

Fluidization Characteristics of a Prototype Fluidized Bed Reactor

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Abstract

The fluidization characteristics of a prototype-fluidized bed laboratory reactor were understudied in order to investigate the suitable conditions at which the dehydrogenation reaction of butane could be carried out. To achieve this, a reactor with an effective volume of 1100ml was fabricated and coupled with temperature and pressure accessories.

Zeolites were obtained from the market and clay obtained from different sources and pre-treated was used as catalyst. Airflow at high velocity between 3000-7000ml/hr was used as the fluidising medium to obtain the bed characteristics while butane gas was used to obtain the dehydrogenation kinetics.

The temperature of the reactor system was varied between 353K and 413K while maintaining constant pressure of $1.5 \times 10^5 \text{ N/m}^2$ through a manifold gauge and a constant catalyst weight. Various methods such as pressure fluctuations, visual observations, and bed expansion were used to determine the transition velocity at which fluidization begins. It was observed that this depends on factors such as mean particle size, particle size distribution, and column diameter.

The minimum fluidizing velocity obtained for zeolite was 0.0133m/s and 0.0102m/s for treated clay materials both for a particle size of 250 μm . The conversion of butane over the catalysts showed an increase in both cases with a maximum at 0.9813 at 413K. This decreases as the reaction progresses.

Keywords

Prototype-fluidized bed reactor, Dehydrogenation, Minimum fluidising velocity, Conversion

Introduction

Historical development

The turbulent fluidization flow regime is commonly considered to lie between bubbling fluidization and the fast fluidization regime. It has been characterized by low amplitude of pressure fluctuations, resulting from the disappearance of large bubbles/voids. The first photograph of a turbulent fluidized bed, distinctly different from bubbling fluidization, was published by Matheson, Herbst and Holt (1949). A turbulent fluidization regime was introduced in the flow regime diagram of Zenz (1949). The first quantitative study seems to have been performed by Lanneau (1960) who measured local voidage, voidage fluctuations and pierced void lengths in a 76mm ID fluidized bed with fine catalyst particles at high gas velocities, although the transition from bubbling/slugging to the turbulent regime was not quantified. Kehoe and Davidson (1970) extended their work on slugging to higher velocity operation and identified the transition from bubbling to turbulent fluidization based on visualization of a 2-D bed and bubble rise velocity and capacitance traces in a 3-D column. Later the turbulent fluidization regime was reported by Massimilla (1973), Thiel and Potter (1977) and Crescitelli, Donsi, Russo and Clift (1978). In these early studies, transition to turbulent fluidization was generally determined based on visual observations and local voidage or pressure traces. The first transition criterion was proposed by Yerushalmi, Cankurt, Geldart and Liss (1978) to quantify the transition from bubbling/slugging to turbulent fluidization. The gas velocity, at which the standard deviation of pressure fluctuations reached a maximum was said to mark the beginning of the transition to turbulent fluidization. In recent years, the gas velocity has been widely adopted to define the transition to turbulent fluidization. Grace, Issangya, Bai and Bi (1999a) proposed several criteria to be satisfied for any flow regime:

- There must be distinctive features. The darting transitory voids viewed in Type I turbulent fluidization distinguish turbulent fluidization from the other fluidization flow regimes;

- There must be distinctive trends. Such features as the radical improvement in gas/solid contacting (Massimilla, 1973), reversal of the direction of the pressure fluctuation versus gas velocity plots (Yerushalmi & Cankurt, 1979), change in the tendency for gas mixing to vary with gas velocity, unique statistical and chaotic properties (Bai, Issangya & Grace, 1999) and a decrease in the amplitude of the root mean square force on immersed horizontal tubes (Grace & Hosny, 1985) are clear examples of distinctive trends;
- A flow regime should also be capable of being both fully developed and statistically steady. This criterion also appears to be met by the turbulent fluidization flow regime as defined above.

When the flow of fluid passes upwards through a particle bed and the drag force acting on the constituent particles is attained to balance the gravitational force exercised on the particles, the bed starts to be fluidised. With a further increase in the flow rate, each particle is put in motion from its quiescent state to a continuously agitated state in the upward stream of fluid. Therefore such a fluidized bed –which is used for heat exchangers, chemical reactors and drying equipment, etc-can well be expected to have an overall efficient contact of particles with fluid which results in a uniform temperature in the bed and a high rate of heat transfer between particles.

In general, it is well known that the mixing state in the fluidized bed is classified into two categories, that is, a state of aggregative fluidized bed and that of particulate fluidized one. In the former case, bubbles form in the particle bed while, in the latter case, bubbles do not form.

Regarding the clarification of a characteristic difference between the two beds, some light has been thrown on the subject, experimentally or theoretically, by studies of pressure drop across the bed, drag force acting on the particles and interstitial flow between particles (Pigford and Baron, 1985). Wilhelm et al proposed the use of the Froude number, $F_{r_{mf}} = u_{mf}^2 / d_p g$, where u_{mf} and d_p are minimum fluidization velocity and particle diameter, respectively based on the concept of interactive forces in the vicinity of bubbles generated. They predicted experimentally that the fluidized bed would become particulate if $F_{r_{mf}} < 0.13$ and aggregative if $F_{r_{mf}} > 1.0$. For instance, estimating their criterion of Froude number by using experimental data, one finds that the evaluated critical Froude number results in less

than 0.13, when the behaviour of fluidization observed is found to still be aggregative (Fetterman, 1984).

This might be caused by mainly the fact that their resulting formulation was derived from only the interaction force balance at the minimum fluidization. Romero et al (1982) suggested a modified criterion of dimensionless groups including Froude number and Reynolds number, $Re_{mf} = u_{mf}d_p/\nu_f$ where ν_f is the cinematic viscosity of fluid, on the basis of the instability theory of the bed as developed by Wilhelm et al (1983) whose bed stability theory is based on considering the fluidising medium and dense bed as physically separate phases, especially when the interface of physical system pertinent to bed stability adopted in their study is perpendicular to the fluidising fluid flow direction. They suggested that the criterion between particulate fluidized bed and aggregative fluidized bed is about 100 in a product of dimensionless groups, such as $(\rho_p - \rho_f)/\rho_f$, $F_{t,mf}$, Re_{mf} , and L_{mf}/D_c . Their prediction, however, is no more than agreeable to the experimental results in comparison with the Froude number criterion by Wilhelm et al (1983). Moreover, the use of a product of these groups as a criterion of fluidization is seen to be nebulous (cloudlike or vague) in the cases of air fluidization of mica carried out in the present study. Jackson (1983) and Pigford (1985) carried out their investigation on a premise that the criterion should be based on a consideration of the stability or growth rate of disturbances. However, it might be said that they made their prediction without detailed discussions of whether the solution derived from the equation of motion would diverge or not. Concluding this, notwithstanding the recognition that one of the factors comprised in the criterion is the Froude number, it should be noticed that these previous predictions for the criterion have not been met with satisfactory success. On the other hand, Harrison et al (1971) predicted a stable bubble to be possibly generated in the particle bed after fluidization. Theory of Harrison, et al. merely classifies fluidization into bubbling, transition, and non-bubbling fluidization. However, it is clear that the theory does not predict in advance which types of fluidization in a given system would exhibit in future. Moreover, Verloop et al (1971) confirmed analytically that the particles bed behaves homogeneously at a low voidage after minimum fluidisations. These results may well suggest that both the generation of bubble nuclei and the homogeneous expansion of particles bed could be assumed in the vicinity of minimum fluidization. Furthermore, it is considered that these interpretations throw a meaningful light on the clarification of the criterion problem.

Characteristics of fluidized bed reactor at high velocity fluidization

The behaviour of fluidized bed reactor at its fully developed stage requires the understanding of particles-fluid motion at the beginning of and after fluidization. It is experimentally well known that the pressure drop across the bed after fluidization could be described by Carman-Kozeny equation. When the fluid velocity has reached minimum fluidized one, the pressure drop across the bed will be equal to the total weight of the constituent particles per unit area. At the moment of minimum fluidization or the onset of particles motion, the net gravitational force acting downwards on each particle is balanced by the upwards component of the drag force. In order to ascertain the physical validity of Froude number as the criterion for prediction, it is required to consider the particles motion from their initial contact to onset of minimum fluidization. At the minimum fluidization, the well-known dimensional analysis of the particles motion under equilibrium is presented as follows:

$$U_{mf}^2/d_p g = 4/3(1/C_{dmf})(\rho_p - \rho_f)/\rho_f, C_{dmf} = C_{dt}f(\varepsilon) \quad (1)$$

where g is the gravitational acceleration, ρ_p density of particle, ρ_f density of fluid, C_{dmf} apparent drag coefficient of bed, C_{dt} drag coefficient of a single particle, and $f(\varepsilon)$ voidage function.

Since the drag coefficient is usually a function of Reynolds number, that is, $C_d = f(Re_{mf})$, then $U_{mf}^2/d_p g$ is the same as Froude number. Considering these facts, equations above could be rearranged as follows:

$$Fr_{mf}/(\rho_p - \rho_f)/\rho_f = 4/3 f(\varepsilon)f(Re_{mf}) \quad (2)$$

ΔP , the pressure drop across the bed is closely related to drag force, F_d acting on the particles bed. The pressure drop across the bed is expected to be dissipated as the energy suspending the particles bed:

$$\Delta P/L = F_d f(\varepsilon).N = \Phi(\varepsilon)U_f \quad (3)$$

where L is the bed height, N the number of particles per unit volume, and $\Phi(\varepsilon)$ is the drag coefficient.

The above equation is based on the assumption that the drag force on the particles bed would be expected to depend on the relative velocity ($U_f - V_p$) between the fluid and the constituent particles as reported by Jackson (1963). However, in the steady state, V_p is negligibly small and also the available domain for $\Delta P/L$ equation is approximately $Re_p < 500$. Accordingly, it is expected that the bed expansion could be evaluated by $\Delta P/L$ equation when $\Phi(\varepsilon)$ is obtained.

One of the purposes of present study is to discuss the parameters dominating the behaviour of particles fluidized bed and to establish a theoretical model, which might be more pertinent to the behaviour of fluidized beds, is made for a prototype fluidised bed reactor. For this purpose, the reactor bed was packed with catalyst (aluminosilicates) and is fluidised by air under controlled pressures.

Experimental

Equipments

The equipment used in this work includes vacuum dryer, tray dryer, water bath and hot plate manufactured by Gallemp Kemp Ind. USA. Similarly equipments such as Atomic Absorption Spectrophotometer (AAS), Colorimeter, Electronic weighing balance, and mercury thermometer, manufactured by Cole-Parmer Instrument Company, East Bunker Court, USA. All the glassware's are products of Pyrex and Technico Ltd, UK.

Materials

The chemical used are concentrated H_2SO_4 of analytical grade manufactured by Fisons Ltd., England. Clay samples were collected from different locations. For example, table 1 below shows the details.

Table 1. Source of clay samples

Sample	Location
A	Paiko
B	Kutigi
C	Erio
D	Kaduna

Sample Analysis

The analysis carried out on these samples was aimed at quantitative determination of the mineral constituents such as: SiO_2 , Al_2O_3 , Fe_2O_3 , CaO , TiO_2 , K_2O , and Na_2O among others. The physical characteristics of importance such as surface area, particle size distribution, and particle density were also determined (see Appendix for details).

Catalyst Pre-treatment

The catalyst was prepared from clay obtained locally. It was ground and sieved to a particle size of 250 microns and later calcined between 550-600 °C. The catalyst was acidified using 0.1% w/w dilute sulphuric acid. 0.15kg of the sample was placed in a beaker where distilled water was added to make a paste slurry. 150ml of dilute acid was added to soak the paste for 60 minutes at a controlled temperature range of 80-100°C. This was later dried at 393K for 12 hours. The stated procedure was carried out since catalyst surface is where the conversion in a catalytic reaction takes place. The surface of the catalyst is non-uniform or heterogeneous and, certain spots or locations on the catalyst which actually participate in the reaction are referred to, as active centers or sites. As regards activity and selectivity, active centers have a certain activity associated with them. Catalyst activity refers to the rate at which the catalyst causes a reaction to proceed toward equilibrium while selectivity is for a certain product or reaction, normally some intermediate product.

Methodology

The kinetic experiments were carried out in a lagged fluidized bed reactor at high-pressure equipped with a cyclone, thermometer, and a pressure gauge. The effective volume of the reactor is 1100mL (or 1.1 Litre). A thermal sensor, an external heating element together with a heating regulator provided temperature control in the reactor with an accuracy of $\pm 1^\circ\text{C}$. The gas sample was collected through a delivery tube into petroleum ether for absorption. This was analyzed using a gas chromatograph with the liquid-injection method at Kaduna refinery and petrochemical company (KRPC) laboratory from where the percentage conversion and composition of other products were obtained.

The experiments were carried out in a fluidized-bed operation in a 0.085m diameter reactor with a total height of 0.195m. The distributor plate has holes with diameter of 0.002 m and is 0.005m thick.

The gas-phase feed to the reactor is through a manifold gauge connected to the butane cylinder source and volumetric meter where the pressure was obtained. The kinetic experiments were undertaken in two stages: in the first stage, the reactor was operated at low gas velocity of 2500ml/h within temperature range of 353K-413K and at a constant pressure

of $1.5 \cdot 10^5 \text{N/m}^2$. The second stage involved the operation of the reactor at high gas velocity of 7000ml/h at the same temperature and pressure conditions as in the first stage. Both stages of the reactor operation were maintained at steady state conditions.

The dimensions of the reactor are as given above. The test section of the reactor is made of pyrex transparent glass in order to visually observed the motion of the particles in the bed. In the present experiment, the pressure of supplied gas (butane or air) is measured by a manifold gauge from which the minimum fluidizing velocity was calculated. The solid particles adopted in these experiments are aluminosilicates (zeolite, clay and mica). The physical properties of these particles are given in table 2 below. The size of the particle listed in this table is evaluated by the mesh screen analysis. Prior to each run, the particles bed is prefluidized to ensure uniformity of packing. The pressure drop across the bed is measured before and after fluidization.

In a fluidising state, the expansion of the bed is also measured. All the experimental data are obtained under a steady state conditions. A relation between pressure drop and flow rate of gas is examined for several height ranging from 10mm to 100mm.

Table 2. Experimental condition

Solid Material	Particle Size (μm)	Particle Density (kg/m^3)	Fluidizing Fluid (Medium)
Zeolite	250	1120	Air
Clay	250	1110	Air
Mica	250	1110	Air

Results

Table 3 shows the characteristics of zeolite at a gas flow of 1000-5000 cm^3/min . The results showed that the U_{mf} increases as the gas flow increases. A similar trend was obtained for Reynolds number. Table 4 shows that the characteristics of clay at a gas flow of 1000-5000 cm^3/min . The results showed an increase in the bed expansion as the gas flow increases. A similar trend was obtained for Reynolds number and pressure drop. Table 5 below showed an increase in the conversion of butane over the range of temperature considered until a certain time when conversion was observed to decrease.

Table 3. Bed characteristics of zeolite

No	Gas Flow (cm ³ /min)	U ₀ (m/s)	Particle diameter (m)	Pressure drop (kg/ms ²)	U _{mf} (m/s)	N _{Re} (dim. less)
1	1000	1.75e-01	8.50e-04	3.447e+01	0.024546	2.39e+03
2	2000	3.50e-01	6.00e-04	6.894e+01	0.020623	4.78e+03
3	3000	5.25e-01	4.25e-04	1.034e+02	0.017357	7.16e+03
4	4000	7.00e-01	3.00e-04	1.379e+02	0.014583	9.55e+03
5	5000	8.75e-01	2.50e-04	1.723e+02	0.013312	1.19e+04

Table 4. Bed characteristics of clay

No	Superficial velocity (m/s), U ₀	Bed expansion (dimension less)	Particle diameter (m)	Pressure Drop (kg/ms ²)	U _{mf} (m/s)	N _{Re} (dimension less)
1	1.75e-01	0.00111	8.50e-04	61.846	0.0189	2.37e+3
2	3.50e-01	0.01000	6.00e-04	123.692	0.0158	4.73e+3
3	5.25e-01	0.03857	4.25e-04	185.541	0.0133	7.10e+3
4	7.00e-01	0.10667	3.00e-04	247.387	0.0112	9.47e+3
5	8.75e-01	0.25000	2.50e-04	309.237	0.0102	1.18e+4

 Table 5. Conversion data of butane over a range of temperature 353-413K at constant pressure of 1.5·10⁵ N/m²

No	Time (s)	Conversion, X			
		T=353K	T=373K	T=393K	T=413K
1	300	0.15738	0.34201	0.4800	0.54002
2	600	0.14339	0.5400	0.62002	0.6400
3	900	0.75001	0.64112	0.8097	0.9813
4	1200	0.7600	0.7680	0.8674	0.8200
5	1500	0.7800	0.81002	0.8696	0.78002
6	1800	0.70002	0.6500	0.70001	0.7411

Table 6 shows the X-ray Analysis of Sample A. This analysis shows the composition of the clay sample. Table 7 shows the constituents of sample B. Table 8 shows the composition of sample C. Table 9 shows the composition of sample D.

Table 6. The X-ray Analysis of Sample A

Conc. (%)	S _i O ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	K ₂ O	Na ₂ O	SO ₃	CaCO ₃	MgCO ₃	SR	AR	LSF	total
	81.13	10.42	4.05	0.06	0.25	1.33	0.06	0.08	0.11	0.52	5.61	2.57	0.03	97.37

Table 7. Constituents of sample B

Conc.	Al ₂ O ₃	S _i O ₂	Fe ₂ O ₃	CaO	MgO	K ₂ O	Na ₂ O	SO ₃	Cl	CaCO ₃	MgCO ₃	SR	AR	total
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(%)	51.79	29.78	6.99	-0.21	-0.39	0.24	0.1	0.09	0.061	-0.38	-0.81	1.41	4.26	88.44
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Table 8. The composition of sample C

Conc.	S _i O ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	K ₂ O	Na ₂ O	SO ₃	Cl	CaCO ₃	MgCO ₃	SR	AR	total
(%)	73.55	18.14	3.14	-0.08	-0.47	1.28	0.1	0.08	0.049	-0.14	-0.98	3.46	5.77	95.80

Table 9. The composition of sample D

Conc.	S _i O ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	K ₂ O	Na ₂ O	SO ₃	Cl	CaCO ₃	MgCO ₃	SR	AR	total
(%)	62.31	20.64	4.28	1.17	-0.44	1.79	0.1	0.08	0.056	2.1	-0.93	2.5	4.83	89.98

Discussion of Results

The X-ray analysis of the clay samples reveals some differences in their constituents. For instance, it was observed that sample A has the highest Silicon Oxide concentration, and this may be due to the fact that it is sedimentary in nature, that is its formation was as a result of decomposed marine and aquatic matters. Also from table 6 it was observed that sodium oxide (Na₂O) and Calcium Oxide (CaO) has the least concentration in the sample. The total percentage is 97.37 of the constituents with the silicon ratio (SR) being very high at 5.61; this is due to the high amount of concentrations in silicon oxide S_iO₂ of the clay sample. It was observed for sample B from table 7 that the aluminium concentration was quite high when compared with sample A thus having a high aluminium ratio (AR) of 4.26 percent, but on observation also, the calcium oxide (CaO), magnesium oxide (MgO), and their carbonates has a negligible percentage concentration with a negative value with the calcium and magnesium oxides having -0.21 and -0.39 respectively, and their carbonates with -0.38 and -0.81 percents. Due to the high level of the aluminium and iron oxides, the silicon concentration is quite low when compared with sample A as a result low silicon ratio (SR) is observed.

Similarly for sample C from table 8 shows a high concentration of silicon oxide with 73.55 percent and the aluminium ratio (AR) being on the high side of 5.77 percent. This may be due to the low level of concentrations of the aluminium (Al₂O₃) and iron Fe₂O₃ oxides with 18.14 and 3.14 respectively. The oxides of calcium (CaO), magnesium (MgO) and their carbonates were observed to carry a negative value depicting a negligible value of concentrations of these constituents. For sample D in table 9, the aluminium oxide

concentrations are quite high when compared with sample C with 20.64 percent, thereby having a higher aluminium ratio (AR) of 4.83 percent. The magnesium oxide and carbonates of this clay shows a negligible concentration of -0.44 and -0.93 respectively.

The above characteristics exhibited by the clay samples indicate their suitability as catalyst. For example, the loss of weights indicates the fineness of the clay particles, thus increasing the surface area for catalytic processes. The loss of ignition observed (table 10), gives a loss of weight that is associated with the ionic charges of the clay samples thereby making it suitable for use as a catalyst. Comparing these with the properties of a catalyst possessing an active sites that is highly concentrated, and are structurally arranged, the same can be said of the clay minerals, which have a well structured constituents, such as the sample A with high silicon level of concentration, the silicon oxide forms an hexagonal structure, this structural bonds gives room for catalytic process.

Table 10. The loss of ignition test of the clay samples

SAMPLE	A	B	C	D
Weight of crucible + contents before ignition test	55.4755	54.8269	15.2919	55.5925
Weight of crucible + contents after ignition	55.4620	54.7737	15.2828	55.5728
Loss of ignition	0.0135	0.0532	0.0091	0.0197
Percentage loss of ignition	1.35	5.32	0.91	1.97

The conversion of butane is low at the beginning and increases with the time until a steady state is reached. The product of dehydrogenation of Butane using aluminosilicates (zeolite and clay) as catalyst yields but-1,3-diene alongside other products and some unconverted butane. The profiles of conversion versus gas feed rate had been presented earlier. It indicates that at high gas flow rate, the conversion of reactant to products drops; the reason adduced for this could be largely attributed to deposition of coke on the active surface of the catalyst thus bringing about a decrease in conversion. Thus, the fluidization characteristics of the bed were understudied. From the results, a minimum fluidising velocity, U_{mf} of 0.0133 m/s for zeolite and 0.0102 m/s for clay were used for a particle size of 250 μ m at a high velocity of 7000 ml/hr. This is not contradictory for the simple fact that coking could be minimized and the high conversion of the reactants observed before the conversion drops could be marked and the catalyst removed for regeneration. However, when the results obtained were compared with un-fluidized beds, that are fixed beds, there is a marked

difference. The catalyst particles are stationary at low velocities. Thus, conversion is quite low and it was observed that the process took a longer time that in economic sense is not desirable.

The plots of conversion versus the process parameters showed a curved shaped graphs showing that the relationship between conversion or the catalyst activity and these parameters are not directly proportional, rather, there is a point of diminishing returns at which a change in the given process variable would have no positive effect on the conversion level. This point may be regarded as optimal value of some sort; and increasing the process parameters beyond this point would lead to a decrease in conversion and hence reduction in the performance of the catalyst activity. It was also observed that as the temperature range and molar flow rate increases for the same weight of catalyst, there was an increase in conversion. The high values of conversions obtained are primarily due to the effectiveness and high activity of the aluminosilicate catalyst.

The influence of gas velocity on particle mixing was analysed in terms of internal solid fluxes calculated by means of the bed pressure equations (see the attached figures). It will be observed from tables 3 and 4 above that as the velocity is increased, the pressure drop increases across the bed length and once the bed is fluidised, the pressure drop remains constant but the height continue to increase with increase in flow. The pressure drop is dependent on the porosity of the particle considered. In this case it is higher in clay used than in zeolite. The lack of accuracy may be due to the wide particle size distribution that resulted in segregation during the experimental runs.

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