Abstract

The aim of this work was to study the production of diethyldichlorosilane [$(C_2H_5)_2SiCl_2$] by the catalytic reaction of ethylchloride (C_2H_5Cl) with silicon (Si) (purity 99.9%) and using copprus chloride (CuCl) (purity 99.9%) as catalyst in a fluidized bed reactor. Gas chromatographic technique was used on-line for analysis of the reaction product.

The experimental work was carried out using two columns: the first used for the hydrodynamic study was a QVF of 5*cm* internal diameter and 50*cm* height, the second which has the same dimension was stainless steel used as a fluidized bed reactor.

Gas distributor was fitted at the bottom of column with 25*holes* of 0.1*cm* holes internal diameter.

The work is divided to five sections: hydrodynamic, experimental design, system design, reaction, mathematical models.

The hydrodynamic experiments were concerned mainly with the finding of the minimum fluidization velocity (U_{mf}) for different particle diameters range (139-400)*μm*, these data of U_{mf} was used to check the validity of two empirical equations [(4.27), (4.28)], and the ability of using them to find U_{mf} for any particle diameter , they show good agreement within abs. error less than 5%.

$$U_{mf} = \frac{\mu}{\rho_f d_p}\left\{(1135.7 + 0.0408 Ar)^{1/2} - 33.7\right\} \quad \text{.....(4.27)}$$

$$U_{mf} = \frac{(\rho_p - \rho_g)^{0.934} g^{0.934} d_p^{1.8}}{1.111 \mu^{0.8} \rho_g^{0.07}} \quad \text{.....(4.28)}$$

Other parameters such as; pressure drop across, the bed and distributor, also the height of bed for various gas velocities, were predicted from the hydrodynamic study.

On the other hand porosity of the bed was predicted from the height of the bed Eq.(4.14), which is important variable in calculating theoretical pressure drop across the bed by Ergun, Eq.(4.19).

$$\epsilon = 1 - \frac{M}{\rho_p V_b} \qquad \text{.....(4.14)}$$

$$\frac{\Delta p}{H} = \frac{p}{\bar{p}} \left\{ 150 \frac{(1-\epsilon)^2}{\epsilon^3} \cdot \frac{\mu U_1}{d_p^2} + 1.75 \frac{1-\epsilon}{\epsilon^3} \cdot \frac{\rho_m U_1^2}{d_p} \right\} \qquad \text{.....(4.19)}$$

Good agreement was also obtained between theoretical and experimental pressure drop across the bed with abs. error less than 10%.

Experimental design of Box Wilson method was adapted to find minimum number of experiments required for the reaction, because of lake in both of HCl and silicon, also the range of values of these three variables were predicted : particle diameter [(10-450), (10-200)]*µm*, gas velocity (1.41-2.83)*cm/s*, and weight of bed (100-200)*gm*.

Experimental data collected by this design are successfully fitted a second order polynomial empirical correlations.

System design was concerned about all parts of the system: bed, distributor, cyclone, and filter.

The aim of bed design was to find a suitable correlation between three variables affecting the type of fluidization : gas velocity, bed height and bed diameter.

The new model has predicted on the assumption that bed height taller than $0.6D$ causes slug formation that must be prevented because it decreases the reacted gas conversion, these two equations are the results:

$Z = f(D,U)$, $U_{mf} = f(D,Z)$, these two equation solved by trial and error method.

After registering the range of particle diameter, weight of bed, and reacted gas velocity from the hydrodynamic and experimental design sections, a reaction section began with experiments at different bed temperature (200-300) C in order to anticipate the optimum temperature value required for the best EtCl conversion, also to determine the reaction rate constant, which is very important in the kinetic model (model-1).

Eighteen experiments were performed to reflect the effect of each variable alone and as interact on the conversion of EtCl: bed temperature, particle diameter, bed height and reacted gas velocity.

It is found that conversion of EtCl increase with: a-decreasing reacted gas velocity, b-increase bed height and c-increasing particle diameter (45-150)μm. However the selectivity of Et_2SiCl_2 was not effected by the above variables, in comparison with the bed temperature which has a profound effect.

Two mathematical models were depended to predict theoretical data for conversion of EtCl.

Model-1 (Eq.(6.60)) takes into consideration only kinetic reaction variables: reaction rate constant, deactivation rate constant, reaction temperature, reacted gas inlet temperature, and molar flow rate of reacted gas. On the other hand model-2 (Eq.(6.73)) takes into consideration all above variables for model-1 and also fluidized bed variables: height of the bed, particle diameter, porosity, bed diameter, orifice diameter and minimum fluidization velocity.

$$X_A = \left(\frac{wt}{F_{...}} \right) \cdot 13.95797 \cdot \exp\left[\frac{-220314.57}{R_g \cdot T} \right] \cdot \frac{P_t \cdot Y_{A0}}{R_g \cdot T} \cdot \frac{(1 - X_A)}{(1 - 0.667 X_A)} \cdot \exp[-K_1 \cdot t] \quad \text{....(6.60)}$$

$$X_A = \frac{K_1'(1 - \beta\ e^{-t})}{1 - \beta\ e^{-t} + K_1'} = 1 - \frac{1 - \beta\ e^{-t} + \beta K_1' e^{-t}}{1 - \beta\ e^{-t} + K_1'} \quad \text{.....(6.73)}$$

These models were elaborated into a computer programs making the process of producing theoretical data fast and accurate.

The comparisons between Exp. and Theo. data shows good agreement with abs. error less than 9%

الخـــلاصـــة

يهـدف هـذا الـبحث دراسة انتاج مادة ثنائي اثيل ثنائي كلوروسلين (Et_2SiCl_2) من تفاعل الاثيل كلورايد (EtCl) مع السليكون (Si) بوجود عامل مساعد هو كبرس كلورايد (CuCl) في مفاعل لطبقة المميعة.

انجـز العمـل باستخدام نوعان من الاعمدة، الاول زجاجي (Q٧١) و بقطر 5سم و ارتفاع 50سم و استعمل لانجاز تجارب الهايدرو-ايلمك و الاخر نوع ستنلس ستيل و بنفس القياسات استعمل لغـرض انجـاز تجارب التفاعل. اما موزع الغاز فقد احتوى على 25 ثقب قطر الواحدة 1.0سم.

قـد العمـل في عدة اقسام هي : تجارب الهايدروايلمك، تصميم التجارب، تصميم المنظومة، تجارب التفاعل، النماذج الرياضية، و برامج الكومبيوتر.

فالهدف من تجارب الهايدروديلمك هو لايجاد اقل سرعة للغاز تتميع فيها الحشوة (Si) لعدة اقطار من الجسيمات.

اسـتعملت القـيم المستخرجة لتقييم معادلتان تستعملان لايجاد U_{mf} لاقطار مختلفة من الجسيمات و قد وجد تطابق جيد بين القيم المستخدمة من التجارب و تلك المستخرجة من المعادلتين (بنسبة خطأ 5%).