Integr. Res. Adv. 2015, 2(1), 5-10 Article

Integrated
Research Advances

Integrated Science

Residence time distribution study for continuous column packed with tea waste biomass

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Received Date: 18-Sept-2015 Published: 4-Nov-2015

ABSTRACT

In this study residence time distribution (RTD) and its deviation from ideal plug flow conditions was studied in a packed bed reactor under plug flow condition. The flow rate of the inlet stream was kept constantfor all the three case studies (step input without agitation in feed tank, step input with agitation in the feed tank and pulse input). Dispersion model was employed to investigate the presence of axial dispersion inside the packed bed reactor. The mass transportphenomena i.e. advection or dispersion was determined by the help of peclet number.

Keywords: Packed bed, Residence time, dispersion, axial, peclet

INTRODUCTION

The continuous flow process has been used widely in the industries dueless time consumption and cost effective process. The reactor flow configuration is an important parameter for the design of reactor weather it is plug flow, CSTR (continuous stirred tank reactor), fluidized bed reactor, Packed bed reactor.[1] There are two types of flow in ideal reactor i.e.completely mixed flow or completelyplug flow,[2] but in real reactor there is a deviation in flow patterns from ideality therefore the reactor in the real scheme is neither completely mixed flow nor plug flow.[3]

The flow model of real reactor is usually lies somewhere between plug flow and mixed flow. The reason for the deviation from ideal condition is the recycling of the fluid or the creation of stagnant region or channeling of fluid, differences in temperature, air gap inside the reactor, inadequate mixing with the influent and axial dispersion. Therefore, the determination of fluid velocity distribution is important for the economic design of the reactor.[4] Consequently, for the better understanding of velocity distribution of fluid particles inside the reactor is important to study the distribution of the residence time (RTD) because it determines the way in which an individual molecule passes into a reactor.[5]

There are two phenomena i.e. advection or dispersion by which fluid flows inside the reactor.[6, 7] In advection, the influent flows in the reactor with the current velocity due to the laminar flow but in dispersion, there is a longitudinal or axial material transport due to velocity differences, formation of turbulent eddies and molecular diffusion[8]. In the present study, RTD (Residence time distribution) of continuous reactor and the prediction of its flow pattern based on the theories of plug flow and mixed flow patterns is studied. Different case studies (step input without agitation, step input with agitation and pulse input) were performed. The RTD curves were plotted for all the different case studies and compared with ideal plug flow conditions to determine the actual flow behavior of packed bed reactor.

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Cite as: *Integr. Res. Adv., 2015, 2(1), 5-10.*

MATERIALS AND METHOD

Experimental Setup

The experimental setup consists of a packed bed column made of stainless steel, an inlet feed reservoir and feed pump (peristaltic pump) for controlling the flow rate of influent passed through the reactor. The reactor consists of a packing of tea waste biomass and was 1 m long with an outside diameter of 8 cm. The reactor block diagram is shown in figure 1.

Chemicals used for experimentation

All the chemicals used in this experiment were of analytical grade and obtained from HIMEDIA Laboratories Pvt. Ltd., Mumbai, India. NaOH solution is prepared by adding 400 grams of NaOH pellets in 1 liter of Millipore water. 0.2N solution of oxalic acid was prepared by adding 9 g of oxalic acid in 1 L of Millipore water. Phenolphthalein indicator was used for the

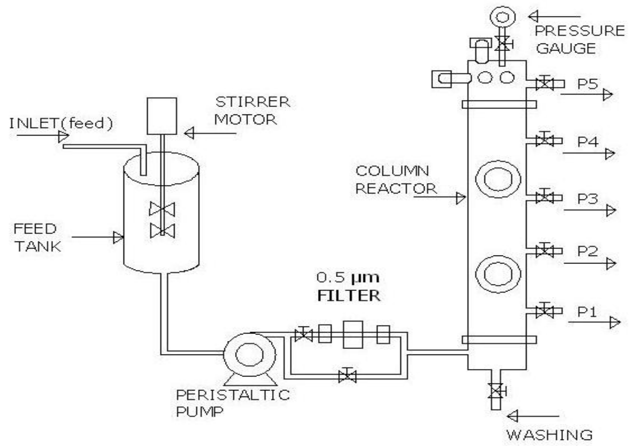

Figure 1. Block diagram of packed bed reactor used for RTD study

analysis of effluent concentration. 100 ml of phenolphthalein indicator was prepared by adding 0.5 g of phenolphthalein in 100 mL of ethyl alcohol 50% solution.

Experimental procedure

The packed bed reactor was filled with a packing of tea waste biomass. The reactor packing and walls were washed with simple tap water for the removal of dirt particles. After cleaning with tap water the packing was washed 2-3 times with Millipore water to further clean it. Tracer used in all the trials was NaOH. The NaOH solution was first standardized by titrimetric method using 0.2 N oxalic acid solutions. HCl solution was then standardized using the standardized NaOH by titrimetric method. The titrations were conducted using phenolphthalein as an indicator. First filling the system fully with water and then draining it into a large graduated cylinder calculated working volume of the reactor. After all the water has been drained out, the volume of the water that was emptied into the graduated cylinder was measured. Different case studies were carried out to understand the flow pattern of the reactor. Three types of input step input without agitation, pulse input and step input with agitation was introduced into the reactor[9].

First Case Study (step input without agitation)*:* In the first case study, the rpm of the peristaltic pump was set at 25 rpm and 1 L of concentrated NaOH making it a step input was introduced inside the reactor. The volumetric flow rate of effluent from the reactor outlet at 25 rpm was measured using a measuring cylinder and a stopwatch. Samples were collected from the reactor outlet at an equal time interval of 2 min. 3-4 drops of phenolphthalein indicator were added to the samples were titrated using standardized HCl solution to calculate the concentration of NaOH tracer in the outlet stream. The samples were turned pink due to the presence of NaOH in the effluent sample. The Concentration curve or C-curve was plotted using the output tracer concentration data. The resulting curve in this case is also known as F curve.[10]

Second case study (pulse input): The Packing of the reactor and walls were washed again with Millipore water 2-3 times to ensure that no traces of NaOH were left into the reactor. It was confirmed by the addition of the phenolphthalein indicator to the effluent. In the second experiment, the peristaltic pump was set at 25 rpm and still Millipore water was passed into the reactor until the water ran out from the reactor outlet. Then 100 mLNaOH solution was introduced into the reactor as pulse input. Samples were taken out from the outlet of the reactor over an equal interval of time of 2 min. The tracer concentration in the outlet stream was calculated using the titration method with a standardized HCl solution and the phenolphthalein indicator. Similarly, a curve of effluent concentration vs. time is plotted, which is also known as E curve.

Third Case Study (step input with agitation): The reactor was washed again 2-3 times from Millipore water to remove the traces of NaOH. The peristaltic pump was again set at a 25 rpm. A stirrer was added to the reactor system in the inlet tank, which creates an agitation with a speed of 1400 rpm. After washing of the inlet tank and tubular reactor, 1 L of a standard solution of NaOH was added again as a step input into the feed tank with continuous agitation in the inlet feed tank. The volumetric flow rate was recorded with stirring using a measuring cylinder and a stopwatch. Output samples collected after every 2 minutes were analyzed using a standardized HCl solution and phenolphthalein indicator.

Experimental methods for plotting an RTD curve:

RTD curve or the E-curve is typically plotted according to levenspiel using stimulus response experiment. Where the stimulus is a non-reactive tracer, which is introduced inside the stimulus-response reactor. The tracer concentration at the exit provides the response of the reactor or vessel[11].

The tracer used must be

1. non-reactive
2. easily detectable
3. The viscosity should be the same as the carrier or solvent fluid
4. It should not be absorbed by the packing material of the reactor or the reactor walls.
5. There should be not much difference in density between the tracer and the transport solution in the reactor.

THEORY:

Residence Time Distribution: Different fluid particles pass through the reactor through different path in different times. The age distribution of the fluid stream leaving the reactor is called exit age distribution E also known as residence time distribution. The unit of E is time^{-1}. Area under the curve is unity[12].

$\int_0^\infty Edt = 1$ (1)

E(t)dt = fraction of material in exit stream with ages between (t, t+dt)

Where E= exit age distribution

Fraction of material in the reactor between ages of (t, t+dt) is represented by internal age distribution I(t).RTD curve for a tubular reactor given by Levenspeil is shown in figure 2.

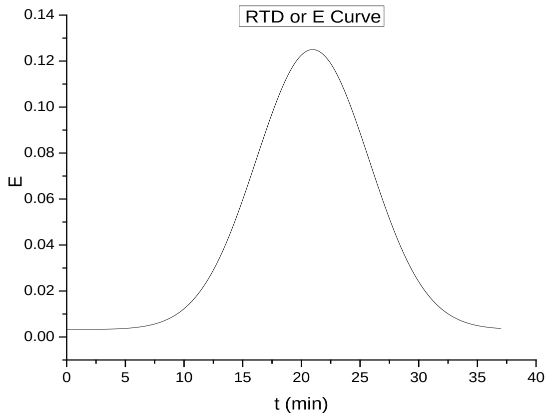

Figure 2. Typical RTD Curve for a vessel

Fluid element with age less than 't_1' or younger than age 't_1' can be represented by $F(t_1)$ which is the "cumulative distribution" of the fluid fraction in the reactor[13,14].

$$\int_0^{t_1} Edt = F(t_1) \quad (2)$$

And the fluid element older than t, can be represented as

$$\int_{t_1}^{\infty} Edt = 1 - \int_0^{t_1} Edt = 1 - F(t_1) \quad (3)$$

The residence time distribution can also be seen as a characteristic of mixing that takes place in the reactor.

Average residence time given by t_m is the mean value or centroid of the distribution, which is also known as the holding time, and it has the units of time[15].

$$t_m = \frac{\int_0^{\infty} tE(t)dt}{\int_0^{\infty} E(t)dt} \quad (4)$$

In the absence of stagnant zones or fluid channeling in the reactor or for a constant density system mean residence time 't_m' is equal to space time of the reactor 'τ'[16,17].

$$\tau = V/Q \quad (5)$$

Where V = working volume of the reactor, m^3 or mL

Q = volumetric flow rate of the fluid, m^3/min or mL/min.

t_m = mean residence time, min

The curve obtained by plotting concentration of fluid element in the outlet vs. time is called C-Curve. Area under the C-curve can be calculated as

$$A = \int_0^{\infty} Cdt \cong \sum_i C_i \Delta t_i = M/Q \quad (6)$$

Where C_i= Concentration of a fluid element at any time t_i, moles/L or g/L

M = Units of tracer introduced (g or moles)

Q = Volumetric flow rate of the fluid, m^3/min or mL/min.

The mean residence time can also be calculated from C-curve i.e. the mean of the concentration vs. time curve[18].

$$t_m = \frac{\int_0^{\infty} tCdt}{\int_0^{\infty} Cdt} \quad (7)$$

In discrete form t_m can also be written as

$$t_m = \frac{\sum t_i C_i \Delta t_i}{\sum C_i \Delta t_i} \quad (8)$$

Where C_i= Concentration of a fluid element at any time t_i, moles/L or g/L

E-curve can also be plotted from C-curve by using the following equation

$$E = \frac{C_{pulse}}{M/Q} \quad (9)$$

Another RTD function measured in terms of mean residence time is E_θ, which can be used further to understand flow models clearly.

$$E_\theta = \tau E = \frac{V}{M} C \quad (10)$$

$$\theta = \frac{t}{\tau} \quad (11)$$

The spread of distribution is measured in terms of a descriptive quantity called variance σ^2, defined as[19].

$$\sigma^2 = \frac{\int_0^{\infty} (t-t_m)^2 Cdt}{\int_0^{\infty} Cdt} = \frac{\int_0^{\infty} t^2 Cdt}{\int_0^{\infty} Cdt} - t_m^2 \quad (12)$$

This in discrete form appears like[20]

$$\sigma^2 \cong \frac{\sum (t_i - t_m)^2 C_i \Delta t_i}{\sum C_i \Delta t_i} = \frac{\sum t_i^2 C_i \Delta t_i}{\sum C_i \Delta t_i} - t_m^2 \quad (13)$$

Where Δt_i = time interval, time

The unit of σ^2 is $time^2$.

The real flow reactor can be compared from ideal flow patterns using different models to further categorize a particular flow pattern. Two flow models used for this purpose are dispersion model and tank in the series model.[21, 22] In this study dispersion model has been used for the description of the flow behaviour of the reactor.

Dispersion Model: C-distributing curve can be characterized using this model. A term dispersion or longitudinal dispersion D (m^3/sec) is used to describe the C curve. Large value of D signifies for broad spreading and small D is for slow spreading of C curves. If the value of D is zero, there is no mixing which states that it is plug flow reactor.[23]

A dimensionless group (D/uL) describes the flow inside the reactor. Dispersion model is used to describe the flow between the two extremes of plug flow and mixed flow. If (D/uL) approaches towards zero, there is plug flow. Whereas if (D/uL) tends to be infinite, there is a large spread and the pattern is mixed flow. Closed container for contour conditions, i.e. no diffusion, turbulence or swirling upward flow in the input or output of vessels, variance and dispersion may be interlinked using van der Laan (1958).

$$\sigma_\theta^2 = \frac{\sigma^2}{\tau^2} = 2\left(\frac{D}{uL}\right) - 2\left(\frac{D}{uL}\right)^2 (1 - e^{-\frac{uL}{D}}) \quad (14)$$

For open vessel boundary condition i.e. presence of no discontinuity of flow at the entry or exit of the reactor the relation can be expressed as

$$\sigma_\theta^2 = \frac{\sigma^2}{\tau^2} = 2\left(\frac{D}{uL}\right) + 8\left(\frac{D}{uL}\right)^2 \quad (15)$$

For both open and closed vessel condition i.e. presence of dispersion only at one end i.e. either at tracer injection point or at tracer recording point, the relation can be further expressed as

$$\sigma_\theta^2 = \frac{\sigma^2}{\tau^2} = 2\left(\frac{D}{uL}\right) + 3\left(\frac{D}{uL}\right)^2 \quad (16)$$

For small D/uL, equation 13, 14 and 15 can be further reduced to equation 16

$$\sigma_\theta^2 = 2\left(\frac{D}{uL}\right) \quad (17)$$

The dispersion values depict the axial dispersion degree in reactors is given in table 1.

Table 1. Dispersion number and flow pattern

Condition	D/uL, Dispersion Number	Flow pattern
No dispersion	0	Ideal plug flow
Low dispersion	<0.05	-
Moderate dispersion	0.05 to 0.25	-
High dispersion	>0.25	-
-	∞	Complete Mix

The inverse of dispersion number is peclet number. Peclet number denotes the ratio of mass transfer by advection to the mass transfer by dispersion. If the peclet number is greater than 1 then mass transfer by advection dominates and if peclet number is less than 1 mass transfer by dispersion dominates.

Results and Discussion

In this study, the volume of the reactor was calculated as 2900 mL. At 25 rpm of peristaltic pump the volumetric flow rate obtained without any agitation was 176 mL/min. In this case, a random tracer input was introduced into the feed tank then passed to the reactor through peristaltic pump when it started to drain out from the top of the reactor its concentration was analyzed in the effluent.

First case study: Figure 3 shows the curve representing the concentration of tracer in the outlet port with respect to time. There was no change in colour of the samples collected during the first 8 minutes. The concentration of tracer in the effluent was increased after 10 minutes of experimental run, and then showed a sudden increase after 18 minutes. After that the concentration of tracer in the effluent becomes constant up to 32 minutes. Then, finally, showed a decreasing trend for the next 34 minutes and finally the tracer was completely disappeared after 98 minutes. From equation 5, the theoretical average residence time of the reactor at a flow rate of 176 mL/min was calculated as 16 min and 47 s or about 17 min. The average residence time using the C curve or by using Equation 8 is obtained as 28 minutes and 66 seconds, or about 29 minutes.

Differences in retention time can be attributed to fluctuating flows due to intermixing or eddies in the reactor. The variance (σ_θ^2) in this case is found to be 142.46, which shows the presence of very large dispersion in the reactor. By using the dispersion model equation 16, the value of D/ul comes to be 71.23 whichshow large deviations from plug flow. According to Levenspeil[24,25] these large deviations are influenced by the input and output conditions of the reactor. The Peclet number which is the inverse number of dispersion was 0.014 which is smaller than 1 showing that the dispersion is the dominant factor in mass transferthan advection.[26]

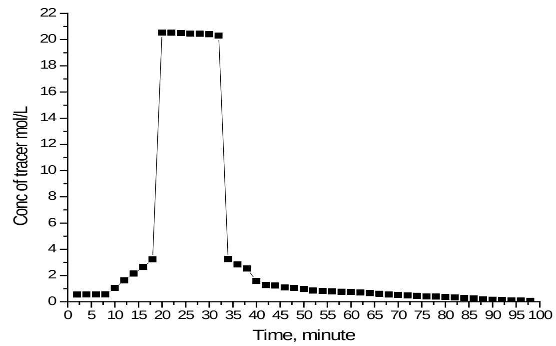

Figure 3. Response of Step input curve

Second case study: The volumetric flow rate in the second case studywas same as first case study176 mL/min. The tracer pulse input produces an output curve as shown in Fig. 4.In this case first, the tap water was passed through the reactor, when it starts to flow out from the top port of the reactor, 30 mL of tracer as a pulse input was introduced inside the packed bed reactor through peristaltic pump. The concentration of tracer in the effluent was increased up to 18 minute very slowly then increased suddenly and reached to the maximum value at 20 minutes. After that the concentration of tracer in the effluent was started to decrease. The C-pulse curve then followed a regular C_{pulse} curve fashion. The concentration curve did not show a zero concentration of tracer to the output indicating certain amount of mixing inside the reactor. The average residence time in this case obtained from the C-pulse curve is 20 min and 67 sec, which is about 21 min.

The theoretical average residence time is same as 17 min as in first case study. The quite large difference in the residence time is definitely showing a large amount of intermixing, presence of turbulence or the presence of dead or stagnant zones in the reactor[27]. The variance (σ_θ^2) in this case is about 0.231 and the dispersion number (D/ul) is approximately 0.116. From Table 1, it is evident that as the dispersion number is between 0.05 and 0.25, so there is a moderate deviation from plug flow. The peclet number is 8.66, which is more than 1, showing that the movement of the tracer is mostly by advection.

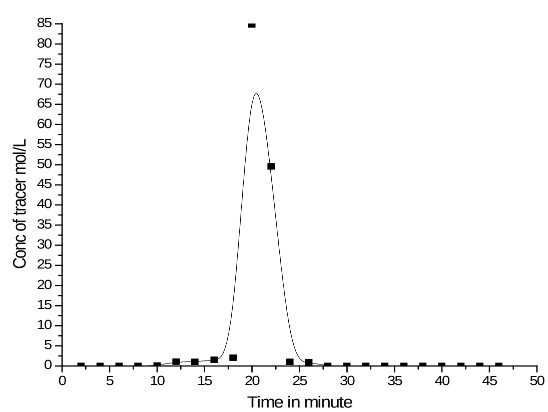

Figure 4. Response of pulse input

Third case study: In this case agitation of tracer was carried out in the feed tank attached to the reactor after introducing concentrated NaOH as tracer in the feed tank as pulse input. The rpm for the third case was again 25 but an agitation was produced in the feed tank by a stirrer at an rpm of 1400 rpm. The volumetric flow rate at this inlet condition was reported as 128 mL/min. The C_{pulse} curve for this case is shown in Figure 5.

The C curve obtained in this case does not follow the regular trend of C-curve of step input. The C pulse curve obtained from the reactor outlet samples itself shows a quite large deviation from plug flow behaviour. The curve pattern obtained is more like the pattern obtained for the mixed flow reactors. The stirrer at a high rpm of 1400 mixed the tracer completely inside the feed tank which completely created an open flow boundary condition at the entry point of the tracer creating a mixed flow boundary condition both at entry and exit points of the tracer. Tracer first appeared after 2 min and its concentration remained constant till 10 min which was due to the mixing in the inlet feed tank then the tracer concentration increases itself for the next 18 min and finally after 20 min the tracer concentration reached its maximum value and remained at the maximum point for the rest of the time the experiment was carried out. The uniformity in tracer outlet concentration in this case was due to the vigorous mixing inside the inlet tank. The mean residence time as calculated using equation 5 is 17 min. Residence time obtained from the mean of C_{pulse} curve is 31 min and 92 sec which is approximately 32 min. The difference in residence time can be attributed to the fact that vigorous mixing takes place in the inlet feed tank at an rpm of 1400 due to which there is absence of closed boundary conditions at the tracer entry and exit points. The variance calculated in this case is 52.67 and therefore the dispersion number is 26.34 which show moderate dispersion. The peclet number comes to be 0.038 depicting dispersion to be dominating over advection mode of mass transfer.

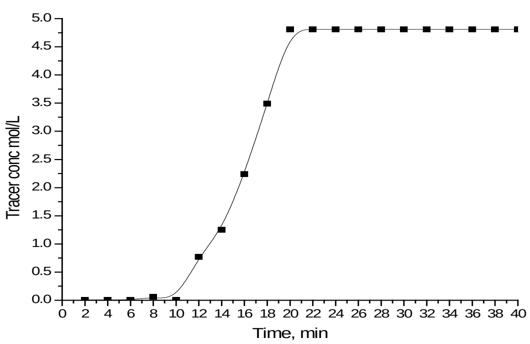

Figure 5. Response of Step input with agitation

Table 2 shows the data of the RTD studies carried out on the reactor. According to the table the maximum difference in residence time is observed in third case study. This difference can be due to the vigorous intermixing produced and the absence of closed boundary condition in the tracer entry and exit points.

Table 2 Experimental data for the RTD studies

S. No.	Tracer Input	Mean Residence Time (τ), Min	Mean of C-Curve (t_m), min	σ_θ^2	D/uL	Difference (t_m-τ), min
1	Step	17	29	142.6	71.3	12
2	Pulse	17	21	0.231	0.16	4
3	Step	17	37	52.67	26.34	20

Fig. 3, Fig. 4 and Fig. 5 shows the concentration of the effluent as a function of time for all case studies. All curves are non-symmetrical in nature that shows the moderate to large differences from plug flow conditions. The curve is not even close to the regular pattern of the RTD curve for the experiment where agitation was introduced into the inlet tank which can be due to the large difference between mean residence times. From Table 2, one can see that the value of the dispersion number is minimal for the second case for the pulse input without any agitation which shows that the longitudinal dispersion in this case is relatively less compared to other case studies. But there is a significant difference in the mean residence time theoretically and experimentally obtained may also be the result due to dead zones or stagnant region or channelling of fluid inside the reactor.

CONCLUSION

RTD studies were carried out in a tubular reactor using three different inputs, step, pulse and pulse input with agitation in the inlet feed tank. The experimental results of three different case studies were compared with ideal case of plug flow. Stirring of the influent solution into the reactor caused the reactor to move towards mixed flow behaviour after a certain time interval giving a constant output for the rest of the operating time. RTD study shows that in case of pulse input without agitation the mass transfer process was through advection while in case of step input and pulse input with agitation the mass transfer was through dispersion. In the case of pulse input with agitation there is less longitudinal dispersion, due to the presence of dead zones or stagnant areas. Whereas in the case of step input dispersion was more as peclet number was less than that of pulse input with agitation. The plots of the concentration curve, E-curve, F-Curve, C- curve is used to describe the flow of reactor, but are not very accurate. RTD studies are important in designing of the reactor as it provides the economic design of the reactor by understanding the actual flow conditions in the reactor to overcome the energy losses. It also provides the information about the time period during which a molecule spends within the reactor which in turn describes the flow behaviour of the reactor.

Acknowledgement

The author gratefully acknowledges financial support provided by the MHRD assistantship by Government of India and Chemical engineering Department IIT Roorkee for the facility provided for conducting research work.

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