

Study of Residence Time Distribution in Chemical Industry

A Review

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ABSTRACT

Article Info

Volume 9, Issue 1

Page Number : 47-55

Publication Issue

January-February-2022

Article History

Accepted : 05 Jan 2022

Published : 17 Jan 2022

The concept of residence time distribution (RTD) is an important tool for the analysis of industrial units and reactors. The RTD of fluid flow in process equipment analyses their performance and checks the feasibility of a reactor. RTD method has been widely used in industry to optimize processes, solve problems, improve product quality, save energy, and reduce pollution. The technical, economic, and environmental benefits have been well understood and recognized by various sectors such as industrial and environmental sectors. The petrochemical industries, mineral processing, and wastewater treatment sectors are identified as the most appropriate target beneficiaries. This review traces current applications of the residence time theory in various chemical industries. Besides reviewing recent experimental studies in the literature, some common modeling, tracer injection and detection technique, and different parameters are studied to understand RTD. **Keywords** : Residence time distribution, Modeling, Tracer injection, parameters.

I. INTRODUCTION

The residence time distribution can be termed as the probability distribution of time that solid, or fluid particle stays in that unit operations. It is mostly equivalent to the time taken to complete that unit operation. RTD has a crucial design. designs are very important and, need improvement, and scale-up of many manufacturing processes. Parameters such as Peclet number (Pe), Dispersion number (Nd), or several tanks in series (not) in junction with mean residence time (T_r) are used by the RTD models to

differentiate between CSTR and PFR reactors. Many chemical processes on large scale are based on continuous flow reactors, which include plug flow reactor and continuous stirred tank reactor in practice, no process is ideal and shows many differences from ideal behavior. Therefore, the behavior of the real system is in between CSTR and PFR. Improper design, operating conditions that are fluctuating, scale-up effect, different raw material sources, non-uniform heating cooling, etc. are many reasons for deviation of processes from ideal behavior. These are one of the processes which can lead to a decreased nonuniform

quality of the product. Fluid mechanics and residence time distribution are used to describe the flow irregular patterns and hydrodynamics of any continuous flow reactor. RTD theory is best suited to predict nonideal flow patterns. Also the probability distribution function of time can be said with the help of RTD a material flowing inside the process equipment. Also process efficiency, mixing time, passing, channeling, dead zone, etc. can be studied in the designing stage itself, the improper design of reactor vessel can be obtained by RTD.

II. TRACERS

Different types of tracers are used in different phases like solid, gaseous, liquid. Mainly a tracer of unique property is chosen. This property can be nuclear, physical, chemical, or biological. The amount of tracer used is very small. Tracer is injected with the flow, and it moves along the flow. Tracer cannot be used mostly at high temperatures and pressure. Also, it is quite impossible to take readings of RTD under such conditions and there are chances of tracer getting spoiled. We cannot take RTD readings at difficult sections. Therefore, it is impossible to study RTD complex structure. Dyes are used because of their coloring properties. These can be traced by the naked eye because of the color or by spectrophotometer. On the other hand, in many places, these are not used because of their coloring properties only. Also using a small amount of dye cannot give proper results. Ionic and molecular structure influence the selection of these dyes as a fluorescent dye in high concentration can be toxic. Also, by the process chemicals, the fluorescent dyes can be changed e.g., pH. Ions are also used as tracers in less quantity. The problem of ion being used as a tracer is that it is not detectable in less quantity and can be regarded in a porous medium. Some ions have less stability in polluted water such as nitrates. Because of its low cost and non-hazardous nature, lithium chloride and sodium chloride are used. The good point of electrolyte tracer is that it can be

detected even in a small amount. The drawback is that it may sometimes react with the fluid. Acids and bases are used only when any change in pH is not affected but the process.[14],[15].

III. MODELING

Synman, G. C., and Smith, S. W studied the next step to study hydrodynamics by models. Advantages of RTD Modelling are any technical error or missing data can be found out by RTD measurements. The basic information is given in the RTD experiment. Further treatment modeling for the process is essential. Modeling gives more idea about the performance of reactor through different parameters depending on models. A model can be classified depending on several parameters as being either a one-parameter model or a two-parameter model. The RTD, which is completely an experimental method, is used to evaluate the parameter(s) in the model.

1. Axial Dispersion Model

Synman, G. C., and Smith, S. W studied ADM is more generalized for non-ideal reactors. ADM is used generally in long pipes with laminar flow, turbulent flow, packed bed, etc. The dispersion coefficient is the parameter to find non-ideality. Although it is a combination of PFR and perfect mixers, ADM is also an ideal substitute to generate the concept of RTD for non-ideal reactors. Boundary Conditions for the closed-closed system, (Without dispersion), in the case of closed-closed vessels, we assume that there is no dispersion of radial variation in concentration either upstream (closed) or downstream (closed) of the reaction section; hence this is a closed-closed vessel. Boundary Condition for this is Open-open system, (With dispersion) In an open vessel, dispersion occurs both upstream (open) and downstream (open) of the reaction section; hence this is an open-open system.[19]

2. Tank-In-Series model

This model illustrates N number of tanks of equal volume linked in series, according to Synman, G. C., and Smith, S. W. Even a minor change in N can cause a massive change in the residence time distribution for a small value of N, yet for a high value of N, the large change is insignificant and the model acts as a plug flow. In other words, the degree of mixing reduced as the number of tanks increased.[19]

IV. PARAMETERS

Variance, mean residence time are the essential parameters to find from the experiment. In the Axial Dispersion Model, Peclet number and Dispersion number are found. The dimensionless group, Peclet number, which is the ratio of convective to dispersive flow, is used to quantify the amount of dispersion present in a particular reactor. The inverse of the Peclet Number is the Dispersive Number. The axial dispersion method uses the Peclet number, a dimensionless criterion, to characterize mixing. Peclet number for non-ideal reactor has an estimating range from 0.5 to 24, Dispersion Number for PFR is 0, for CSTR is 3-4. In the Tank-in-series model Number of tanks (N) is found from variance and mean residence time. There are several approaches for estimating parameters. The most widely utilized approaches are the moment's method and the least square curve fitting methods. The variance from the experimental response curve was used to calculate the parameters for ADM and TISM. To determine the ADM parameters for a minimal amount of dispersion. The sum of the squares of the difference between the model response and the empirically measured response was minimized using this technique. The parameters with the smallest sum of squares of the difference between experimental and model responses are the best-fit ones. [24]

V. TYPES OF REACTORS

The continuous stirred tank reactor and the plug flow reactor are the two types of chemical reactors. In terms of kinetic characteristics, a plug flow reactor is more efficient than a stirred tank reactor. The flowing nature of a plug flow reactor is one reason that renders the process unsuccessful. Reactants and reaction products flow at varying speeds throughout the cross-section of the tube when the reactor flow is not optimal. This is due to the reactor's hydraulic elements being overlooked. A chemical reaction takes place inside the reactor, changing the substance of distinct chemical molecules. Every industry relies on the efficiency of its reactors. This has an impact on the materials used in processing, the amount of energy required, and the process's dependability.[10]

PFR

Because of its narrow residence time distribution, high surface-to-volume ratio, and low wall strength, PFR is beneficial. As a popular way of obtaining that distribution, the pulse experiment relies on the tracer injection having a perfect pulse. Errors occur when there is a variation from a perfect pulse. This impact is measured in turbulent and laminar flow regimes using numerical analysis of experimental data, and the findings are compared to an analytical technique. The turbulent regime, which has the most technical significance, shows the most significant variations. The characterization of continuous reactors' residence time behavior is critical for their design. The residence time distribution of ideal PFRs is quite narrow (RTD). This means that a chemical's residence period in the reactor is precisely determined. A PFR has variations in residence time, described through the RTD function $E(t)$. [9]

CSTR

Short-circuiting, dead zones, and recirculation are

some of the key mixing parameters defining the degree of mixing in a CSTR. The degree of mixing significantly affects the conversion of reagents, as well as the molecular structure of the product. RTD provides an approach to characterize the non-ideal mixing in a reactor, thus allowing the process engineer to understand and analyze better mixing performance of the reactor. The RTD information can be used to design an appropriate reactor model system to reflect the actual mixing behavior in the tank. An appropriate reactor model, with accurate reaction kinetics, can provide accurate predictions of reactor performance and is useful in both process design and optimization. Numerous experimental studies on RTD in continuous stirred tanks have been carried out covering a wide range of tank sizes, impeller designs, baffle designs, and operating conditions. Continuous stirred-tank reactors (CSTR) are still widely used in polymerization processes. Viscosity changes rapidly during polymerization, and in general, a nonideal CSTR behavior is observed.[8]

VI. RTD IN INDUSTRIES

BIO-PROCESSING/BIO TECHNOLOGY

The impact of the hydraulics, the oxygenation, and the substrate composition at the manufacturing of lignin peroxidase (LIP) by *Phanerochaete chrysosporium* immobilized in polyurethane foam in a packed bed bioreactor was studied. The hydrodynamic behavior of the reactors was reformed by selecting various flow recycling ratios. The best results were when the bioreactor operated at plug flow with partial mixing. After deciding the best conditions, lignin peroxidase production in packed bed bioreactors operated at alternating growth-production cycles become accomplished for 25 days acquiring an average activity of 6136 U. The impact of bioreactor hydrodynamics on LIP synthesis by *P. chrysosporium* immobilized on polyurethane foam for successive growth-idiopathic phase cycles were observed in the experiments

corresponding to series of Packed bed bioreactors, were operated at three different recycling ratios. Their hydrodynamics were analyzed by residence time distribution experiments. The obtained data were adjusted to the tanks-in-series model. The bioreactor operated with partial mixing displayed the best performance since it shows the highest productivity, also relatively stable production. The LIP profiles concentration along each reactor closely agreed with the results obtained from the RTD experiments.[1]

Simultaneous bio-adsorption and biodegradation were discovered to be effective in removing fluoride ions from waste by TEJ PRATAP SINGH, JATIN BHATNAGAR, and C.B. MAJUMDER. In the bio-column reactor, bacteria from *Acinetobacter baumannii* were immobilized on the java plum seed. The bed depth service time design model and empty bed residence time were used to assess the bio-performance. In the columns on this simplified bio-column reactor design model, the effect of various operational parameters like flow rate, bed depth, and initial concentration was observed. A desorption experiment was carried out to see if the medium could be regenerated and reused.[2]

Luc De Backer & Gino Baron examined the liquid phase residence time distribution (RTD) in a packed bed bioreactor containing porous glass particles. A model including axially dispersed flow for the external fluid phase and an effective diffusivity that combines diffusion and convection, predicts experimental RTD data satisfactorily. There was immobilization of yeast cells on porous glass. High Biomass loading affected the mass transfer whereas low biomass loading didn't affect the mass transfer rates. Comparison of the RTD data from experiments performed in the presence and absence of cells in the external fluid phase revealed that the mass transfer rate is influenced by the cells immobilized inside the porous particles and not by the cells present in the external fluid phase. The presence of yeast cells only at a high biomass loading influenced

the RTD significantly. Also, at the high liquid flow rates, the decreased mass transfer rates are the strongest. The yeast cells which are immobilized inside the porous particles, and not the cells present in the interstitial spaces of the packed bed, are responsible for an increased intraparticle mass transfer resistance. The same mathematical model can be used for transport in porous particles with or without immobilized cells.[30]

PHARMACEUTICAL

Antoni Sanchez, Francisco Valero*, Javier Lafuente, Carles Sola studied using isooctane as solvent and butanol as esterification agent a study of the enantioselective resolution of ibuprofen by industrial *Rhizomucor miehei* lipase was been carried out The Residence-time distribution (RTD) technique which is a strong tool to observe the behavior of continuous reactors and to analyze possible deviation from ideality. RTD was carried out in the tubular packed reactor by pumping into the reactor a solution of N-butyl propionate, an inert compound, in pulse. This tracer was chosen because of the physical properties that are the same as those of the reacting mixture and it is easy to predict. [6]

MC. Martinetz, A-P. Karttunen, S. Sacher, P. Wahl, J. Ketolainen, J.G. Khinast, O. Korhonen researched that residence time refers to the time spent by a drug in the part of the body where it needs to be absorbed. The more the residence time, the more of it can be absorbed. If the drug is delivered in an oral form and destined for the upper intestines, it usually moves with food and its residence time is roughly that of the food. This generally allows 3 to 8 hours for absorption. If the drug is delivered through a mucous membrane in the mouth, the residence time is short because saliva washes it away. Strategies to increase this residence time include bio adhesive polymers, gums, lozenges, and dry powders.[5] A large class of drugs is enzyme inhibitors that bind to enzymes in the body and inhibit their

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activity. In this case, it is the drug-target residence time that is of interest. More the residence time of drugs more they are desirable because it is effective and hence can be used in lower doses. The residence time is determined by the kinetics of the interaction, such as how complementary the shape and charges of the target and drug are and whether outside solvent molecules are kept out of the binding site and are proportional to the half-life of the chemical dissociation. One way to measure the residence time is in a preincubation-dilution experiment where a target enzyme is incubated with the inhibitor, allowed to approach equilibrium, then rapidly diluted. The amount of product is measured and compared to a control in which no inhibitor is added. [5]

Aparajith Bhaskar1 & Ravendra Singh found the content uniformity of each tablet must be guaranteed before it can be released to the market. This work aimed to develop and analyze a strategy to change the non-confirming tablets in real-time and thereby assure drug concentration of final tablets. RTD-based strategy is proposed to be applied for real-time tablet diversion. For manufacturing equipment, the RTD is a characteristic of the mixing. Typical chemical engineering jargon differentiates the RTD at the definitional stage using a continuous stirred tank reactor (CSTR) and plugged flow reactor (PFR) where the former exhibits a thorough mixing characteristic and the latter introduces a time delay. For a system, the RTD may be derived by conducting tracer experiments. The increased involvement of RTD in the chemical engineering field has also led to the number of models. This work made use of the tank in the series model. In this work, an RTD-based control system was designed and implemented in silico. The developed system's application is directed mainly towards continuous pharmaceutical manufacturing processes where it can facilitate more production efficiency. However, does not restrict its use to a direct compaction continuous pharmaceutical line. It can be adapted and used in any continuous process. The future work includes the

implementation of an RTD-based control strategy into the pilot plant facility.[4]

FERTILIZER INDUSTRY

Abellon et al. studied that the procedure for phosphate treatment is extremely complicated: it has many inlets, and each inlet has its flow rate, which is not very steady. As a result, we chose to inject at the granulated phosphate rock inlet was a more accessible in-site inlet. The Inlet of phosphate to the central unit of the reactor has an open side, which allows us to practice radiotracer injection. Because the flow rate of that inlet fluctuates a lot (sometimes up to 20%), a precise radiotracer injection with the same behavior as the phosphate flow isn't as important in our scenario. The intake, as visible on the right side of Fig. 5, has reinforced concrete thick walls that act as a barrier between the device's radiotracer and the operator's body. Following the fast injection of the radiotracer, data gathering of the RTD curve vs time begins for at least 6 hours. Treatment and interpretation of data Subtracting the background level should be the first step in the RTD curve data handling. Then, if the acquisition was successful, to repair an error that occurred during the reactor's transient-state run, a mathematical interpolation approach should be used. Sections of the RTD curve have been disrupted. When the acquisition time is insufficient to return count rates to an exponential decline with a linear behavior towards zero at the end of the experiment, a mathematical extrapolation of the RTD curve is required. Engineers may use this technique to figure out why phosphate treatment plants aren't performing as they should. as well as to achieve precise flow rate, inventories, and mixing efficiency measurements. For phosphate, however, a chemical reaction can't take place in a vacuum. complete the task to the nth degree. As the % completion rises, the pace of reaction slows until it reaches a plateau. Our long-term aim for radiotracer applications in the phosphatic sector, as well as for radiation safety concerns, is to maximize the

quantity of information collected using radiotracers while using the least amount of radioactivity possible.[31]

OIL AND GAS INDUSTRY

Abellon et al. (1997) conducted research that was utilized to calculate the circulation rate in FCCU. In a cold four-cell environment fluidized bed reactor with interconnecting a bead of glass, MRT was utilized to analyze solid particles. The findings were tracked using the radioisotope ^{24}Na . An IR radiotracer was used on molten glass in such a way that the density of the glass bead did not change. It was ensured that the MRT would not be hampered. The radiotracer's size has an effect. The liquefied bed appeared to be mixed, and static electricity altered the existence of the residence time distribution. Button on the inside of the bed. Catalyst dynamics in fluid catalytic cracking. Two separate refineries' fuel cell control units (FCCUs) were evaluated. Samples of catalysts Instrumental neutrons were used to activate them. analysis of activation twelve distinct components makes up Although the catalyst had been activated, the radioactivity of high levels of lanthanum and sodium was discovered. in comparison to the other components on the page catalyst. It was also discovered that lanthanum had a negative charge. Higher levels of Lantium (^{140}La) and sodium (^{24}Na) were found. Concentration compared to other components The impetus fluid dynamics These samples had been irradiated. Used as a natural radiotracer to determine. In the FCCU, MRT of the catalyst and axial mixing is performed. The radial distribution in the riser and stripper was determined using the radiotracer concentration curve obtained from detectors positioned across the diameter of the column at various axial points. This sort of measurement is only possible using the radiotracer approach. The ADM was used to replicate the experimental results, and the riser had a lot of axial mixing, which was problematic.

Pant et al. (2009) investigated how coal particles migrate in a coal gasifier with a fluidized bed. The radiotracers ^{140}La and ^{198}Au deposited on the surface of coal particles were used as independent tracers. The gamma function model, which is an extension of TISM, is used to simulate. The value of N in the function model is in the gamma range (number of tanks). It's possible to have a fractional value. This model can interpret the little departure from a well-mixed state ($N=1$); however, it is not physically interpretable. In sieves, the radiotracer method was also employed. Using a plate extraction column to calculate the axial distance. In the laboratory, mixing, liquid holdup, and slide velocity are all factors to consider. An ADM with unrestricted access to replicate the situation, an open boundary condition was employed data from an experiment It was discovered that the brief Radiotracers with a half-life of less than a year are great instruments for studying the environment. According to the generated curve, the flow rate of water was faster than crude oil due to friction between the layers of the two fluids. Because water has a larger density than crude oil, it dominates the system.[29]

WASTEWATER TREATMENT

Abellon et al. found RTD testing useful for determining whether the system is nearing any of the targets. Whether or if the behavior is ideal. There are no conventional procedures for evaluation in the literature of continuous-operation systems' mixing performance. There are no conventional methods for evaluating the mixing performance of continuous systems in the literature. As a result, stimulus-response methods are used to assess RTD. A tracer pulse input injected abruptly, or a step input supplied continuously at a steady rate can be utilized as the stimulus. The RTD parameters calculated can be used to investigate flow and mixing parameters in continuously operating systems. The RTD parameters are calculated using the concentration vs. time data from the stimulus-response tests. The closed-vessel boundary condition was used

for pulse tracer studies, starting with mixing-cup measurements. This differs from the open vessel boundary condition, which uses through-the-wall measurements at the outflow to determine the boundary condition. The axial dispersion, mixing condition, and segregated flow are investigated using a dispersion model. For modeling purposes, the computed dispersion coefficient is utilized. The dimensionless group termed dispersion number, which is used to measure axial dispersion was introduced by RTD research. The smaller the dispersion number, the less dispersion, and the possibility of plug flow in the column. The higher the value, the greater the dispersion and the likelihood of a mixed flow. As a result, RTD investigations were used in the current study to determine non-ideal flow behavior it was completed the tests were carried out in a column containing lantana Camara adsorbent utilizing sodium chloride as a tracer at various phenol flow rates. [29]

VII.CONCLUSION

The concept of RTD is spreading through many areas from solid processing used in continuous manufacturing of chemicals, plastics, polymers, food, catalysts, and pharmaceutical products, to hydrodynamic modeling of real. Tracer technology shortens the diagnosis time and provides the data quickly. Tracer technology has many applications outside the traditional chemical engineering and extensions of the traditional RTD have appeared, and still experimental improvements are reported. The recent push of continuous flow chemistry in the pharmaceutical industry and the development of milli- and microreactors renewed the interest in RTD for the characterization of the flow behavior in such devices. The best way to improve the design of the reactor is to understand what is happening internally and RTD has proved to be the most efficient to check the feasibility of the reactor.[10]

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Cite this article as :

Shivani Kaustubh Chitale, Pranjal Nitin Jadhav, Snehal Suresh Dhoble, Dr. Mrs. Sonali Dhokpande, Prashant Ingole, " Study of Residence Time Distribution in Chemical Industry - A Review", *International Journal of Scientific Research in Science and Technology(IJSRST)*, Print ISSN : 2395-6011, Online ISSN : 2395-602X, Volume 9, Issue 1, pp.47-55, January-February-2022. Available at
doi : <https://doi.org/10.32628/IJSRST22919>
Journal URL : <https://ijsrst.com/IJSRST22919>