$/s (2570.4 m$^3$/hr.) at 4.6 bar, 155°C and Recyle gas flow of (6800 Nm$^3$/h) to reduce the entrained solids out of the bed to the lowest possible values.
- It is recommended to resizing the distributor orifice diameter for this flow rate, 2570.4 m$^3$/hr. to be 1cm in order to reduce the entrained solids out of the bed.
- At a velocity of 6800 Nm$^3$/h, the lowest concentration of copper in the sample is observed, hence minimum blockage of quencher can be achieved.

ACKNOWLEDGE

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NOMENCLATURE

- $U_{mf}$: velocity of gas at minimum fluidization, cm/s.
- $\Psi$: sphericity of the catalyst particle.
- $d_p$: average diameter of the particle size, micron.
- $\mu$: gas viscosity, gm/cm.s.
- $g$: gravity acceleration, cm/s$^2$.
- $\rho_p$: density of catalyst particle, gm/cm$^3$.
- $\rho_g$: density of gas, gm/cm$^3$.
- $\varepsilon_{mf}$: porosity at minimum fluidization.
- $\eta$: $g(\rho_p - \rho_g)$
- $U_o$: superficial gas velocity, cm/s.
- $V_o$: Inlet fluidizing gas velocity, cm/s.
- $A_c$: cross sectional area of the bed, cm$^2$.
- $U_b$: rising bubble velocity, cm/s.
- $d_{mb}$: maximum bubble diameter, cm.
- $d_{bo}$: initial bubble diameter, cm.
- $h$: height of the bed, cm.
- $D_t$: diameter of the bed, cm.
- $\delta$: fraction of the bed occupied by the bubbles.
- $\alpha$: the ratio of the volume of wake to the volume of bubble.
- $W_c$: total mass of catalyst in the bed, gm.
- $TDH$: transport disengaging height, cm.
- $Gs^*$: the mass flow rate of entrained catalyst particles.

REFERENCES


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