

# **Study of hydrodynamics and residence time distribution of activated sludge process**

Thesis

Submitted in partial fulfilment for the award of the Degree  
of

*Doctor of Philosophy*

in

**CHEMICAL ENGINEERING**

by

**METALI SARKAR**

**(Registration No. - 951501001)**

*Under the guidance of*

**Dr. Vikas K. Sangal**

Associate Professor

Department of Chemical Engineering,  
Malviya National Institute of Technology,  
Jaipur

**Dr. Haripada Bhunia**

Professor

Department of Chemical Engineering,  
Thapar Institute of Engineering &  
Technology, Patiala



**DEPARTMENT OF CHEMICAL ENGINEERING  
THAPAR INSTITUTE OF ENGINEERING & TECHNOLOGY**

**(Deemed to be University)**

**PATIALA - 147004, PUNJAB (INDIA)**

**[www.thapar.edu](http://www.thapar.edu)**

*August 2019*

*This thesis is dedicated to Almighty God, my gurus  
and to my loving family for all the blessings and  
unyielding support and for being my source of  
strength.*

## CANDIDATE'S DECLARATION

---

I hereby certify that the work which is being presented in the thesis entitled “**STUDY OF HYDRODYNAMICS AND RESIDENCE TIME DISTRIBUTION OF ACTIVATED SLUDGE PROCESS**” in partial fulfilment of the requirements for the award of the degree of Doctor of Philosophy and submitted in the Department of Chemical Engineering of Thapar Institute of Engineering and Technology, Patiala is an authentic record of my own work carried out during a period from January 2016 to August 2019 under the supervision of Dr. V. K. Sangal, Associate Professor, Department of Chemical Engineering, Malaviya National Institute of Technology, Jaipur (*Former Associate Professor, Department of Chemical Engineering, Thapar Institute of Engineering and Technology, Patiala*) and Prof. H. Bhunia, Professor, Department of Chemical Engineering, Thapar Institute of Engineering and Technology, Patiala. The matter presented in the thesis has not been submitted by me for the award of any other degree of this or any other Institute.



**(METALI SARKAR)**

---

This is to certify that the above statement made by the candidate is correct to the best of our knowledge.



**Dr. V. K. Sangal**  
**Supervisor**



**Prof. H. Bhunia**  
**Supervisor**

## CERTIFICATE

---

*This is to be certify that thesis entitled “Study of hydrodynamics and residence time distribution of activated sludge process” being submitted by Ms. Metali Sarkar to the Department of Chemical Engineering, Thapar Institute of Engineering & Technology, Patiala for the award of degree of **Doctor of Philosophy** is a record of bonafide research work carried out by her. Ms. Metali Sarkar has worked under our guidance and supervision. She has fulfilled the requisite standard for the submission of the thesis.*

*To the best of our knowledge, the matter embodied in this thesis are original, and has not been submitted to any other university/institute for the award of any degree or diploma.*



**Vikas K. Sangal**  
Associate Professor  
Department of Chemical Engineering,  
Malaviya National Institute of Technology  
Jaipur



**Haripada Bhunia**  
Professor  
Department of Chemical Engineering,  
Thapar Institute of Engineering & Technology  
Patiala

## ACKNOWLEDGEMENTS

---

*Words are often less to reveal one's deep gratitude. With an understanding that work like this can never be the outcome of a single person. Through this acknowledgment I would like to express my special thanks, gratitude and regards to all those who supported, helped and guided me in completing this work.*

*At first, my heartfelt thanks to the **ALMIGHTY** for his abundant blessing showered on me throughout this endeavour to complete this successful work of mine.*

*I express my deep and sincere gratitude as well as profound regards to **Dr. Vikas K. Sangal** and **Dr. Haripada Bhunia** for providing me an opportunity to work under their guidance. Their wide knowledge, expertise, valuable suggestions, encouragement and freedom of independent work provided a platform for learning and performing research. Their enthusiasm and optimism made this experience both rewarding and enjoyable. Their feedback and editorial comments were also valuable for the writing of this thesis.*

*I would like to thank my doctoral committee members **Dr. Amit Dhir, Dr. R.K. Gupta and Dr. S.K. Singh** for their guidance and support through the duration of this work. I would like to thank **all the faculty members** of the Department of Chemical Engineering for encouraging me to write the thesis report. I would like to thank **all the employees** of the Department of Chemical Engineering for their everlasting support.*

*I would express my sincere gratitude to **Dr. P. K. Bajpai**, Ex-Distinguished Professor, for providing guidance and support for successfully conducting this work.*

*I am extremely thankful to **Dr. H. J. Pant** and his team members **Dr. V. K. Sharma** **Mr. Sunil Goswami** and **Jitendra Samantray** for providing radiotracer and support during industrial experiments.*

*I sincerely thank the **DAE-BRNS** and **Thapar Institute of Engineering and Technology** for providing financial support and conducive environment to undertake the research work.*

*I would like to thank **Mr. Anil Kumar, Dr. Anil Nathani, Ms. Pritha Bhattacharya** and all other industry personnel for their support in carrying out industrial scale experiments at M/s Shreyans Industries Ltd., Ahmedgarh, Punjab, India. I am also grateful to the industry personnel at Trident Industries Ltd., Barnala, Punjab, India for their support in carrying out my experimental work.*

*I express my deep sense of gratitude to my family. I am thankful to my parents (**Mr. R.M. Sarkar, Mrs. Tapashi Sarkar**) and my brother (**Mr. Palash Sarkar**) for their great support throughout my life. Words cannot express how grateful I am to my parents or all of the sacrifices that they have made on my behalf. Their prayers for me were what sustained me thus far. I would cherish every moment where my parents were so keen and curious to know about the details and progress of my work, which boosted my confidence.*

*I would like to thank all my colleagues and friends (**Ms. Meenakshi Sheoran Nandal, Ms. Steffi Talwar, Ms. Parminder Kaur, Ms. Sehaspreet Kaur, Ms. Bandhana Sharma, Ms. Ravneet Kaur, Dr. Dev Kumar Mandal, and Mr. Kamaljeet Singh**) for their support and encouragement in carrying out my research smoothly. I would also like to thank my close friends (**Ms. Sitakshi Pal and Mr. Chatendra Kumar**) for constant motivation to finish my work and **Mr. Mohit Dayal** for believing in me and continuous emotional and mental support.*

**Patiala**

**August, 2019**

**Metali Sarkar**

## ABSTRACT

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Water is an important commodity for manufacturing and process Industries. The enormous amount of fresh water used by these Industries generates tons of wastewater that not only pollutes the environment, but also causes irreversible damage to the ecological system. Industries require highly efficient and competent water treatment for responsible handling and management of the effluent generated.

Activated sludge process (ASP) is widely used for treatment of wastewater produced by process industries, especially in pulp and paper industry. The hydraulic performance of ASP is an essential factor as it can directly affect the wastewater treatment efficiency in the reactor. The hydrodynamics of wastewater treatment reactors can be conveniently studied by residence time distribution (RTD) technique. RTD study was carried out on an industrial scale ASP system of an effluent treatment plant in an integrated pulp and paper industry using radiotracer Iodine-131. The system consisted of two stage aeration tank and a secondary clarifier connected in series. The primary objective of the investigation was to measure actual mean hydraulic retention times (MHRTs) and analyze the hydraulic performance of the complete ASP and individual system (aeration tank-1, secondary clarifier-1, aeration tank-2 and a secondary clarifier-2). Two sets RTD experiments were performed on the industrial scale ASP. The measured RTD data was treated to remove experimental errors and noises. Several treatment methods like dead-time correction, background correction, radioactive decay correction and tail correction were applied to the raw data before it can be used for further assessment. The experimental MHRTs were estimated for all the reactors. The analysis of RTD curves indicated small fraction of bypassing stream (3%) in the aeration tank-1. The dead volume in the aeration tank-1 and the secondary clarifier-1 was found and estimated to be 2.34% and 4.6%, respectively. In order to obtain detailed information about flow structure of wastewater within the aeration, secondary clarifier and complete ASP, the measured RTD data was modeled using suitable and representative mathematical models. The modeling of the measured RTD data of the aeration tank-1 revealed that the hydraulic behavior of the aeration tank-1 could be represented by two CSTRs with a moderate degree of the back-mixing between

them. In the secondary clarifier-1, it was also found to be operating normally without and significant malfunctioning. A simple axial dispersion model was found suitable to describe the flow behavior of the secondary clarifier-1. Based on the prior information and results of the modeling of the individual systems, a compartment model was proposed for the entire activated sludge processing system. The compartment model was a combination of tank-in-series with back-mixing component connected in series with an axial dispersion model component along with a recycle line, which was a well representation of the system. Convolution method was applied to model the system with an imperfect impulse radiotracer input to aeration tank-2 and secondary clarifier-2. The treated RTD curves were further simulated using suitable hydraulically representative mathematical models and detailed flow patterns in the reactors were deciphered. The aeration tank-2 was fitted with tank-in-series model and was found that the reactor was working approximately as a complete mixing tank. The aeration tank-2 was working efficiently in the absence of any dead zones or bypassing. The secondary clarifier-2 was simulated with a simple axial dispersion model, with Peclet number equals to 10, which signifies that the clarifier was acting precisely as a plug flow reactor.

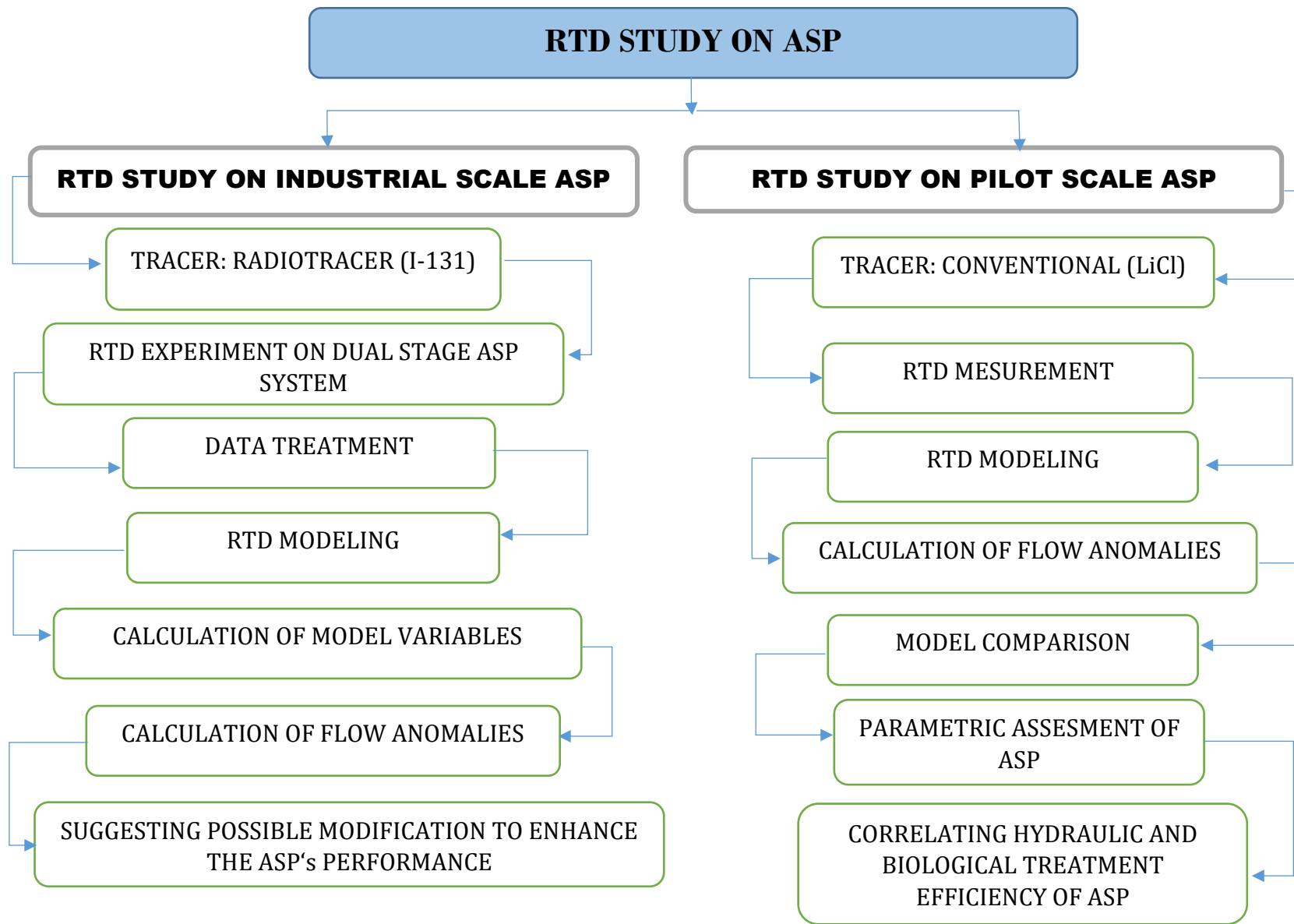
It was impractical to change the operating parameters in a full-scale industrial system. Hence, a pilot scale ASP was constructed to study the effect of MHRT and sludge recycle ratio, on the hydrodynamic efficiency and biological treatment efficiency of the system. RTD study of the pilot scale ASP treating the pulp & paper mill effluent has been performed using LiCl as tracer. The hydraulic performance and treatment efficiency of the aeration tank and ASP at different operating parameters like residence time, recycle rate was investigated. The residence time of aeration tank was varied at 14 h, 16 h, 20 h and 24 h and operated with no sludge recycle. For each set of MHRT the sludge recycle rate from the bottom of the clarifier to the aeration tank was adjusted to 10% and 20% of the main flow and RTD experiment was performed to examine the effect of sludge recycling on the hydrodynamics of the reactor. The flow anomalies were identified and based on the experimental data empirical models were suggested to interpret the hydrodynamics of the reactors using compartment modeling technique. The analysis of the RTD curves and the compartment models indicated bypassing stream for aeration tank operating at 14 h MHRT, but disappeared at higher MHRTs. Increase in back-mixing ratio was observed as the MHRT of the tank was increased. The model

proposed for the aeration tank consisted of tank-in-series with back-mixing component along with recycle line or bypassing line. Based on the modeling of aeration tank, hydrodynamic model was proposed for ASP and the model block consisted of the tank-in-series with back-mixing model component connected in series with axial dispersion model component. The maximum fraction of dead zone estimated in the complete ASP was approximately 7%. It was found that the dead zone fraction increased by approximate 20 - 25% with increase in recycle rate. The fraction of the stagnant zone was found well below experimental limits for all performed experiments. The wastewater treatment parameters were also evaluated. The substrate removal of 91% for COD and 96% for BOD were observed for the ASP working at a hydraulic mean hydraulic residence time 39 hr MHRT with a 20% recycling of activated sludge, which was well below the discharge limit set by the pollution control board.

The results of the investigation shows that tracer technique is suitable for evaluating performance of complex ASP. The hydrodynamic study can provide valuable information to assess the actual behaviour of the reactors and the extent of deviation from the expected behaviour. The study can also help industries to take corrective measures to improve reactor performance to achieve optimum operation and maximum treatment efficiency of the effluent treatment plant.

Figure 1 shows the schematic of the overall thesis work.





**Figure 1** The schematic of the overall thesis work.

# TABLE OF CONTENTS

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CANDIDATE’S DECLARATION	iii
CERTIFICATE	iv
ACKNOWLEDGEMENTS	v
ABSTRACT	vi
LIST OF CONTENTS	xi
LIST OF FIGURES	viii
LIST OF TABLES	xv
NOMENCLATURE AND ABBREVIATIONS	xviii
<b>Chapter 1 – Introduction</b>	<b>1</b>
1.1 Effluent treatment plant	1
1.1.1 Activated Sludge Process	4
1.1.1.1 Aeration Tank	4
1.1.1.2 Secondary Clarifier	5
1.1.2 Operational problems associated with ETP	6
1.2 Hydrodynamics study of ASP	6
1.3 Residence Time Distribution Technique	7
1.3.1 Conventional tracers	8
1.3.2 Radiotracers	9
1.3.3 Advantages of Radiotracers	10
1.4 RTD modelling	12
1.5 Thesis motivation and objectives	14
1.6 Thesis overview	16
<b>Chapter 2 – Literature Review</b>	<b>18</b>
2.1 RTD study in industrial WWTP	18
2.1.1 RTD study on industrial scale WWTP with conventional tracer	18
2.1.2 RTD study on industrial scale WWTP with radiotracer	22
2.1.2.1 RTD study in industrial primary wastewater treatment processes	22

---

2.1.2.2 RTD study in industrial secondary wastewater treatment processes	23
2.1.2.3 RTD study in industrial scale sludge treatment unit	27
2.2 RTD study on pilot scale WWTP	28
2.2.1 RTD study on pilot scale WWTP using conventional tracer	29
2.2.2 RTD study on pilot scale WWTP using Radiotracer	31
2.3 RTD modeling	31
<b>Chapter 3 – Material and Methodology</b>	<b>37</b>
3.1 Materials	37
3.1.1 Wastewater	37
3.1.2 Chemicals	37
3.1.3 Radiotracer experiment on industrial scale WWTP	38
3.2 Equipment	37
3.2.1 Industrial scale activated sludge process	38
3.2.2 Detectors	44
3.2.3 Data Accusation System	44
3.2.4 Pilot scale ASP design	44
3.3 Methodology	47
3.3.1 Radiotracer Detection and measurement in industrial scale ASP	47
3.3.2 RTD Formulation and data correction	48
3.3.2.1 Dead time correction	48
3.3.2.2 Background correction	48
3.3.2.3 Radioactive decay correction	48
3.3.2.4 Filtering (or smoothing)	48
3.3.2.5 Tail Correction	49
3.3.3 Pilot scale ASP study	49
3.3.3.1 ASP start up	49
3.3.3.2 Microorganism growth and acclimatization	49
3.3.3.3 Analytical methods for parametric assessment	50
3.3.3.4 RTD tracer experiment of pilot scale ASP	50

---

3.4 RTD system analysis	51
3.4.1 Calculation of RTD moments	51
3.4.2 RTD modeling	52
3.4.2.1 Tank in series (TIS) model	53
3.4.2.2 Tank-in-series with back-mixing model (TISBM)	54
3.4.2.3 Axial dispersion model (ADM)	54
3.4.2.4 Compartment model	55
3.5 Convolution Technique	55
3.5.1 Benefits of convolution technique for non-ideal tracer input	55
3.5.2 Convolution integral	56
3.5.3 Estimation of parameters	57
3.6 Optimization procedure — Curve fitting method	57
3.7 RTD Software	57
<b>Chapter 4- Industrial Scale RTD Experiments and Modelling</b>	<b>59</b>
4.1 RTD Experiment on the Industrial scale wastewater treatment plant	57
4.1.1 Modification and refining of data obtained from radiotracer experiments	57
4.1.1.1 Pre-treatment of experimental data	61
4.1.1.2 Calculation of Moments	66
4.2 RTD modeling	65
4.2.1 Diagnosis and modeling of RTD data for Aeration Tank -1	66
4.2.2 Modeling of Secondary clarifier-1	70
4.2.3 Modeling of complete Activated sludge process	72
4.3 Modeling non-ideal tracer input	74
4.3.1 Modeling of Aeration Tank-2	74
4.3.2 Modeling of Secondary Clarifier-2	77
<b>Chapter 5- Pilot scale RTD Experiment and Modeling</b>	<b>80</b>
5.1 Design objectives of pilot scale ASP	80
5.2 Microorganism growth and acclimatization	80
5.12.1 Change in pH	81

---

5.2.2 Change in average MLSS concentration	82
5.2.3 Change in Chemical Oxygen Demand (COD)	83
5.3 RTD study of pilot scale ASP	84
5.3.1 Calculation of Moments	85
5.4 RTD Modeling	87
5.4.1 Modeling of pilot scale Aeration tank at varying MHRT	87
5.4.2 Modeling of pilot scale ASP at varying MHRT	91
5.4.3 Modeling of Aeration Tank at varying MHRT with sludge recirculation	94
5.4.4 Modeling of complete ASP at varying MHRT with sludge recirculation	100
5.5 Treated effluent-pollution parameters	107
<b>Chapter 6 - Conclusions and Recommendations for Future Work</b>	<b>114</b>
6.1 Conclusions	114
6.1.1 RTD study on Industrial scale ASP	114
6.1.2 RTD study on Pilot scale ASP	115
6.2 Recommendations for future work	117
<b>References</b>	<b>118</b>
<b>List of Publications</b>	
<b>Reprints of Published Articles</b>	

## LIST OF FIGURES

Figure No.	Title	Page No.
Figure 1	The schematic of the overall thesis work.	x
Figure 1.1	General layout of a typical ETP	2
Figure 1.2	General tracer experiment principle	8
Figure 2.1	E(t) curve revealing bypassing and dead-zones in the tank	33
Figure 3.1	Schematic diagram (front view) of the complete industrial ASP setup at Shreyans Industries	38
Figure 3.2	Industrial scale aeration tank-1	39
Figure 3.3	Industrial scale secondary clarifier-1	40
Figure 3.4	Industrial scale aeration tank-2	41
Figure 3.5	Industrial scale secondary clarifier -2	42
Figure 3.6	Schematic diagram of pilot scale ASP	45
Figure 3.7	Pilot scale ASP system	45
Figure 3.8	A generic procedure of RTD modelling	53
Figure 3.9	Interference with input signal due to a non-ideal impulse input	56
Figure 3.8	Schematic diagram of pilot scale ASP	54
Figure 4.1	Schematic diagram (front view) of the complete industrial scale secondary wastewater treatment system	60
Figure 4.2 (a)	Radiotracer signal obtained from aeration tank-1	61
Figure 4.2 (b)	Corrected radiotracer signal obtained from aeration tank-1	61
Figure 4.2 (c)	Radiotracer signal obtained from secondary clarifier-1	62
Figure 4.2 (d)	Corrected radiotracer signal obtained from secondary clarifier-1	62
Figure 4.2 (e)	Radiotracer signal obtained from aeration tank-2	63
Figure 4.2 (f)	Corrected radiotracer signal obtained from aeration tank-2	63
Figure 4.2 (g)	Radiotracer signal obtained from secondary clarifier-2	64
Figure 4.2 (h)	Corrected radiotracer signal obtained from secondary clarifier-2	64
Figure 4.3	Schematic diagram (front view) of the complete industrial ASP setup	63

---

Figure 4.3	Schematic diagram (front view) for first radiotracer experiment (Run-1) in ASP System	63
Figure 4.3	Conceptual physical model representation of aeration tank-1	64
Figure 4.3	Schematic diagram (front view) for first radiotracer experiment (Run-1) in ASP System	64
Figure 4.4	Conceptual physical model representation of aeration tank-1	65
Figure 4.5	Comparison of experimental (D5) and model simulated RTD curves at the outlet of the aeration tank-1 (Run 1)	66
Figure 4.6	Comparison of experimentally measured and model simulated RTDs at location D2 within the aeration tank	67
Figure 4.7	Comparison of experimentally measured and model simulated RTDs at location D3 within the aeration tank	67
Figure 4.8	Conceptual physical model for the secondary clarifier-1	68
Figure 4.9	Experimental and model simulated RTD curves at the outlet of the secondary clarifier-1 (Run 2)	69
Figure 4.10	Phenomenological model for the entire activated sludge processing system	71
Figure 4.11	Experimental (D6) and model simulated RTD curves at the outlet of the ASP system (Run1)	72
Figure 4.12	Schematic diagram of system reported for non-ideal impulse input	75
Figure 4.13	RTD curve monitored at the outlet of aeration tank-2 (D9) and model adjustment by convolution	77
Figure 4.14	Comparison of RTD models for aeration tank-2	77
Figure 4.15	RTD curve monitored at the outlet of secondary clarifier-2 (D10) and model adjustment by convolution	79
Figure 4.16	Comparison of different models and their parameters for aeration tank and secondary clarifier	79
Figure 5.1	The pH change of wastewater during the acclimatization process	82
Figure 5.2	The change in average MLSS during the acclimatization process.	83

---

Figure 5.3	The substrate degradation (COD) during the acclimatization process	84
Figure 5.4	Experimental setup of pilot scale ASP	85
Figure 5.5 (a)	The RTD curves obtained at different MHRT for aeration tank	87
Figure 5.5 (b)	The RTD curves obtained at different MHRT for ASP	87
Figure 5.6	Compartment model for the hydraulic representation of aeration tank at varying MHRT without recycle	88
Figure 5.7 (a)	Comparison of experimentally measured and model-simulated RTD curves at the outlet of the aeration tank for MHRT 24 h	90
Figure 5.7 (b)	Comparison of experimentally measured and model-simulated RTD curves at the outlet of the aeration tank for MHRT 20h	90
Figure 5.7 (c)	Comparison of experimentally measured and model-simulated RTD curves at the outlet of the aeration tank for MHRT 16 h	91
Figure 5.7 (d)	Comparison of experimentally measured and model-simulated RTD curves at the outlet of the aeration tank for MHRT 14 h	91
Figure 5.8	Compartment model for the hydraulic representation of ASP at varying MHRT without recycle	92
Figure 5.9 (a)	Comparison of experimentally measured and model-simulated RTD curves at the outlet of the ASP MHRT 39 h	93
Figure 5.9 (b)	Comparison of experimentally measured and model-simulated RTD curves at the outlet of the ASP MHRT 33.5 h	93
Figure 5.9 (c)	Comparison of experimentally measured and model-simulated RTD curves at the outlet of the ASP MHRT 26.8 h	94
Figure 5.9 (d)	Comparison of experimentally measured and model-simulated RTD curves at the outlet of the ASP MHRT 21.5 h	94
Figure 5.10	Compartment model for the hydraulic representation of Aeration Tank with recycle	95
Figure 5.11 (a)	Comparison of experimentally measured and model-simulated RTD curves aeration tank for 10 % sludge recycle and MHRT 24 h	97
Figure 5.11 (b)	Comparison of experimentally measured and model-simulated RTD	97

---

	curves aeration tank for 10 % sludge recycle and MHRT 20 h	
Figure 5.11 (c)	Comparison of experimentally measured and model-simulated RTD curves aeration tank for 10 % sludge recycle and MHRT 16 h	98
Figure 5.11 (d)	Comparison of experimentally measured and model-simulated RTD curves aeration tank for 10 % sludge recycle and MHRT 14 h	98
Figure 5.12 (a)	Comparison of experimentally measured and model-simulated RTD curves aeration tank for 20 % sludge recycle and MHRT 24 h	99
Figure 5.12 (b)	Comparison of experimentally measured and model-simulated RTD curves aeration tank for 20 % sludge recycle and MHRT 20 h	99
Figure 5.12 (c)	Comparison of experimentally measured and model-simulated RTD curves aeration tank for 20 % sludge recycle and MHRT 16	100
Figure 5.12 (d)	Comparison of experimentally measured and model-simulated RTD curves aeration tank for 20 % sludge recycle and MHRT 14 h	100
Figure 5.13	Compartment model for the hydraulic representation of ASP with recycle	101
Figure 5.14 (a)	Comparison of experimentally measured and model-simulated RTD curves at the outlet of the ASP for 10% recycle and MHRT 39 h	102
Figure 5.14 (b)	Comparison of experimentally measured and model-simulated RTD curves at the outlet of the ASP for 10% recycle and MHRT 33.5 h	102
Figure 5.14 (c)	Comparison of experimentally measured and model-simulated RTD curves at the outlet of the ASP for 10% recycle and MHRT 26.8 h	103
Figure 5.14 (d)	Comparison of experimentally measured and model-simulated RTD curves at the outlet of the ASP for 10% recycle and MHRT 21.5 h	103
Figure 5.15 (a)	Comparison of experimentally measured and model-simulated RTD curves at the outlet of the ASP for 20 % recycle and MHRT 39 h	104
Figure 5.15 (b)	Comparison of experimentally measured and model-simulated RTD curves at the outlet of the ASP for 20 % recycle and MHRT 33.5 h	104
Figure 5.15 (c)	Comparison of experimentally measured and model-simulated RTD curves at the outlet of the ASP for 20 % recycle and MHRT 26.8 h	105

Figure 5.15 (d)	Comparison of experimentally measured and model-simulated RTD curves at the outlet of the ASP for 20 % recycle and MHRT 21.5 h	105
Figure 5.16	Degradation of BOD for aeration tank	109
Figure 5.17	Degradation of COD for aeration tank	109
Figure 5.18	Degradation of BOD for ASP	110
Figure 5.19	Degradation of COD for ASP	110

## LIST OF TABLES

---

<b>Table No.</b>	<b>Title</b>	<b>Page No.</b>
Table 1.1	Conventional tracer and their application on WWTP	10
Table 1.2	Commonly used radiotracer for various applications	11
Table 1.3	Effluent discharge standards for pulp and paper industry	15
Table 2.1	Summary of literature review on RTD studies of WWTP units with conventional tracer	21
Table 2.2	Summary of literature review on radiotracer studies in primary WWT unit	24
Table 2.3	Summary of literature on radiotracer studies in secondary WWT unit	24
Table 2.4	Summary of the literature on pilot scale WWT reactor with conventional tracer	27
Table 2.5	Summary of the literature on pilot scale WWT reactor with conventional tracer	30
Table 2.6	Frequently used RTD models in the literature	35
Table 3.1	Properties of the radioisotope	37
Table 3.2	Dimensional and process parameters of the ASP system at Shreyans Industries Ltd.	44
Table 4.1	Experimental and theoretical mean hydraulic retention times (MHRT)	66
Table 4.2	Comparison of different models and their parameters for aeration tank and secondary clarifier	80
Table 5.1	Characteristics of the influent of treatment systems	81
Table 5.2	Parameters of Aeration tank at different MHRT and sludge recycle rate	106
Table 5.3	Parameters of ASP at different MHRT and sludge recycle rate	107
Table 5.4	Concentration of BOD and COD obtained at varying MHRT and recycle rate	111
Table 5.5	Design parameters of ASP	113

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## NOMENCLATURE AND ABBREVIATIONS

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$C$	Concentration of the tracer
$E$	Exit age distribution of the models
$Pe$	Peclet number
$Q$	Volumetric flow rate
$t$	Time
$V$	Volume
$V_d$	Dead volume
$\tau_{TISBM}$	Mean residence time for tank-in-series with back-mixing model
$\alpha$	Back-mixing ratio
$\tau$	Experimental mean residence time
$D$	Dispersion coefficient ( $m^2/s$ )
$N$	Number of tanks
$Pe$	Peclet number
$T_{1/2}$	Half life time
$Q_0$	Flow rate of wastewater into the system

$Q_R$       Recycle flow

*Greek letters*

$\alpha$       Back-mixing ratio

$\tau$       Mean residence time

$\tau_{pf}$       MRT for plug flow component

$\tau_{ADM}$       MRT for axial dispersion component

$\tau_{TIBSM}$       MRT for tank-in-series with back-mixing component

$\tau_R$       Recycle time

$\gamma$       Energy (keV)

$\sigma^2$       Variance

$i_d$       Dead space index

$i_b$       Flow bypass index

**LIST OF ABBREVIATIONS**

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ADM	Axial dispersion model
AR	Analytical Reagent
AT	Aeration Tank
ASP	Activated Sludge Process
BOD	Biochemical Oxygen Demand
BRIT	Board of Radiation and Isotope Technology
COD	Chemical Oxygen Demand
CFD	Computational Fluid Dynamics
DAS	Data acquisition system
DO	Dissolved Oxygen
DAP	Di-ammonium Phosphate
ETP	Effluent Treatment Plant
MRT	Mean Residence Time
MO	Microorganisms
MLSS	Mixed Culture Suspended Solids
MHRT	Mean residence time (min)
PF	Plug flow component
PFR	Plug flow reactor
RTD	Residence time distribution

RPT	Radiotracer particle tracing
RMS	Root mean square
SS	Suspended Solids
TIBM	Tank-in-series with back-mixing model
TIS	Tank-in-series model
TDS	Total dissolved solids
TSS	Total suspended solids
WWTP	Wastewater treatment reactor
WWT	Wastewater treatment

## **INTRODUCTION**

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Water is the core of our existence, from food security, economic growth, energy requirement to human and environmental sustainability, the contribution of water is enormous. Water is a renewable resource, but in the past few decades, several factors like climatic change and excessive carefree use have made it a valuable commodity. The competition for water consumption among water users is affecting economies and human well-being globally (Connor, 2015). Due to the ever-increasing population and urbanization, the accessibility of fresh water has become highly limited. A large percentage of world's population live in areas of water scarcity, and in many parts of the world, people are unable to sustain basic water needs for the people and environment (Cooley *et al.*, 2013).

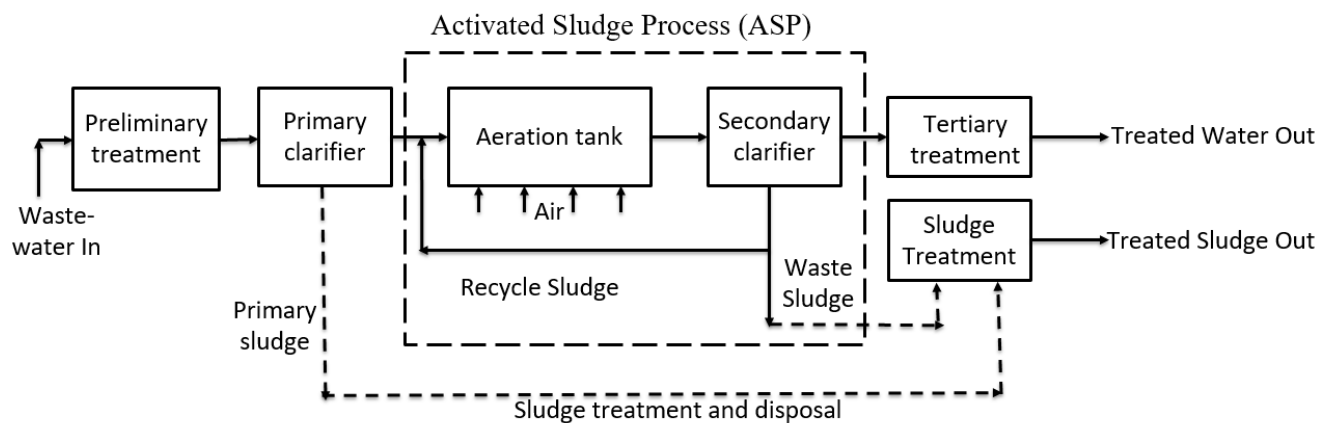
India is a developing country and with the high pace of industrial development, the demand for water in process industries is also increasing, making it an utmost necessity for its optimal use. A significant amount of water is discharged as effluent by industries, which is unacceptable for further use until it is treated to specific standards. According to the World Development Report of 2016 (Water, 2016), 4% of global water is being withdrawn by industries and manufacturing process plants, which will increase to a whopping 400% by 2050. Also, large portions of industrial wastes are dumped into water sources without treatment, hence polluting the usable water supply (Bhushan, 2004; Agarwal and Kumar, 2012).

### **1.1 Effluent Treatment Plant**

Effluent treatment plant (ETP) is a series of unit processes designed to treat the wastewater generated by industries (Figure 1.1). The fundamental objective of a typical ETP is to treat the wastewater produced from the industries to the environmental standards set by the governing bodies, so that it can be safely castoff to the ecosystem or recycled and reused, and contribute in the sustainable development and safeguard of the environment.

Depending on the type of effluent generated by the industry, a combination of treatment processes are applied. These treatment process generally includes a preliminary treatment unit

primary treatment unit, primary clarification, succeeded by secondary treatment, tertiary treatment, and sludge treatment unit.



**Figure 1.1 General layout of a typical ETP**

Preliminary treatment is the foremost step in ETP. It removes the pollutant that is easily visible to the naked eye and collected from the raw wastewater and disposed of by simple physical and mechanical methods. The typical pollutants that are removed during preliminary treatment are floating coarse and fine solid waste by screening, grit removal and equalization of flow in the system (Kamali and Khodaparast, 2015). The preliminary treatment process is followed by the primary treatment unit where the suspended solids that couldn't be separated in the preliminary treatment step are removed. This is achieved in a settling tank often termed as the primary clarifier or a primary sedimentation tank. The suspended solids in the effluent are settled in the primary clarifier by the action of gravity. To speed up the settling process and achieve maximum separation efficiency organic coagulants like polyamines, polydadmacs, melamine-formaldehyde, tannins, and inorganic coagulants as aluminum sulfate (alum), aluminum chloride, ferric chloride, etc. are often used (Wang *et al.*, 2011). A skimming mechanism in the settler unit is installed to skim off oil and grease from the surface. The solids settle at the bottom of the clarifier as sludge which is scraped off by means of a sludge scraper mechanism and transported to the sludge treatment unit for further treatment and disposal. The overflow from the primary settling unit may still consist of a high concentration of organic waste that is treated in the secondary treatment unit (Trinh and Kang, 2011). The effluent

treated in the primary clarifier can't eliminate the soluble and colloidal organics present in the effluent. The secondary treatment unit removes the untreated suspended solids that remain after the primary treatment unit and produce effluent that complies according to the environmental discharge standards for dissolved and colloidal organic fractions in the wastewater. In this unit, the treatment is carried out with the help of active microorganisms that degrade the organic content of the effluent into settleable biomass and simple by-products like carbon dioxide and water. The secondary treatment process can be largely categorized into attached growth process and suspended growth process. The attached growth process employs micro-organisms for the conversion of organic matters in wastewater to biomass. The microorganisms are attached to an inert and porous medium such as crushed rocks or stones or some specifically designed polymer materials. The efficiency of the process depends on the area of inert material available for the growth of microorganism. Some of the examples of attached growth processes widely used in industries are contact beds, intermittent filter, trickling filter, rotating biological contractors, etc. The suspended growth process is a secondary biological treatment process in which the microorganisms responsible for the decomposition of organic matters are kept in a suspended state with the help of aerators. In addition to supplying excess air required to sustain the aerobic conditions in the aeration tank for the microbial degradation process, the aerators also provide adequate mixing to maintain homogeneity in the reactor. Examples of the suspended growth process are activated sludge process, aerated lagoon, oxidation ponds and other sludge digestion system (Tchobanoglous *et al.*, 2003; Pokhrel and Viraraghavan, 2004). The tertiary treatments are additional treatment methods employed to improve the quality of treated water when the treated water obtained from the secondary treatment unit doesn't meet the required discharge standards. The advanced treatment methods employed in tertiary treatment often includes removal of excess phosphorus and nitrogen removal, dissolved inorganic salts, toxic chemicals or pathogens (Chevalier *et al.*, 2000; Zhang *et al.*, 2008; Gupta *et al.*, 2012). The other tertiary treatment processes include filtration, photo-Fenton method (Lima *et al.*, 2018), advanced electrochemical method (Brillas *et al.*, 2019), photo-catalysis individually or in a combination of multiple processes (Koivunen *et al.*, 2003; Zhang *et al.*, 2008; Klammerth *et al.*, 2010; Bernabeu *et al.*, 2011). The primary and secondary clarifier unit

produces a large volume of sludge that needs proper management and disposal. The sludge collected at the bottom of the clarifiers is scraped and transported to the sludge treatment unit. The constituents of the sludge have fertilizing characteristics and if handled wisely can prove an excellent substitute for chemical fertilizers in agricultural needs (Fernandes *et al.*, 2007). This unit of the wastewater treatment plant is responsible for sludge thickening, stabilization, dewatering, disinfection, and final disposal.

### **1.1.1 Activated Sludge Process**

The activated sludge process (ASP) is one of the most primitive secondary wastewater treatment methods. ASP was developed in early 1900, remains one of the most widely used and efficient processes for biological treatment of wastewater produced in the industries (Owens, 1991; Tchobanoglous *et al.*, 2003; Sonune and Ghate, 2004; Schraa *et al.*, 2016). The process involves mixing and aeration of suspended microbe culture in a tank to maintain the aerobic conditions, to breakdown the dissolved organic pollutants in the effluent wastewater into biomass. The biomass formed is separated from the treated effluent in a secondary clarifier (Bajpai, 2001; Tchobanoglous *et al.*, 2003; Peavey *et al.*, 2013). The ASP consist of an aeration tank and a secondary clarifier that are elaborated in the subsequent sections.

#### **1.1.1.1 Aeration Tank**

The key process of ASP is the aeration tank that acts as a biological reactor and converts the dissolved and colloidal organic pollutants in the effluent wastewater into biomass at aerobic conditions. The treated wastewater from the overflow of the primary clarifier is sent to the aeration tank. The aeration tank is a bioreactor that essentially functions as a continuous mixing tank (Lone *et al.*, 2019). The mixed population of microorganisms kept suspended in the aeration tank facilitates the treatment of the dissolved organic waste present in the influent wastewater. The microbes convert carbon into biomass and simple products like carbon dioxide and water at aerobic conditions. The rate at which oxygen is consumed by the microorganism in the biological reactor is called the oxygen utilization rate. The excess oxygen supply is required to maintain the aerobic conditions in the tank as the oxygen utilization rate in the activated -sludge processes will always exceed the rate of natural replenishment. Thus,

some artificial means of adding oxygen must be used. The equipment used to provide excess aeration required for the biological process also provides mixing to keep the microorganism in a suspended state for more effective treatment. The two basic methods employed for aerating wastewater are through mechanical surface aeration systems and submerged diffusers. The submerged air diffusers can be further classified based on the size of the air bubble into a fine bubble and coarse bubble diffusers. The Fine - bubble diffusers produce bubbles of approximately 2.0 to 2.5 mm in diameter and are more efficient due to the larger surface area per volume of air provided by them. However, head loss through the pores demands greater compression of the air and thus greater energy requirement. Also, compressed air pumped through fine bubble diffusers also requires to remove all particulates matter as they can plug the tiny diffuser outlets (Tchobanoglous *et al.*, 2003). Coarse -bubble diffuses inject of larger size bubbles (up to 25 mm diameter). They offer less maintenance and less head loss, but poorer oxygen transfer efficiencies. Pure oxygen may be used for high-strength waste-water treatment. Also, stage-wise operation with pure oxygen has been found to be a very effective method of waste-water treatment (Thompson *et al.*, 2001). Mechanical aerators are another available option to supply excess air in the aeration tank. They produce turbulence at the air-liquid interface, and this turbulence entrains air into the liquid. Surface aerators consist of partially submerged impellers attached to motors mounted on fixed structures. Surface aerators cause turbulence on the liquid surface and create rapid changes at the air-water interface to enhance oxygen transfer. They also enhance the local and overall mixing efficiency, hence maintain the homogeneity in the aeration tank (Bode and Seyfried, 1985).

### **1.1.1.2 Secondary Clarifier**

The secondary clarifier follows the aeration unit that acts as a settling tank. The wastewater with suspended biomass from the aeration tank is directed to secondary clarifiers for sediment removal. The sedimentation process is carried out in rectangular or circular clarifiers. The biomass settles at the bottom of the secondary clarifiers. The secondary clarifier must not only produce an effluent of acceptable quality but must also produce sludge of sufficiently high solids concentration. To maintain the active population of micro-organisms in the aeration tank, a pre-calculated amount of biomass collected at the bottom of the clarifier is

recycled back to the aerator. The clarifier, therefore, is a very important treatment unit. The flow structure of wastewater in a secondary clarifier depends largely on the geometry of the clarifier, location of baffle constructions of wastewater inlet and outlet lines and mechanical movement of the scraper.

### **1.1.2 Operational problems associated with ETP**

These full-scale industrial water treatment systems are often unproductive and uneconomical due to irregularities present in the system. The irregularities may be caused due to various factors like poor design of reactor, inefficient mixing, advection or dispersion. These factors lead to large dead spaces, bypassing streams and internal recirculation, which highly affects the treatment efficiency of the ASP (Tchobanoglous *et al.*, 2003). The primary treatment process includes the equalization tanks that are positioned upstream of the secondary treatment systems and serves the purpose of equalizing chemical composition and volumetric flow rate of wastewater in successive treatment reactors. The presence of irregular mixing zones, dead zones and bypassing stream can lead to instability of microorganism growth in biological treatment units. The performance of various designs of primary clarifiers operating on a variety of operating conditions can affect its efficiency. The accumulation of excessive solids at the bottom of the tank may cause hindrance to flow of wastewater to the following treatment reactors. The aeration tanks are an imperative section of biological treatment units. The aeration requirements vary depending on the strength of the wastewater and cell concentration. Oxygen requirements for typical aeration tanks are about 30 to 60 m<sup>3</sup> O<sub>2</sub>/kg of BOD removed (Lemoullec *et al.*, 2008). The aeration tank used in an ASP consumes a major portion of the total energy required to operate a wastewater treatment plant (WWTP) in a pulp and paper industry resulting in high operating cost (Rieger *et al.*, 2006; Karpinska and Bridgeman, 2016). Hence the efficient operation of the aeration systems is very important in an economic and environmental point of view. Operational problems may originate in the aeration tank due to inefficient mixing, bypassing streams, parallel streams, and dead zones due to solid accumulation. Operation of treatment unit in ETP is complex can often need appropriate assessment. Residence time distribution (RTD) technique is a useful tool in investigating the

unsatisfactory performance in the ETP reactors and provide means to improve the efficiency of wastewater purification and assist in their design and performance optimization.

## 1.2 Hydrodynamics study of the ASP

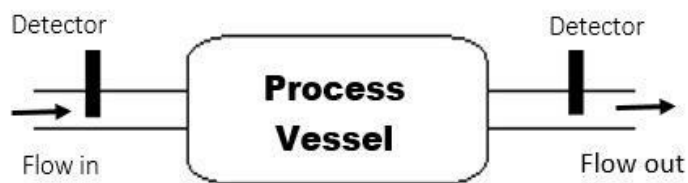
Hydraulic behavior in an activated sludge tank influences the performance of the tank and is dependent on the design of the reactor, the shape and position of the inlet and the outlet lines, external mixers in the aeration tank, baffles and water flow rates. Inappropriate design of a tank can cause flow anomalies like short-circuiting of streams, parallel paths, internal recirculation, and dead volume. Any dead volume in the tank reduces the actual volume available for reactions. An unfavorable hydraulic situation may also result in poor settling properties of sludge in the secondary clarifier. The aerators employed in the aeration tank can account for up to 70% of the total energy consumption of WWTP (Rieger *et al.*, 2006). Hence, it is important to optimize the processes in ASP, in order to save energy and ensure efficient treatment. Hydrodynamics of a reactor can be conveniently studied using residence time distribution (RTD) technique and has been reported by many researchers (Khudenko and Shpirt, 1986; Lourenço and Campos, 2009; Pant *et al.*, 2009a; Kumar *et al.*, 2012; Karches and Buzas, 2013; Jung *et al.*, 2015). Measurement and analysis of RTD of the ASP can provide insight into its hydrodynamic performance.

## 1.3 Residence Time Distribution (RTD) Technique

Residence time distribution (RTD) is a probability distribution function used to study the amount of time that process material spends inside a reactor. In industries, all the process reactors seldom behave ideally and in many cases, deviation from ideal behavior is significant and may severely affect the efficiency of the reactor. RTD analysis is an effective tool used to evaluate hydrodynamic characteristics and performance of process reactors. RTD first proposed by Macmullin and Weber (MacMullin and Weber, 1935) and developed by P.V. Danckwerts (Danckwerts, 1953; Levenspiel, 2008) has been realistically applied to numerous process to diagnose possible anomalies causing system malfunctions like dead zones, back mixing, bypassing and ineffective mixing (IAEA, 2008, 2011; Danckwerts, 1953; Farooq et

al., 2003; Kim et al., 2005; Koennecke et al., 1984; MacMullin and Weber, 1935; Levenspiel, 2008 ; Othman and Kamarudin, 2014; Pant et al., 2001; Fogler, 2011).

RTD of any reactor is experimentally determined by introducing an adequate amount of a particular tracer at the inlet of process equipment (Figure 1.2). A tracer is an inert chemical, molecule, or atom which is inserted in the reactor and its concentration is measured in the effluent stream as a function of time. Several input methods as an impulse, step, periodic may be used. In the impulse input method, the injection process is simple and the least amount of tracer is needed for the experiment (Teefy, 1996). Also, the impulse input method is usually applied due to ease in calculations of residence time distribution function  $E(t)$ , mean residence time (MRT), variance and tracer recovery rate. A productive RTD study requires selecting the best possible tracer for the given system under consideration. There is a wide range of tracers available for these applications. Since wastewater treatment plant (WWTP) units often are multiphase systems, selecting the appropriate tracer is more complex. A number of factors that need to be considered before finalizing the tracer includes, stability of tracer in the reactor process conditions (range of temperature, pH etc.), passive to the material in the reactor, detection in strong coloured effluent, absorption on to the sludge and other phases or the walls of the reactor, and convenience of tracing, cost-effectiveness, and availability (Jimenez *et al.*, 1988; IAEA, 2008). Traces available for WWTP applications have been discussed vastly in the next section.



**Figure 1.2 General tracer experiment principle**

### 1.3.1 Conventional Tracers

Chemicals like sodium dichromate, sodium iodide, sodium chloride, sodium nitrite, lithium chloride, potassium chloride, and manganese sulfate have been actively used for water

tracing in wastewater treatment units (Teefy, 1996; Tchobanoglous *et al.*, 2003). The instruments and analytical techniques used to analyze the tracer concentration throughout the RTD study includes gas chromatography, inductively coupled plasma spectroscopy, high-performance liquid chromatography, and neutron activation analysis (Teefy, 1996). Their analysis is made by chemical methods generally off-line (by sampling) and often leads to high experimental error probabilities and leads to inefficient RTD analysis. Chemical tracer studies using lithium chloride (LiCl) solution to trace liquid phase in different wastewater treatment plant (WWTP) units were reported due to easy in availability and simplicity to analyze LiCl concentration (Ronkanen and Kløve, 2007). Conventional tracers are useful at lab scale and pilot scale, but generally not suitable for application to the full-scale industrial WWTP units. This problem intensifies for RTD studies in industrial-scale reactors where high dispersion of tracer makes it difficult to trace by present analytical methods. Use of optical tracers for the RTD analysis of WWTP units can be divided into color tracers and fluorescent tracer. Use of color tracer is rarely found as the colored effluent often causes interference with the tracer analysis. The fluorescent tracer is organic substances, for example, urine, rhodamine-B, sulforhodamine-B, sulforhodamine-G, Rhodamine-WT (Di Fazio and Vurro, 1994; Kumar and Bansal, 2012). Online tracing of Rodhamine tracer is available which saves the effort of manual sampling. However, Rodhamine is not entirely inert and tends to absorb with the microorganisms used in the water treatment processes leading to their degradation (Horan *et al.*, 1991). Also, studies have found that Rodhamine tends to decay in the presence of sunlight. Fluorescent tracers are difficult to be used in biological wastewater applications as they require clear fluid for the detection of the tracer. However, for on-line investigation of water phase dynamics in some WWTP units using Rhodamine-WT and a fluorometer have been reported in the literature (Burrows *et al.*, 1999; Burrows *et al.*, 2001). The industrial wastewater typically possesses an identifiable color which causes hindrance in RTD data analysis. Table 1.1 lists common conventional tracers reported for WWTP unit study.

### 1.3.2 Radiotracers

Radiotracers are primarily radioisotope that can emit nuclear energy at detectable frequencies. They are produced in a nuclear reactor by irradiating a target element or as a by-

product during the nuclear fission reaction (IAEA, 2008, 2011). George de Hevesy (Hevesy, 1955) an early innovator of isotope technology played a key role in the development of radioactive tracers and its applications in botanical, chemical and biological research initiated the application of radiotracer and since then it has been successfully applied to study the performance of numerous industrial processes (Ambrose Jr *et al.*, 1957). Selection of a suitable radiotracer for a process study depends on its physical and chemical compatibility with the process material, its half-life, specific activity, type of radiation energy emitted and radiological protection and regulations rules (IAEA, 2008).

**Table 1.1 Conventional tracer and their application on WWTP**

Conventional tracer	Analysis methods	Field of Application
NaCl solution	Conductivity meters, ion chromatography, argentometric method, potentiometric method	
Rhodamine	Fluorometer	ASP, biological clarifiers, trickling filters
Lithium Chloride (LiCl)	Atomic absorption (AA), spectrometric inductively coupled plasma (ICP), conductivity meter	
Sodium fluoride (NaF)	Ion-selective electrode method	

The application of radiotracer in wastewater treatment plants (WWTP) is growing rapidly due to its many advantages over commonly used conventional tracers (Pant *et al.*, 2001; IAEA, 2008; Othman and Kamarudin, 2014). Characteristics as high detection sensitivity, online data measurements and requirement in minute quantity make them highly suitable for tracer experiments. As every radiotracer emits radiation of different frequency, multiple radiotracers

can be employed to study multiphase processes simultaneously. Radiotracer Bromine- 82 ( $^{82}\text{Br}$ ) and Iodine-131 ( $^{131}\text{I}$ ) have been frequently used for liquid phase tracing in wastewater treatment systems (Shin *et al.*, 2003; Kumar *et al.*, 2012). For solid-phase tracing normally used radiotracer are Gold-198 ( $^{198}\text{Au}$ ), Lanthanum-140 ( $^{140}\text{La}$ ) and Technetium ( $^{99\text{m}}\text{Tc}$ ) radiotracers are used (Pant *et al.*, 2009b; Kumar *et al.*, 2012). Commonly used radiotracer in WWTP processes is listed in Table 1.2 according to their field of application.

### 1.3.3 Advantages of Radiotracers

There are various advantages of using radiotracer over conventional tracer. They are:

- i. The radiotracer has high detection sensitivity for extremely small concentrations.
- ii. The quantity of radiotracer used for RTD study is virtually insignificant and when injected, does not cause any disturbance to the dynamics of the system under study.
- iii. For industrial plant studies, the gamma signals emitted by the radiotracers can be measured from the outside of a pipe or vessel. Hence, ease in measurement for processes dealing with toxic or selective process material.
- iv. Radiotracers allow “in-situ” measurements, hence providing valuable information about the system and save time.
- v. The radiotracer disappears from the medium under investigation through radioactive decay within a short period of time, providing scope for a repetition of experiments on the same system using the same tracer, hence minimizing the radioactive pollution.
- vi. Radioactive tracers are highly selective in nature, which implies that several tracers may be employed simultaneously to detect multiple phases of a reactor by characterizing the type of radiation emission.

The selection of the most suitable radiotracer is an important factor for the given RTD study. Several factors like physical/chemical compatibility with the material to be traced, type and energy of radiation emitted by a radioisotope, the half-life of the radiotracer, method of

measurement (sampling or in-situ measurement), specific activity, its availability, cost-effectiveness, and disposal should be considered for the selection of a radiotracer.

**Table 1.2 Commonly used radiotracer for various applications (IAEA, 2011)**

Isotope	Half-life	$\gamma$ Energy (keV)	Field of Application
Liquid phase radiotracer			
<sup>137m</sup> Ba	2.6 min	662	Equalization Tank Mixers Clarifiers Aeration Tanks Anaerobic digester
<sup>113m</sup> I	100 min	410	
<sup>99m</sup> Tc	6.02 hr	140	
<sup>82</sup> Br	1.5 days	700	
<sup>131</sup> I	8.04 days	360	
<sup>46</sup> Sc	84 days	900-1100	
Solid-phase radiotracer			
<sup>113m</sup> I	100 min	410	Sand and grit removal Clarifier (sludge) Biological reactors Collection and Discharge units
<sup>99m</sup> Tc	6.02 hr	140	
<sup>140</sup> La	1.7 days	330-1600	
<sup>198</sup> Au	2.7 days	410	
<sup>192</sup> Ir	74 days	296-468	
<sup>52</sup> Mn	5.7 days	730-1460	
<sup>175-181</sup> Hf	45 days		
<sup>160</sup> Tb	73 days	Complex spectrum	
<sup>46</sup> Sc	84 days		
<sup>147</sup> Nd	11 days		
Gaseous phase radiotracer			
<sup>82</sup> Br	36 hours	700	Biological filters
<sup>76</sup> As	26.5 hours	550-2020	Disinfection Units
<sup>79</sup> Kr	34 hours	136-830	Anaerobic digesters

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<sup>41</sup> Ar	110 min	1370	Aeration tank
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## 1.4 RTD modeling

The tracer experiment is carried out with the aim to analyze the hydrodynamic characteristic of the system. The simple data treatment applied to the RTD data is the calculation of moments. Moments are used to characterize the RTD functions in terms of statistical parameters such as mean residence time and standard deviation. The experimental RTD data obtained may only provide the real MRT of the system, but is incapable of specifying the actual hydrodynamic behavior of the tank, like the kind of flow extent of mixing, etc (Levenspiel, 1999; Fogler, 2010). RTD experimental data can be further analysed to predict the hydrodynamic behaviour of the reactor by applying various available flow models. RTD flow models provide a quantitative description of hydrodynamic characteristics of the transported material and help in understanding of a process and its prediction for additional conditions. Modeling of the experimental RTD curve with theoretical representations of different flow patterns can be performed using different software. The arrangements of basic flow elements are used to provide a proper model that gives a response identical to the tracer data obtained from the tracer experiment from the system under study. The flow in real reactors often behave as intermediates between pure convection (plug flow) and pure mixing (perfect mixer) and RTD models quantifies the deviation of the system from the ideal nature. As every reactor has its identical behavior a model provides a reasonable explanation of the flow in the real reactor. These consists of some basic RTD model that assumes the flow in the reactors is between two extremes of plug flow or mixed flow. Each model has a specific number of parameters that have to be optimized, however with an increase in a number of parameters the model becomes more and more complex. Some of these models where only one parameter accounts for the non-ideal behavior of the reactor are axial dispersion models (ADM), tank in series model (TISM), perfect mixture with plug flow, perfect mixture with dead space, perfect mixer in parallel (Wolf and Resnick, 1963; Thýn,

1983; Jager *et al.*, 1995; Martin, 2000; Burrows *et al.*, 2001; Abu-Reesh and Abu-Sharkh, 2003; Saravanathamizhan *et al.*, 2008; Pant *et al.*, 2009b; Gao *et al.*, 2012; Othman *et al.*, 2013). For a more complex system two-parameter models are used such as perfect mixers with dead space and recirculation, perfect mixture with interchange, perfect mixture with recycle (Wolf and Resnick, 1963; Roemer and Durbin, 1967; Martin, 2000; S, 2001; Kasban *et al.*, 2010; Othman *et al.*, 2013). Compartment models are multi-parameter models based on combinations of continuously stirred-tank reactors and plug-flow reactors arranged in different configurations and different parameters can be added to characterize for possible flow anomalies (Levenspiel, 1999). The structure of the compartment model resembles as closely as possible the actual flow pattern of a complex reactor that can't be depicted by general one and two parameters RTD models. The parameters of the compartment model are adjustable and can be predicted following multiple simulations with the experimentally obtained RTD data. The parameters can be used to predict the macroscopic flow model and mixing pattern in complex systems as the ASP.

## **1.5 Thesis motivation and objectives**

The industrial-scale ASP seldom behaves as ideal reactors and several operational problems may originate in the system resulting in flow anomalies like inefficient mixing, bypassing and parallel streams and dead zones. The flow anomalies in the ASP can highly affect its hydrodynamic performance and result in poor wastewater treatment efficiency. For this study, a case of integrated pulp and paper mill's ETP has been taken as a case study. The pulp and paper industries are highly dependent on water resources for their manufacturing processes. The wastewater produced by the industry has detrimental impacts on the environment and all living organisms. The paper industry is the third-largest producer of wastewater after primary metals and chemicals industries (Ali and Sreekrishnan, 2001; Agarwal and Kumar, 2012; Ashrafi *et al.*, 2015). The wastewater produced from the various processes comprises of suspended solids, biodegradable organics, pathogens, nutrients and toxic chemicals like ammonia, phosphorus, nitrates, etc. and other pollutants. Hence, it is required to treat this wastewater prior to discharge into any receiving surface or water bodies. The adverse impacts of paper and pulp industry wastewater on the environment has been

studied by many researchers, which includes oxygen depletion of receiving waters, solids settling on the bottom of lakes, rivers or marine areas, bioaccumulation of persistent organic chemicals in the aquatic food web, and eutrophication due to effluent loading of phosphorus and nitrogen (Andersson *et al.*, 1988; Owens, 1991; Ali and Sreekrishnan, 2001; Bajpai, 2001; Phiri *et al.*, 2005; Suthar *et al.*, 2009). The treated effluent should also meet environmental regulations to minimize environmental impacts on the environment. In India, the discharge standards set for pulp and paper industry by the central pollution control board (CPCB, 2000) has been shown in Table 1.3.

Tracer technique is a simple yet effective way to determine the hydrodynamic efficiency of complex processes like ASP. The tracer studies and modeling performed on the ASP help in detecting possible anomalies in the system, hence improving the treatment efficiency. The mixing pattern observed in the aeration tank can determine the optimum amount of aeration needed for the given effluent, hence decreasing the cost of aeration and making the system more economical. The RTD study on clarifier also predict the settling properties of the secondary clarifier. The fluids hydrodynamics in ASP is very important in the design of paper-pulp reactors (Upadhyay *et al.*, 1994). The study was initially based on industrial scale radiotracer experiment and later followed by a pilot scale reactor. The focus of the study was to identify anomalies in an Activated Sludge Process and to enhance the performance of existing operating Industrial reactors, rather than introduction of new treatment methods. Several studies present in literature clearly indicates the use of ASP for secondary biological treatment of Industrial wastewater.

The motivation of the study was to enhance the performance of the existing industrial ASP system and use the similar conditions in a pilot-scale reactor to optimize the performance and predict the best working parameters at which maximum treatment efficiency can be obtained. The Industrial scale ASP at Shreyans Industries Ltd. had a unique setting with two aeration tanks and two secondary clarifiers connected in series. This setting for biological treatment of pulp and paper effluent has not been studied in literature. The industrial scale plant was scale down to a pilot plant to study the effect on performance of ASP when the system

parameters are varied. The overall objective of the present work is to conduct RTD studies of activated sludge process:

- To study the performance of activated sludge process (ASP) using RTD of aeration tank, secondary clarifier and complete activated sludge process under different operating condition.
- To identify flow anomalies present in the system based on the obtained RTD data.
- To develop suitable hydrodynamic model fitting well for the system.

**Table 1.3 Effluent discharge standards for the pulp and paper industry (CPCB, 2000)**

<b>Large pulp &amp; paper/newsprint/rayon grade pulp plants of capacity above 24000 tonne/annum</b>		
<b>Parameter/ Flow</b>	<b>Concentration Limit</b>	
pH	6.5 -8.5	
SS	100 mg/L	
BOD	30 mg/L	
COD	350 mg/L	
Particulate matter	250 mg/ m <sup>3</sup>	
<b>Small pulp &amp; paper industry capacity above 24000 tonnes/annum</b>		
<b>Parameter</b>	<b>Concentration Limit</b>	
	<b>Mode of Disposal</b>	
	<b>Land</b>	<b>Inland Surface Water</b>
pH	5.5-9.0	5.5-9.0
SS	100 mg/L	100 mg/L
BOD	100 mg/L	30 mg/L

## 1.6 Thesis overview

The thesis has been divided into 6 chapters

**CHAPTER 1** covers the background effluent treatment plant and the role of the activated sludge process in biological wastewater treatment process. It also includes the importance of hydrodynamic study for aeration tank and activated sludge process and RTD technique to study hydrodynamics using various available tracers, their selection criteria, advantages and disadvantages for different tracers and available modeling techniques.

**CHAPTER 2** covers the literature review for the various tracer experiments carried out in industrial as well as pilot scale for the characterization of the WWTP reactors. Literature is categorized into particularly emphasized on the use of radiotracers and conventional tracers in industrial as well as pilot and lab-scale RTD study and finally, research gaps were identified.

**CHAPTER 3** includes the materials and methods used during the RTD experiments. It also includes the experimental setup for various experiments, methodology for RTD experiment, RTD data treatment, selection of various RTD models and its equation that have been used to characterize the reactor performance. The procedure for parametric analysis and characterization of wastewater have also been discussed.

**CHAPTER 4** deals with the RTD experiments performed in an industrial-scale fully integrated pulp and paper mill activated sludge process (ASP) using Iodine-131 as the radiotracer. Anomalies were identified in the reactor and appropriated RTD models developed for aeration tank, secondary clarifier, and complete ASP. To predict the flow behaviour in the reactors, model fitting was done using the fundamental one-parameter models like axial dispersion model (ADM) and tank-in-series with back mixing model (TIBM) and multi-parameter models like tank-in-series with back-mixing (TISBM) and other combination of TIS and ADM models represented as compartment models. This chapter also discusses the use of convolution techniques to develop models for aeration tank, secondary clarifier for distorted impulse RTD signals inputs and the subsequent modeling techniques.

**CHAPTER 5** discuss the RTD experiments performed on pilot scale ASP at various operating parameters and their effect on the hydrodynamic performance. The operating parameters, mean hydraulic residence time (MHRT) and the recycle rate were varied and a range of RTD experiments were performed to characterize the hydrodynamic behavior of the reactors with varying operating parameters. RTD models were suggested to obtain a complete overview of the reactors flow dynamics. The parametric characterization of the wastewater and the substrate removal efficiency at different operating parameters were also analyzed. The chapter

also relates the biological and hydrodynamic performance of the ASP and provides validation to the industrial scale RTD experiments.

**CHAPTER 6** summarizes the conclusion of the present work.

At the end of the chapters references cited and publication have been listed.

## **LITERATURE REVIEW**

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The residence time distribution (RTD) analysis is very useful for flow characterization and investigation of hydrodynamic flows in industrial and lab-scale wastewater treatment plant units. The knowledge of RTD is also important for accurate kinetic modeling of the system, and help reactor design to achieve the desired flow pattern (Levenspiel, 1999). This chapter presents review of research work for the RTD study on industrial-scale and pilot-scale ASP using tracer technique.

### **2.1 RTD study in industrial-scale WWTP**

In the early 1900 century, the flow behavior of various treatment unit in effluent treatment plant became a study interest. It was realized that the flow in the full-scale industrial treatment units is rarely ideal. The deviation of the processes from the ideal behavior be dependent on various factors like its inflow and outflow conditions, type and intensity of mixing, no of phases involved (Levenspiel, 1999; Tchobanoglous *et al.*, 2003). Many times this may affect the working efficiency of the process equipment. RTD study is a very efficient tool to successfully determine the hydrodynamic characteristic of a process. It can provide information on how long the process material spends in the reactor. The RTD data not only provides hydrodynamic information but can be further used for modeling and improvement of design.

#### **2.1.1 RTD study on industrial scale WWTP with conventional tracer**

The use of convention tracer like coloured dyes, chemical salts for RTD study of wastewater system has been reported for the RTD study of industrial wastewater treatment unit (Bode and Seyfried, 1985; Makinia and Wells, 2005). Lithium Chloride (LiCl) is the most common salt used for tracer study (Olivet *et al.*, 2005; Frącz and Wotzka, 2016). Lithium chloride is preferred over other tracers for WWTP unit studies because it is not a normal component of wastewater. Also, it is inert in nature and does not react with any substance

present in the wastewater system, and it is easily detected by basic analytical methods and recovery rates of LiCl are higher than those reported for fluorescent dyes (Kjellstrand *et al.*, 2005).

Fluorescent dye tracer, Rhodamine WT has also been applied for RTD studies and determine flow anomalies like dead zones and short-circuiting stream in full-scale WWTP reactor studies (Di Fazio and Vurro, 1994; Burrows *et al.*, 2001). Rhodamine WT allows RTD study at low concentrations, but it is susceptible to absorption to the microorganism flocs utilized in the activated sludge treatment process. Additionally, influent wastewater may contain pollutants exhibiting fluorescent properties, which can cause background interference (Knap and Balbierz, 2017).

The difficulty of using conventional tracer in full-scale industrial systems was mentioned by many researchers (Teefy, 1996; IAEA, 2008). There were many problems related to manual data collection, inadequate efficiency in recycling systems and a large amount of tracer used in the RTD analysis have become the critical problems associated with the use of conventional tracer (Bode and Seyfried, 1985). In large scale industrial wastewater treatment units, the tracer dispersion leads to low concentrations at the outlet making it difficult to detect and hence resulting in non-uniform concentration profile of the RTD curve. Also, in secondary wastewater treatment units if the concentration of salts used for tracer exceeds a certain limit can hinder the growth of microorganisms employed in the secondary treatment process (Knap and Balbierz, 2017).

The sampling of conventional tracers is carried out offline, by a periodic collection of the sample at various location. However, every point in the industry is not accessible for sampling, hence providing less knowledge of the system. Many of the initial RTD analysis of industrial-scale secondary units have been performed without considering the effect of recycling on the hydrodynamics of the system due to difficulty in the analysis of a very small concentration of tracer due to dispersion in the reactor. (Makinia and Wells, 2005) mentioned the difficulty in interpreting RTD data in wastewater system with recycling line using conventional tracer. Most of the RTD experiments performed using conventional tracer on the WWTP were to trace liquid phases only and were unable to consider the effect of gasses and

solid due to a limited number of option available to trace solid and gaseous phases (Horan *et al.*, 1991). The RTD curves obtained from tracer experiments can be utilized to predict appropriate hydraulic models that provide a comprehensive view of the hydrodynamics of a reactor. The modeling approach in conventional tracer approach is limited to single parameter model or those available in literature as the sampling data is limited and prone to errors (Coen *et al.*, 1998; Burrows *et al.*, 1999). The inefficiency of manual sampling with conventional tracers limits its use to such enhanced modeling techniques (Kjellstrand *et al.*, 2005). The critical literature available for tracer studies in WWTP has been presented in Table 2.1.

### **2.1.2 RTD study on industrial scale WWTP with radiotracer**

Industrial wastewater treatment reactor are complex systems that are a combination of mechanical chemical and biological processes. Radiotracer technique is the only fine and non-intrusive technique for characterizing the flow characteristics and the measurement of local residence time distributions (RTD) in WWTP reactors (Blet *et al.*, 2000). Many researchers have successfully applied radiotracer to study the hydraulic behaviour of various units in WWTP (Ambrose Jr *et al.*, 1957; Thýn *et al.*, 1992; Borroto *et al.*, 2003; Farooq *et al.*, 2003; Calvo *et al.*, 2004; Kim *et al.*, 2005; IAEA, 2011; Debien *et al.*, 2013) due to ease of data collection and high accuracy.

#### **2.1.2.1 RTD study in industrial primary wastewater treatment processes**

The wastewater collected from any industry passes through various stages of processing before it is recycled or discharged in the environment. The initial treatment process starts with the screening of large solids of size range 6 to 150 mm diameter that can disrupt other processes. After required pre-treatment, the wastewater is transferred to a settling basin also known a primary clarifier. The primary clarifier is often a rectangular or circular sedimentation tanks that allow settling of the solid particle of size less than 1 mm and that could not be separated by screening. These solids flocculate and settle down at the bottom of the clarifiers as sludge which is removed with the help of a scraper. Primary clarifiers are responsible for removal of 50% to 70% of the suspended solids present in the wastewater and in a few industries they are sufficient in obtaining the desired removal of solids.

**Table 2.1 Summary of literature review on RTD studies of WWTP units with conventional tracer**

<b>System</b>	<b>Phase</b>	<b>Tracer</b>	<b>Model</b>	<b>Findings</b>	<b>Reference</b>
Plug flow Bioreactor	liquid	LiCl	Axial dispersion model and Tank-in- series model and Compartment model	Local and global RTD in the aeration lanes identifies the mixing regime in the tank ( $V_d=5\%$ )	(Olivet <i>et al.</i> , 2005)
Activated sludge reactor	liquid	Rodhamine WT	Axial dispersion model	Empirical models to determine dispersion coefficient $E_L$ ( $E_L$ calculated within an optimum range of 1043-1550 $m^2/h$ )	(Makinia and Wells, 2005)
Activated sludge reactor	liquid	LiCl, Rodhamine WT, spores of bacteria(bac teriophage and B.globigii spores)	-	Comparison of different tracers (based on their binding capacity with the microorganisms present in the system) for RTD and performance evaluation of ASP. (Rodhamine WT most	(Horan <i>et al.</i> , 1991)

				suitable tracer with least binding of 57% )	
Activated sludge reactor	liquid	LiCl	Tank-in-series model	Non-uniform distribution of flow in different lanes of the ASP causing dead volume.	(Coen <i>et al.</i> , 1998)
Oxidation ditches	liquid	Rodhamine WT	Tank-in-series model	Flow characterization and prediction of dissolved oxygen profile	(Burrows <i>et al.</i> , 2001)
Denitrifying activated sludge tank	liquid	LiCl	Tank-in-series model and Martin model	Identify short circuiting streams and dead zones. At normal flow conditions ( $V_d=12.8\%$ , $V_b=1.3\%$ ) and for max. flow ( $V_p=24.3\%$ )	(Kjellstrand <i>et al.</i> , 2005)
Denitrification tank	Liquid	LiCl	Tank-in-series model	Dead space- 25-40%, flow bypass – 40%, removal of the dead zone by changing reactor orientation.	(Collivignarelli <i>et al.</i> , 2018)

\* $V_d$ : dead volume,  $V_b$ : bypassing volume,  $E_L$ : Dispersion coefficient

The hydrodynamics of primary clarifier has been studied by the researcher to understand the flow behavior and detect anomalies like dead zones, bypassing, internal

recirculation, etc. Radiotracer Bromine-82 ( $^{82}\text{Br}$ ) is commonly used to study the liquid phase tracing (Chmielewski *et al.*, 1998; Farooq *et al.*, 2003). Different modeling approach like using theoretical model equations (Burrows *et al.*, 2001; Fogler, 2010) or computer software-based modeling (DTS PRO) were used to get a systematic view of the flow in the clarifier. From these experiments, it was found that primary clarifiers may contain 25-50% dead-zones and strong internal recirculation (Koennecke *et al.*, 1984; Kasban *et al.*, 2010).

Equalization tank that also an essential part of primary treatment that provides uniform effluent stream for further processing of wastewater. The major function of the equalization tank is to provide homogenization of solid and liquid phase by evenly distributing the pollutants in the wastewater. The hydrodynamics of liquid phase in the equalization tank of various shapes (circular, triangular and rectangular) and volumes were studied using radiotracer technique (Chmielewski and Selecki, 1979; Chmielewski *et al.*, 1998; IAEA, 2011). Performance of an industrial scale equalization cum clarifier tank was analyzed using radiotracer Br-82, the total activity of 3.7 GBq. The study revealed the presence of dead zones in the tank and was estimated to be 25% at the time of the experiment. From the study, they also found that the flow stagnation was located in the bottom regions of the tank due to sedimentation. The flow model proposed to represent the system was two CSTR in series with dead volume. The summary of the literature available for radiotracer study in primary WWTP has been presented in Table 2.2.

### **2.1.2.2 RTD study in industrial secondary wastewater treatment processes**

Wastewater with high biochemical oxygen demand (BOD) and chemical oxygen demand (COD) contains a substantial amount of dissolved organics that cannot be eliminated by primary treatment. They are further treated in secondary treatment units which include activated sludge process, trickle bed filters, aeration tanks, secondary clarifiers, ponds, and lagoons, etc. Several radiotracer studies have been performed on individual and combinations of secondary WWTP processes (Farooq *et al.*, 2003; Gul *et al.*, 2003; Kim *et al.*, 2005; Le Moullec *et al.*, 2008; Le Moullec *et al.*, 2010a, b; Kumar *et al.*, 2012).

**Table 2.2 Summary of literature review on radiotracer studies in primary WWT unit**

System	Phase	Radiotracer	Model	Findings	Reference
Sand Separator	liquid	$^{82}\text{Br}$	Tank-in-series with dead volume	Active volume of the separator ( $V_d = 40\%$ )	(Chmielewski and Selecki, 1979)
Equalization Tank cum primary settler	liquid	$^{82}\text{Br}$	Tank-in-series with dead volume	Significance of radiotracer experiments for RTD investigation of industrial-scale WWTP units ( $V_d=25\%$ )	(Chmielewski and Selecki, 1979; Chmielewski <i>et al.</i> , 1998)
Primary clarifier	liquid	$^{82}\text{Br}$	Compartment model	Identification of dead zone and bypassing in the clarifier.  ( $V_d = 43\%$ , $V_b = 19\%$ )	(Farooq <i>et al.</i> , 2003)

\* $V_d$ : dead volume,  $V_b$ : bypassing volume

Aeration tanks, first studied in 1910 (Tchobanoglous *et al.*, 2003), consists population of suspended microorganisms that utilized organics present in the wastewater as a food source and break them down into simpler compounds like  $\text{CO}_2$  and  $\text{H}_2\text{O}$  at aerobic conditions. Air diffuser or mechanical surface aerators are equipped in the aeration tank that not only provide oxygen to maintain aerobic conditions but also provide homogenous mixing (Tchobanoglous *et al.*, 2003; Peavey *et al.*, 2013). The dispersion of oxygen involves high energy consumption resulting in high operating cost, accounting for 45% to 75% of the total energy cost (Rieger *et*

*al.*, 2006; Karpinska and Bridgeman, 2016). Radiotracer studies on aeration tanks (Farooq *et al.*, 2003) has been performed to study the mixing characteristics and detect flow anomalies present in the tank. It was found that aeration tank usually acts as perfect mixers and contains a very low percentage of the dead zone, around 1% to 10%, but very high internal recirculation and bypass stream.

The biomass produced in the aeration tank settles down in the secondary clarifier. The design of secondary clarifier is more complicated. Bench-scale studies using radioactive tracers have corroborated, higher efficiency of the peripheral-feed tank over center feed models. Centre feed basins showed inefficiencies due to short-circuiting of the incoming flow. It was also found that short-circuiting could be reduced 3 - 4 times in of the peripheral-feed basin (Kim *et al.*, 2005; IAEA, 2011). The study of the liquid phase in secondary clarifier was usually done with  $^{82}\text{Br}$ ,  $^{113\text{m}}\text{In}$ , and  $^{131}\text{I}$  radiotracer. It was found that  $^{113\text{m}}\text{In}$  was not a very suitable radiotracer for online RTD analysis of secondary clarifiers due to its very short half-life (99.5 mins) compared to the mean retention time secondary clarifiers (2 to 4 hrs.) (Kim *et al.*, 2005). The study of only the aqueous phase in secondary clarifier is not enough to understand the hydrodynamics of secondary clarifier, as the sludge retention time also plays a very important role. The solid settling profile had been studied by researchers (Audic *et al.*, 1994; De Clercq *et al.*, 2005; Kim *et al.*, 2005). The solid phase tracing was done using  $^{140}\text{La}$ ,  $^{198}\text{Au}$  as a radiotracer. Kim *et al.* (2005) extended the radiotracer experiment to develop a computer program that was useful in determining operational behavior and provided insight to the recirculation zones and settling zone and their effect on removal efficiency of the reactor.

Trickling filters are also used for secondary treatment of wastewater. They have attached a culture system that uses rock, gravel, ceramic or polymer packing as support. The effluent water flows on this packing forming a biofilm. The hydrodynamic characteristics of industrial-scale trickle bed reactor with different kinds of packing have been studied using radiotracers like  $^{82}\text{Br}$  and  $^{99\text{m}}\text{Tc}$  (Pant *et al.*, 2000; Kumar *et al.*, 2012; Debien *et al.*, 2013). Most of these studies were carried out to compare various packing material and assess the liquid holdup and dispersion in the system. Debien *et al.* (2013) compared liquid holdup and axial dispersion for sponge based packing (Rotopack) with polyurethane foams packing, where

short-circuiting and lack of homogeneous mixing in filter with polyurethane packing. Kumar *et al.* (2012) assessed the liquid holdup and dispersion for hydrophobic and hydrophilic packing material. The study found that the liquid holdup for hydrophilic packing material was higher and it increased with the increase in liquid flow rates. It was also found that the liquid dispersion for hydrophobic packing was higher than hydrophilic packing due to excessive fluid exchange between the stagnant and dynamic zone of the trickle bed reactor. Pant *et al.* (2000) also conducted radiotracer with  $^{99m}\text{Tc}$  for three different packing type being nonporous glass beads, porous catalysts of tablet and extrudate shape. They found that all three types of packing the liquid holdup increased with an increase in liquid flow rate and axial mixing was highest for glass beads packing. Table 2.3 lists the summary of literature available for radiotracer investigation in secondary WWTP.

### 2.1.2.3 RTD study in the industrial sludge treatment unit

During primary and secondary treatment of wastewater, a large amount of sludge accumulates at the bottom of the clarifiers. This sludge needs further treatment, which is done in a sludge digester. A sludge digester reduces the amount of sludge by breaking down the organics into  $\text{CO}_2$  and  $\text{CH}_4$  gas. The performance of sludge digester is highly affected by the flow behavior of solid and liquid in the system and have been studied by researchers.

The sludge digesters usually have MRT in days, the RTD investigation of solid-phase particles like sand and mud is done with radiotracers like Hafnium-175( $^{175}\text{Hf}$ ), Hafnium-181( $^{181}\text{Hf}$ ), Iridium-191( $^{191}\text{Ir}$ ) and Scandium-46 ( $^{46}\text{Sc}$ ) that have half-life in days (Jung *et al.*, 1999; Jung *et al.*, 2001, 2005; IAEA, 2011). (Borroto *et al.*, 2003) performed an RTD study on the liquid phase in anaerobic digester in a sugar mill WWTP using commercially generated radioisotope  $^{99m}\text{Tc}$ . They found that the digester acted as a series of perfect mixing tanks and the RTD experiments provided sufficient information on the geometrical and physical behavior of the digester. Summary of literature available for radiotracer studies on wastewater sludge treatment processes has been given in Table 2.4.

**Table 2.3 Summary of literature on radiotracer studies in secondary WWT unit**

System	Phase	Radiotracer	Model	Findings	Reference
Biological clarification Plant	Liquid	$^{82}\text{Br}$	-	Bypassing found in the reactor was reduced by 50% and energy saving.	(Koennecke <i>et al.</i> , 1984)
Aeration Tank	Liquid	$^{82}\text{Br}$	Compartment model	Internal recirculation and dead zones ( $V_d=1.5\%$ , $\alpha=2.7$ )	(Farooq <i>et al.</i> , 2003)
Secondary clarifier	Liquid	$^{82}\text{Br}$	Compartment model	Poor efficiency of secondary clarifier due to significant dead zone ( $V_d=56\%$ )	(Farooq <i>et al.</i> , 2003)
Secondary Clarifier	Solid	$^{113m}\text{I}$	mono-disperse settling model and discrete settling model	Development of computer software to study solid distribution and removal efficiency	(Kim <i>et al.</i> , 2005)
Secondary Clarifier	solid	$^{99m}\text{Tc}$	-	Solid distribution and settling characteristics	(De Clercq <i>et al.</i> , 2005)
Secondary Clarifier	solid	$^{198}\text{Au}$	-	Short-circuiting, sludge distribution, and settling characteristics	(Audic <i>et al.</i> , 1994)
Trickle Bed	liquid	$^{82}\text{Br}$	Axial dispersion model	Comparison of liquid holdup and axial dispersion for hydrophobic and	(Kumar <i>et al.</i> , 2012)

				hydrophilic packing type	
Trickle Bed	Liquid	$^{99m}\text{Tc}$	Axial dispersion model	Comparison of liquid holdup and axial dispersion for different packing of glass beads, porous catalysts, and extrudate shape	(Pant <i>et al.</i> , 2000)
Trickle Bed	Liquid	$^{82}\text{Br}$	Axial dispersion model	Comparison of liquid holdup and axial dispersion for polyurethane and rotapack	(Debien <i>et al.</i> , 2013)
Anaerobic pond	Liquid	$^{131}\text{I}$	Compartment model	Parallel stream- 5%, $V_d$ - 93% and $V_b$ - 3%	(Adwet <i>et al.</i> , 2019)

\* $V_d$ : dead volume,  $V_b$ : bypassing volume,  $\alpha$ - back-mixing ratio

## 2.2 RTD study on pilot scale WWTP

Industrial wastewater system often operated at a predefined set of conditions according to the needs of the industry, which can't be varied or exploited beyond some extent for research purposes. Hence, to study the effect of various parameters on the performance and hydraulic efficiency of the effluent treatment unit and optimize the performance of such industrial reactors, RTD experiments are carried out at lab scale or pilot-scale reactors.

**Table 2.4 Summary of literature review on radiotracer studies in sludge treatment unit.**

System	Phase	Radiotracer	Model	Findings	Reference
Anaerobic	liquid	$^{99m}\text{Tc}$	Compartment	Flow behavior of the	(Borroto <i>et</i>

Sludge Digester			model	opaque digester, and absorption rate of $^{99m}\text{Tc}$ in the sludge.	<i>al.</i> , 2003)
Anaerobic Sludge Digester	Solid (mud and sand)	$^{175}\text{Hf}$ $^{181}\text{Hf}$ $^{191}\text{Ir}$	Axial dispersion model	Tracing the flow of mud and sand in the reactor, mud and sand recirculation at the bottom of the reactor ( $V_d$ : 30- 40%)	(Jung <i>et al.</i> , 1999; Jung <i>et al.</i> , 2001)
2 stage cylindrical sludge digester	Solid (Sludge)	$^{46}\text{Sc}$	Tank in series model	Dead zone (digester 1 $V_d$ - 13% and digester 2 $V_d$ - 27%)	(Jung <i>et al.</i> , 2005)

\* $V_d$ : dead volume

### 2.2.1 RTD study on pilot-scale WWTP using conventional tracer

Conventional tracers may not fulfill as the best tracer choice for industrial-scale RTD studies, however perfectly fits the requirements for the experiments to be performed at laboratory or pilot plants. They are cheap, widely available and easy to analyze applying simple analytical methods. Regular sampling is not a big issue at these small scale treatment setups. Many researchers have used LiCl, NaCl and Rodhamine WT to characterize pilot-scale treatment units and develop models that are useful to enhance the performance of existing industrial WWTP units or even test the use of unconventional tracers for RTD studies (Khudenko and Shpirt, 1986; Ahnert *et al.*, 2010; Le Moullec *et al.*, 2010b; Sánchez *et al.*, 2016). An alternative approach of RTD experiment using wastewater temperature variation as a tracer was presented by comparing with LiCl tracer experiment (Ahnert *et al.*, 2010) in a WWTP reactor. The estimated RTD results both with conventional and temperature tracer methods showed fair agreement and were able to successfully predict exchange zones, dead

zones and short-circuiting streams in the reactor. Prediction of novel empirical formulas to calculate dispersion coefficient in aeration lanes were carried out by performing multiple RTD experiment on a pilot-scale aeration tank at different conditions (Le Moullec *et al.*, 2010a).

The calculated axial dispersion coefficient was further used for CFD simulation of wastewater gas-liquid cross-flow reactor taking into account hydrodynamics, mass transfer, and biological reactions. (Sánchez *et al.*, 2016) tried to model the ASP with varying recycle system and study the effect of sludge recirculation on activated sludge reactor performance. Lab-scale studies to evaluate the hydrodynamic behavior of a lab-scale up-flow anaerobic sludge blanket reactor (UASB) has also been reported using LiCl as a tracer (Lourenço and Campos, 2009). It was observed that the flow inside the reactor was highly dispersed, and the experimental MRT was three times the designed MRT of the tank. The research was very useful to overcome the scaling-up problems in biological reactors. Table 2.5 summarises the literature available for tracer investigation on pilot scale WWTP.

### **2.2.2 RTD study on pilot-scale WWTP using Radiotracer**

Radiotracers are particularly difficult to be used for lab-scale RTD studies due to stringent rules for careful execution and disposal of the radioisotope. Radiotracers are analyzed with on-line sampling equipment that is too expensive. They also may require separate laboratory sections to prevent any undesirable accidents. All these factors tend to make radiotracers a secondary choice in lab and pilot-scale studies, despite their numerous said advantages over conventional tracers. Although, due to unavailability of suitable conventional tracer for gaseous and solid-phase tracing in WWTP units, use of radiotracer becomes obligatory. In such cases, tracers with low activity and short half-life are used. Br-82 was used to study the sewage sludge production in two distinctly constructed wetland to predict an alternative treatment method that results in low operating cost and reduced energy consumption (Matamoros *et al.*, 2005).

### **Table 2.5 Summary of the literature on pilot scale WWT reactor with conventional tracer**

System	Phase	Tracer	RTD Model	Findings	Reference
Aeration Tanks	Liquid	LiCl	-	Experimental evaluation of Bodenstein number ( $B_o$ ) and its importance in Aeration tank's design and scale-up problems	(Khudenko and Shpirt, 1986)
Water treatment cascade reactor	Liquid	LiCl, temperature	Tank-in-series model	Temperature as an alternative tracer for RTD study	(Ahnert <i>et al.</i> , 2010)
Aeration tank	Liquid	NaCl	Axial dispersion model	Identify short-circuiting streams and dead zones	(Le Moullec <i>et al.</i> , 2010a)
Activated sludge process	Liquid	LiCl	Modified tank in series model	Effect of recycling on ASP having different recycling schemes	(Sánchez <i>et al.</i> , 2016)
Anaerobic sludge blanket reactor	Liquid	LiCl	Tank-in-series model	High experimental MRT than defined MRT and dispersed flow, with dispersion number, $d=0.2675$	(Lourenço and Campos, 2009)

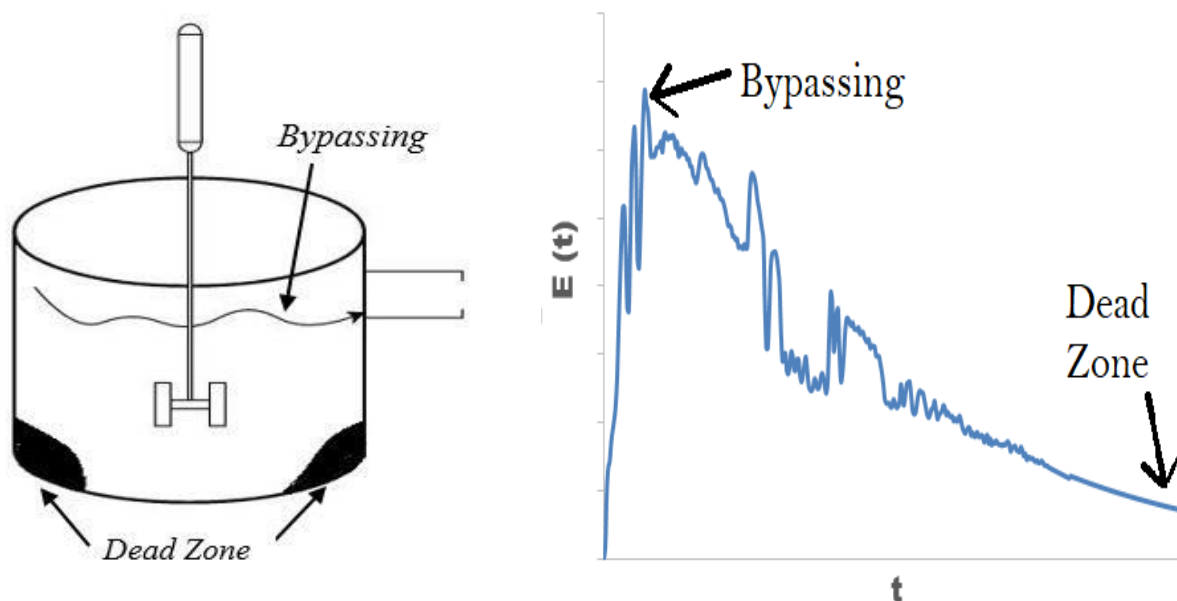
\* $B_o$ - Bodenstein number ( $D/UL$ )

## 2.3 RTD modeling

The experimental RTD data obtained may only provide the real MRT of the system, but is not sufficient to specify the actual hydrodynamic behavior of the tank, like the flow from and extent of mixing, etc. RTD formulation is the fundamental step for further RTD data processing, modeling, and interpretation. The experimental RTD data contain statistical fluctuations and other background influences (IAEA, 2008; Kasban *et al.*, 2010). Hence some basic corrections are needed before the data can be utilized for further study are dead time correction, background correction, radioactive decay correction, filtering (or smoothing), data extrapolation (or tail correction). The data obtained after treatment is converted to normalized form by calculating RTD function  $E(t)$  at the outlet of the reactor. The RTD function  $E(t)$  is characterized by calculating moments. These moments allow the representation of the RTD functions in terms of statistical parameters such as mean residence time ( $\bar{t}$ ) and variance ( $\sigma^2$ ). Other parameters such as skewness and kurtosis can be calculated using moments of a higher order.

The comparison of experimentally calculated MRT ( $\bar{t}$ ) and theoretical MRT of the process reactor provides insight into the system. MRT and variance are also served as essential parameters required in the modeling of the system. From the literature, it was found that the presence of dead zones and bypassing stream are the most prominent abnormalities present in WWTP reactors as shown in Figure 2.1. Dead zones are those segments of the reactor that are inactive. A long tail in the normalized RTD curve indicates the presence of dead- zones in the reactor.

A part of process material may leave the reactor early, spending barely any time in the reactor. This material stream is termed as bypassing stream. The initial jump or presence of two separate peaks in the  $E(t)$  curve signifies bypassing in the reactor. For a single peak in the  $E(t)$  curve the fraction of bypassing is given by the area under that peak. However, if two distinct peaks are present in the  $E(t)$  curve then the fraction of bypassing can be calculated by the ratio of the areas under the first and second peak.



**Figure 2.1**  $E(t)$  curve revealing bypassing and dead-zones in the tank

As every reactor has its identical flow behavior and RTD model provide a reasonable explanation of the flow in the real reactor. These consists of some basic RTD model that assumes the flow in the reactors is between two extremes of plug flow or mixed flow. Each model has a specific number of parameters that have to be optimized, however with an increase in a number of parameters the model becomes more and more complex. Some of these models where only one parameter accounts for the non-ideality of the reactor are axial dispersion models (ADM), tank in series model (TISM), (Wolf and Resnick, 1963; Thýn, 1983; Jager *et al.*, 1995; Martin, 2000; Burrows *et al.*, 2001; Abu-Reesh and Abu-Sharkh, 2003; Saravanathamizhan *et al.*, 2008; Pant *et al.*, 2009; Gao *et al.*, 2012; Othman *et al.*, 2013). For a more complex system two-parameter models are used such as perfect mixers with dead space and recirculation, perfect mixture with interchange, perfect mixture with recycle, perfect mixture with plug flow, perfect mixture with dead space, perfect mixer in parallel (Wolf and Resnick, 1963; Roemer and Durbin, 1967; Martin, 2000; S, 2001; Kasban *et al.*, 2010; Othman *et al.*, 2013). The modeling approach in conventional tracer approach is limited to single parameter model or those available in literature as the sampling data is limited and prone to errors (Coen *et al.*, 1998; Burrows *et al.*, 1999). Advanced mathematical modeling techniques

as CFD and black box approach help to further identify shortcoming in the system like short-circuiting streams and dead volumes in the system more specifically. CFD modeling is a beneficial and informative tool for analyzing the hydraulics of WWTP reactors, study of theoretical corrective measures and design validation. The process reactors can be further reviewed in detail using CFD. This type of modeling also deserves to have a more central role when designing large reactors and other hydraulic systems being a useful tool for virtual prototyping. These models often compare their results to RTD experimental results for the system. The inefficiency of concentration measurement with conventional tracers limits its use to such enhanced modeling techniques (Kjellstrand *et al.*, 2005). Fracz & Wotzka, 2016 applied Tanks-in-series, Extended Tank-in-Series and modified MARTIN model to identify anomalies like short circuiting flow in Industrial scale WWTP. Sánchez *et al.*, 2016 suggested models based on conventional pulse model to analyse ASP with recycling systems. CSTR-in-series and ADM model was also applied by Youseff *et al.*, 2017 to characterize biomass flow in a pretreatment reactor. The models were used to predict anomalies in the reactor and determine the actual mean residence time of the reactor. The RTD models were extended to study two-phase flow in WWTP reactor and to quantify various anomalies and enhancement of reactor performance Collivignarelli *et al.*, 2018; Delre *et al.* 2018; Jung *et al.*, 2019).

In the last few years simulation-based modeling software, Computational fluid dynamics (CFD) has gained wide acceptance for predicting the hydrodynamics of wastewater treatment reactors. CFD is a computer-based software used to analyze and model hydrodynamics of a system. CFD provides a complete spatial image of the process occurring inside the system. It is a very essential tool that can be used to validate the experimental data collected from the RTD experiments and optimize the system. CFD analysis can be used to predict fluid and mass flow rates, mixing efficiency, sludge distribution, and settling rates, impeller efficiency, etc. The results from CFD analysis can be further extended to troubleshoot or re-design the system or development of new design (Le Moullec *et al.*, 2008; Lemoullec *et al.*, 2008; Le Moullec *et al.*, 2010a, b). Lemoullec *et al.* (Lemoullec *et al.*, 2008) used CFD to predict the axial coefficients of dispersion in an aerated channel reactor and compared the

equation with already existing empirical correlation present in literature. Table 2.6 presents the model equation of various RTD models along with the associated parameters to be determined.

On the basis of the literature review, it was found that although aeration tank and secondary clarifier has been studied separately using radiotracer technique, this technique has not been used to a study their performance and flow characteristics of a complete activated sludge process (i.e. aeration tank and secondary clarifier with return sludge). The available literature also indicates RTD study performed on ASP deals with hydrodynamics performance only but neglect the effect of operating parameters on biological treatment efficiency.

Table 2.6 Frequently used RTD models in the literature (Levenspiel, 2001; Collivignarelli *et al.*, 2018)

Model Category	Model Name	Model Equation	Model parameters to be optimized
Single parameter model	Tank in series Model	$E(t) = (t)^{N-1} \frac{N^N}{\tau^N (N-1)!} \exp\left(\frac{-tN}{\tau}\right)$	$\tau, N$
	Axial Dispersion Model	$E(t) = \frac{1}{2} \left(\frac{Pe}{\pi t \tau}\right)^{\frac{1}{2}} \exp\left(-\frac{Pe(\tau-t)^2}{4t\tau}\right)$	$\tau, Pe$
	Single mixed flow reactor with only dead space	$E(t) = \frac{1}{\tau(1-i_d)} \exp\left(\frac{-t}{\tau(1-i_d)}\right)$	$\tau, i_d$
	Single mixed flow reactor with only flow bypass	$E(t) = i_b \delta(t) + \frac{(1-i_b)^2}{\tau} \exp\left(\frac{-t(1-i_b)}{\tau}\right)$	$\tau, i_b$
Two-parameter	single CSTR with both dead	$E(t) = i_b \delta(t) + \frac{(1-i_b)^2}{\tau(1-i_d)} \exp\left(\frac{-t(1-i_b)}{\tau(1-i_d)}\right)$	$\tau, i_d, i_b$

model	space and flow bypass is: $N$ - CSTR with flow bypass is	$E(t) = i_b \delta(t) + (1 - i_b)^2 \frac{N^N}{\tau^N (N - 1)!} (t(1 - i_b))^{N-1} \exp\left(\frac{-tN(1 - i_b)}{\tau}\right)$	$\tau, N, i_b$
	$N$ -CSTR with only dead space	$E(t) = \frac{1}{(1 - i_d)} \frac{N^N}{\tau^N (N - 1)!} \left(\frac{t}{1 - i_d}\right)^{N-1} \exp\left(\frac{-tN}{\tau(1 - i_d)}\right)$	$\tau, i_d, N$
Three- parameter model	$N$ -CSTR with recycle  $N$ - CSTR with both dead space and flow bypass	$E(t) = \frac{1}{R} \sum_{m=1}^{\infty} \left(\frac{R}{R + 1}\right)^m \frac{1}{\tau^{mN}} \frac{t^{mN-1}}{(mN - 1)!} \exp\left(\frac{-t}{\tau}\right)$ $= i_b \delta(t) + \frac{(1 - i_b)^2}{(1 - i_d)} \frac{N^N}{\tau^N (N - 1)!} \left(\frac{t(1 - i_b)}{(1 - i_d)}\right)^{N-1} \exp\left(\frac{-tN(1 - i_b)}{\tau(1 - i_d)}\right)$	$\tau, N, m, R$  $\tau, i_d, i_b, N$

\*  $\tau$  - theoretical mean hydraulic retention time,  $t$  - theoretical mean hydraulic retention time,  $N$  - number of reactors,  $i_b$  - flow bypass index, and  $i_d$  - dead space index,  $Pe$  - Peclet No,  $R$  - Recycle Ratio,  $m$  - no of recycle passes,  $\delta(t)$  - impulse function.

## **MATERIAL AND METHODOLOGY**

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In this chapter the various materials and methods used during the RTD experiments has been discussed. It also includes the experimental setup for industrial and pilot-scale experiments, methodology for RTD experiment, RTD data treatment, selection of various RTD models and its equation that have been used to characterize the reactor performance. The procedure for parametric analysis and characterization of wastewater have also been discussed.

### **3.1 Material**

#### **3.1.1 Wastewater**

Real-time effluent for was collected from the overflow of the primary clarifier from an indigenous pulp and paper industry in India (M/s Shreyans Industries Ltd., Ahmedgarh, Punjab, India). The characteristics of the wastewater were (mg/L) BOD 580, COD 1050, MLSS 2800-3500 and pH 7.53. The effluent was stored at 4° C before it was used for the pilot scale RTD experiment. The microbial seed for the aeration tank was collected from the industry and aerated for 24 h at room temperature before inoculating the reactors.

#### **3.1.2 Chemicals**

All the chemicals used in the present study were of analytical reagent (AR) grade. Sulphuric acid (H<sub>2</sub>SO<sub>4</sub>), potassium chromate indicator, standard silver nitrate, sodium chloride (NaCl), methyl orange indicator, phenolphthalein indicator, was purchased from Merck, Germany. Ferrous sulphate (FeSO<sub>4</sub>), mercuric sulphate (Hg<sub>2</sub>SO<sub>4</sub>), silver sulphate (Ag<sub>2</sub>SO<sub>4</sub>), potassium dichromate (K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub>), sodium hydroxide (NaOH) were purchased from Sigma Aldrich fine chemicals, America.

#### **3.1.3 Radiotracer experiment on industrial scale WWTP**

Industrial scale RTD experiment was performed using I-131 as a radiotracer. The radioisotope was obtained as potassium iodide (KI) from the Board of Radiation and Isotope Technology (BRIT), Mumbai. The radiotracer I-131 has a half-life of 8 days (gamma energy: 0.36 MeV) which was suitable for tracing the liquid phase in large scale activated sludge

process as it minimizes the radioactive decay errors during the experiment (Kim *et al.*, 2005). The characteristics of the radiotracer have been listed in Table 3.1.

**Table 3.1 Properties of the radioisotope**

<b>Isotope</b>	<b>Half-life</b>	<b>Radiation energy (MeV)</b>	<b>and Chemical form</b>	<b>Tracing of the phase</b>
Iodine-131	8.04 day	Gamma: 0.41(99%)	Potassium Iodide	Liquid

## 3.2 Equipment

### 3.2.1 Industrial scale Activated Sludge Process

The industrial scale tracer experiment was conducted on the secondary treatment unit of the effluent treatment plant at M/s Shreyans Paper Ltd, Ahemedgarh, Punjab, India. The system consisted of two sets of aeration tank connected in series with a secondary clarifier. The schematic diagram of the radiotracer experiment on the effluent treatment plant has been shown in Figure 3.1. The real industrial scale ASP system has been shown in Figure 3.2 to Figure 3.5. The dimensions and process parameters of each system have been mentioned in the Table 3.2. The aeration tank-1 was 76 m long, 18 m wide, 4 m deep with a capacity of 5472 m<sup>3</sup>. The effluent from a primary clarifier enters into the aeration tank with a flow rate of 5.21±1% m<sup>3</sup>/min. Dissolved oxygen (DO) concentration is maintained between 1.5 and 2.0 mg/L with the help of diffused aeration system fitted with fine bubble ethylene-propylene-diene monomer (EPDM) membrane connected to a centrifugal blower. Six mechanical surface aerators are installed in the tank to homogenize and ensure adequate mixing of wastewater and air (oxygen) in the aeration tank. The secondary clarifier-1 was a circular settling tank having a diameter of 18 m, height of 3.71 m and capacity of 944 m<sup>3</sup> with a peripheral feed mechanism. The treated wastewater from the aeration tank-1 flowed into the secondary clarifier-1 which acts as a settling tank where biomass settles at the bottom. About 20% (1.04 m<sup>3</sup>/min) of the underflow from the secondary clarifier-1 is recycled back to the aeration tank-1 to maintain the mixed liquor suspended solids (MLSS) concentration of 3500 mg/L in the aeration tank-1. The aeration tank-2 was 45 m long, 16 m wide and 4.5 m deep with a capacity of 3240 m<sup>3</sup>.

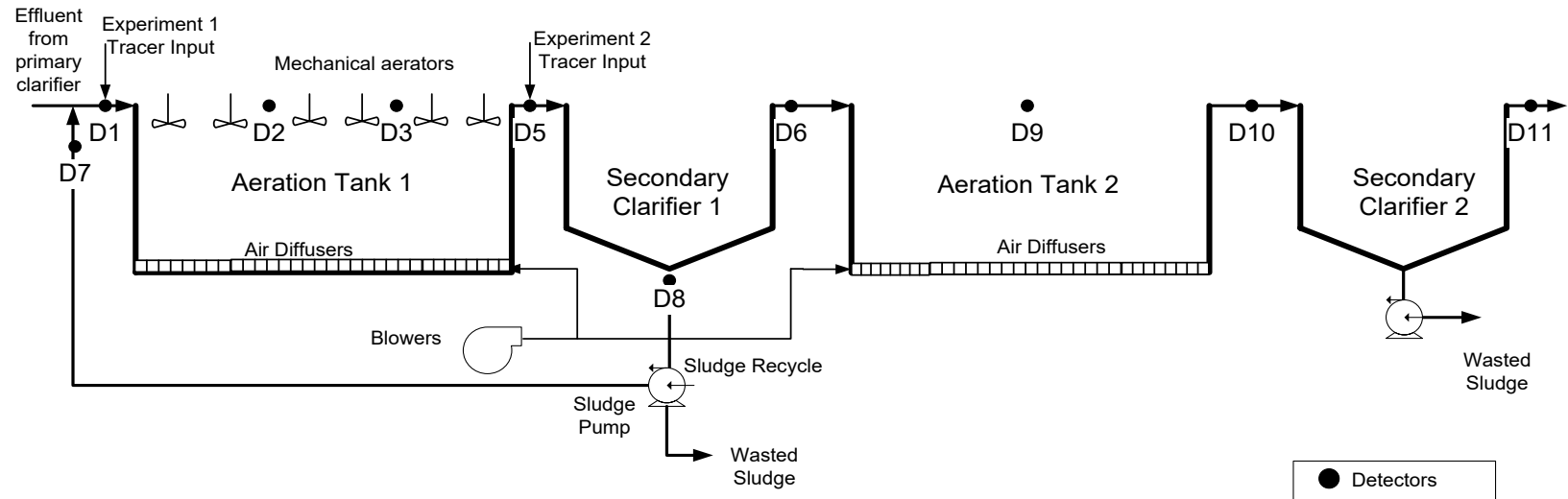


Figure 3.1 Schematic diagram (front view) of the complete industrial ASP setup at Shreyans Industries.



**Figure 3.2 Industrial scale aeration tank-1**



**Figure 3.3 Industrial scale secondary clarifier-1**



**Figure 3.4 Industrial scale aeration tank-2**



**Figure 3.5 Industrial scale secondary clarifier**

It was equipped with a diffused aeration system fitted with EPDM membrane to sparge dissolved air. The effluent feed rate in the aeration tank was 5.21 m<sup>3</sup>/min. The secondary clarifier-2, with a capacity of 890 m<sup>3</sup>, was a circular settling tank with a central feed of 18 m in diameter and a height of 3.5 m. A part of settled biomass was subsequently removed from the bottom of the secondary clarifier-2 and used as organic manure. The treated wastewater is obtained from the overflow of secondary clarifier-2.

**Table 3.2 Dimensional and process parameters of the ASP system at Shreyans Industries**

System	Dimensions (m)	Volume	Flow rate (m <sup>3</sup> /min)	Retention time (min)
Aeration Tank-1	76X18X4	5472 m <sup>3</sup>	6.2 ±1%	881 min (14.7 h)
Secondary Clarifier-1	d=18, h=3.7	944 m <sup>3</sup>	6.2 ±1%	152 min (2.5 h)
Aeration Tank-2	45X16X4.5	3240 m <sup>3</sup>	5.21 ±1%	622 mins (10.4 h)
Secondary Clarifier-2	d=18, h=3.5	890 m <sup>3</sup>	5.21 ±1%	171 mins (2.8 h)

### 3.2.2 Detectors

Radiotracer once injected in the system was monitored by on-line measuring technique continuously using NaI scintillation detectors of dimensions 2” x 2” (height x diameter). The detectors used were gamma-ray scintillator having thallium- activated sodium iodide NaI (TI) single crystal. The detectors were placed in a waterproof casting and dipped at various location in the ASP and connected to a desktop for data logging and visualization.

### 3.2.3 Data Accusation System

The data acquisition system (DAS) is the basic equipment for online radiotracer decay monitoring. The data acquisition system ensures the collection, treatment, and visualization of

the data that pertains to the real-time experiment and provides simultaneous recording of data with the minimal dwelling time of 1-2 milliseconds. PC or laptop based data logger can be used as standard portable data acquisition systems for industrial radiotracer experiments. It collects signals from the detectors and display the acquired RTD curves on a screen. DAS is essentially a multi-counter unit that provides reading of radiotracer in counts/time. The number of channels in DAS can vary from 5 up to 32 channels depending upon the number of detectors connected to the DAS. It is a microcontroller based instrument with a software package to control the data acquisition, data storage and retrieval process. The data in counts that is acquired during one run was simultaneously transferred to PC through cables and later stored on PC.

### **3.2.4 Pilot-scale ASP design**

The schematic diagram of the lab scale ASP is shown in Figure 3.6. The pilot scale ASP system constructed has been shown in Figure 3.7. The aeration tank was a stainless steel tank having dimensions 60 X 15.5 X 15 cm with a working volume of 12000 cm<sup>3</sup>. Diffused aeration system was set in the aeration tank by fitting perforated tubes of diameter 0.5 cm along the length of the tank to provide aeration. An oil free compressor was attached to the aeration line through a rotameter to regulate air flow in the system and maintain the desired dissolved oxygen (DO) level in the aeration tank. Outlet channel of the aeration tank is connected to the clarifier. The clarifier was a long cylindrical tube of diameter 15.2 cm and height 45 cm. The inlet, outlet and recycle line of the ASP are connected with 0.5 mm pipes. Two peristaltic pumps (capacity 0.1-10 L/h) synchronized to a control panel were used to pump the wastewater from storage tanks to the aeration tank and the recycle stream from the bottom of clarifier to aeration tank (AT), respectively.

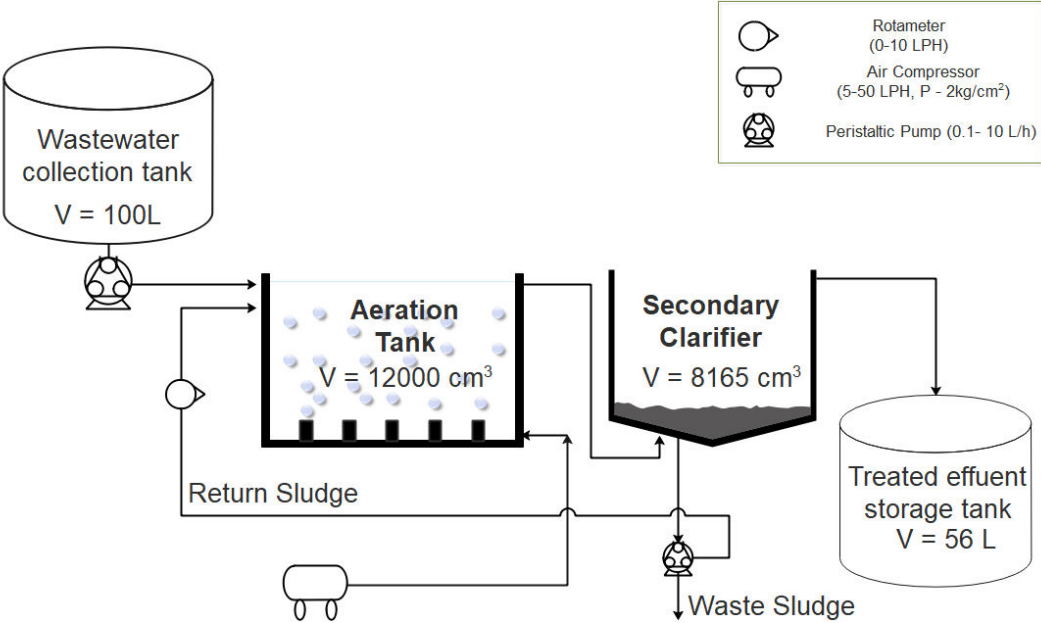


Figure 3.6 Schematic diagram of pilot scale ASP



Figure 3.7 Pilot scale ASP system

### 3.3 Methodology

#### 3.3.1 Radiotracer detection and measurement in Industrial scale ASP

Two sets of RTD experiments were performed on the ASP system. In first run, eight (NaI) (TI) scintillation detectors D1, D2, D3, D4, D5, D6, D7 and D8 as shown in Figure 3.1, were used to study the hydrodynamics of aeration tank-1 and complete ASP (aeration tank-1 and secondary clarifier-1). To initiate the RTD experiment, 5 mL of Iodine-131 (I-131) radiotracer having activity 30 mCi was diluted in about 2 litre of wastewater and instantaneously injected at the inlet of the aeration tank 1 as shown in Figure 1. Since monitoring locations were quite far from one another, it was not possible to connect all the detectors to one common data acquisition system (DAS) due to limited length of the cables connecting the detector to DAS system. Hence, the detectors were connected to two independent DASs and set to record the radiotracer concentration with a sampling time of 5 min. Both the DASs were synchronized and started at the same time to acquire the data. The background radiation levels were measured prior to conducting each run.

The second RTD experiment, 5 mL of I-131 radiotracer was injected at the inlet of the secondary clarifier-1 and monitored at the inlet and outlet of the secondary clarifier 1, the aeration tank 2 and secondary clarifier 2 using the detectors D5, D6, D9, D10, and D11 (Figure 3.1). The data acquisition systems were connected to independent laptops and set to record data points with a sampling time of 5 minutes. The duration of the experiment was 50 hours, which was approximately 3 times longer than the theoretical/expected mean residence time, to obtain a proper description of flow.

#### 3.3.2 RTD formulation and data correction

The correct RTD formulation is the basic requirement for further data processing, modelling and interpretation. The RTD curves obtained from the radiotracer experiment often contain statistical fluctuations and other errors that can manipulate the accuracy of the data (IAEA, 2008; Kasban *et al.*, 2010). Several treatment methods are applied to the raw data before it can be used for modeling purposes. The correction applied for our RTD data are summarized as in the subsequent sections.

### 3.3.2.1 Dead time correction

All detectors have a minimum quantity of time that is needed to distinct gamma photons so that they may be recorded as discrete pulses. This minimum time required may be due to the detection technique or related to efficiency of the electronic devices. The relation between the true count rate  $n_t(t)$  and the measured  $n_m(t)$  count rate is given by Equation (3.1)

$$n_t(t) = \frac{n_m(t)}{1 - \tau n_m(t)} \quad (3.1)$$

This step was used to make certain the consistency of starting point of the statistics at time zero. In this step, the reactors are calibrated so that reactors provide the same count when started.

### 3.3.2.2 Background correction

Before injecting the radiotracer into a system, it is necessary to measure the background radiation level. The counts taken before the injection were removed from the data count and the first point was count after the radiotracer was injected in the system.

### 3.3.2.3 Radioactive decay correction

The radiotracers decay exponentially with time, it was necessary to apply decay correction to the measured data. The decay corrected count rate  $n_c(t)$  is given by Equation (3.2)

$$n_c(t) = n_m(t) \exp(\gamma t) = n_m(t) \exp\left(\frac{0.693t}{T_{1/2}}\right) \quad (3.2)$$

$\gamma$  is the decay constant,  $t$  is the time and  $T_{1/2}$  is the half-life of the radioisotope tracer. If the decay correction is not applied, we might get erroneous tracer concentration when the concentration after half-life starts decreasing by the end of experiment. Applying the decay correction ensures that the tracer concentration has reached a constant stage uniformly.

### 3.3.2.4 Filtering (or smoothing)

Filtering is applied to eliminate fluctuations that occurs due to counting statistics or electronic noise. Several strategies for smoothing a sign are available. Cumulating or resampling counts is simpler approach for smoothing fluctuations in RTD data. However, data

smoothing was avoided in our study to carefully study the reason of every peak and variation and relate it to the hydrodynamics of the system under study.

### 3.3.2.5 Tail Correction

Tracer experiment assumes the count rates goes back to zero after the end of the experiment. Data extrapolation is applied when the concentration of tracer at the end of the RTD experiment is not the same as the background radiation recorded at the beginning of the experiment. A very long tail suggest the presence of dead zone and must be correct before modeling the data. Mostly extrapolation is performed mathematically using exponential decay function is given by Equation (3.3)

$$C_t(t) = C(t)e^{-t/x} \quad (3.3)$$

## 3.3.3 Pilot scale ASP study

### 3.3.3.1 ASP start up

The wastewater for the study was collected from the outlet of primary settling tank of a fully integrated pulp and paper mill (M/s Shreyans Paper Ltd, Ahemedgarh, Punjab) in India. The characteristics of the effluent were (mg/L) BOD 540-580, COD 1050-1150, MLSS 2800-3500, and pH 7–7.66. The effluent was stored at 4° C before it was used for the experiment. A range of MHRT were determined based on the previous industrial experiment performed at a full scale integrated pulp and paper mill effluent. The initial effluent inflow rate was set to 0.75 L/h and the system was operated at batch mode to allow the growth of microorganism with regular monitoring of physiochemical parameters. The microbial seed for the inoculating in the aeration tank was collected from the industry. The sludge was aerated for 1–2 days at room temperature before inoculating the reactors. The microorganisms or bacterial growth was allowed to reach the stationary growth phase so as to maintain the MLSS of 3500 mg/L. Fine bubble spargers installed in the aeration tank maintained the dissolved oxygen (DO) concentration between 1.6-2.0 mg/l for optimum growth of the microorganisms.

### 3.3.3.2 Microorganism growth and acclimatization

ASP is carried out in presence of different kinds of microorganisms (MOs). These microorganisms require certain amount of time and particular conditions for adjustment process in new wastewater environment during the startup of ASP. This is often termed as the acclimatization process. The studies on acclimatization process was important, as the proper growth of MOs can enhance degradation of pollutants compound effectively improving the efficiency of the wastewater treatment process. The exposure to a different organic pollutant may slow the growth of microorganisms and hinder the ability to degrade the pollutant. However, an extended period of exposure to the wastewater can allow the microorganism present in the ASP to acclimatize easily in the process (Wiggins *et al.*, 1987; Rao *et al.*, 2005; Zawani *et al.*, 2013).

The wastewater collected from the outlet of primary clarifier and the inoculum or seed was collected from the recycle stream of the ASP of a pulp and paper mill industry were stored in the cold storage at 4 °C. Initially the aeration tank of the pilot scale ASP was operated at a batch mode and wastewater was allowed to fill the aeration tank. The inoculum was diluted with wastewater and aerated in a separate flask for a period of 12 h. For the batch study, the seed was added to the aeration tank maintaining the wastewater to seed ratio of 3:1 and was continuously aerated. During acclimatization study nutrient agar (1g/L) was added at regular intervals to enhance the growth of microorganisms. Other nutrients as urea (1g/l) and Di-ammonium Phosphate (DAP-1g/l) were also added so as to maintain the carbon, nitrogen and phosphate content and to sustain the growth of microorganisms in the reactor. The batch mode of operation was conducted for a period of 35 days until the concentration of microorganism was found to stabilize in the system. Various parameters such as pH, MLSS and COD were estimated on everyday basis to monitor the changes in the growth of microorganisms in the reactor. To avoid the accumulation of metabolic products, the mixed cultures were settled in the secondary clarifier to remove excess sludge from the reactor. In the preliminary stage of ASP start up excessive foaming was found and during the initial test runs. The rate of air supplied to the reactors was sufficient to keep the sludge flocs suspended in the aerator. There was no significant changes found after 40 days of batch mode operation. The trend analysis of pH, MLSS and COD has been studied during the acclimatization process.

### 3.3.3.3 Analytical methods for parametric assessment

Samples were withdrawn at the beginning and end of each set of experiments and monitored at the outlet of the aeration tank and overflow of secondary clarifier. Analyses were carried out in accordance with standard methods for the examination of Water and Wastewater (APHA., 1976). The standard methods were used to measure the TDS and TSS of the pulp and paper effluent. A closed reflux method was used for the COD measurement of the real paper and pulp effluent. COD was measured using digestive unit (thermo reactor, model 2025D, Spectra lab) and double beam UV visible spectrophotometer (HACH, DR 5000, USA). BOD of the sample was measurement using Oxitop BOD bottles (WTW OxiTop® IS 6, BOD package). A benchtop pH / conductivity meter (Benchtop Water Quality Meter – 860033,) was used for the pH and conductivity measurement. All the chemicals used during the present study were of analytical reagent (AR) grade. The efficiency of substrate decay in the aeration tank and complete ASP was determined by the Equation 3.4.

$$\% \text{ substrate removal} = \frac{\text{inlet substrate concentration} - (\text{outlet substrate concentration})}{\text{inlet substrate concentration}} \times 100$$

(3.4)

The substrate concentration at the inlet and outlet sampling points calculated in terms of BOD and COD.

### 3.3.3.4 RTD tracer experiment of pilot scale ASP

Lithium chloride (LiCl) was used as tracer for RTD analysis of pilot scale ASP. Earlier RTD studies performed using LiCl in wastewater treatment units confirmed that lithium is not adsorbed onto microorganisms. The toxicity of LiCl concentration on the growth of microorganism population in the aeration tank was examined by Kirby Bauer method (Biemer, 1973; Bonev *et al.*, 2008). It was observed that the concentration of lithium chloride above 15% can hinder the microbial growth in the aeration tank. Hence the concentration of LiCl was kept below 15% for all the experiment. A 10% LiCl salt solution was prepared in 100 ml of water and instantly poured at the inlet of the aeration tank. The concentration of LiCl was monitored at the outlet of aeration tank (sampling point S1) and overflow outlet of secondary

clarifier (sampling point S<sub>2</sub>) initially at an interval of 10 mins and then at an interval of 1 hr. The recycle concentration to the aeration tank was varied to 10% and 20% and RTD experiment were performed for concentration, C(t) vs t curve and modelled to obtain suitable hydrodynamic models for the system.

### 3.4 RTD system analysis

#### 3.4.1 Calculation of RTD moments

After treatment the E(t) curves were plotted for the experimental RTD data for the outlets of each individual reactor, given by Equation (3.5).

$$E(t) = \frac{C(t)}{\int_0^{\infty} C(t)dt} \quad (3.5)$$

where, C(t): concentration of tracer (counts of radiotracer) at time t and E(t): normalized RTD curve.

Moments are applied on RTD data to characterize the RTD functions in terms of statistical parameters such as mean residence time, standard deviation etc. The moments in general are defined as:

$$M_i = \int_0^{\infty} t^i E(t)dt$$

Where, i = 0, 1, 2...

The zero<sup>th</sup> moment of the normalized RTD gives the area under the curve, which is equal to unity, given by Equation (3.6).

$$M_0 = \int_0^{\infty} E(t)dt = 1 \quad (3.6)$$

For a perfect impulse injection of radiotracer, the first moment of the RTD curve will directly provide the MHRT of the wastewater in the reactor, that indicates the average time spent by the wastewater in the system (Levenspiel, 2001), given by Equation (3.7).

$$\bar{t} = M_1 = \int_0^{\infty} t E(t)dt \quad (3.7)$$

The theoretical MHRT ( $\tau$ ) of the system was the desired hydraulic time considered at the time of the design of the system at normal operating conditions, and is the ratio of the geometric volume of the reactor to the total volumetric flow rate fed into the system (Battaglia *et al.*, 1993), given by Equation (3.8).

$$\tau = \frac{V}{Q_o + Q_R} \quad (3.8)$$

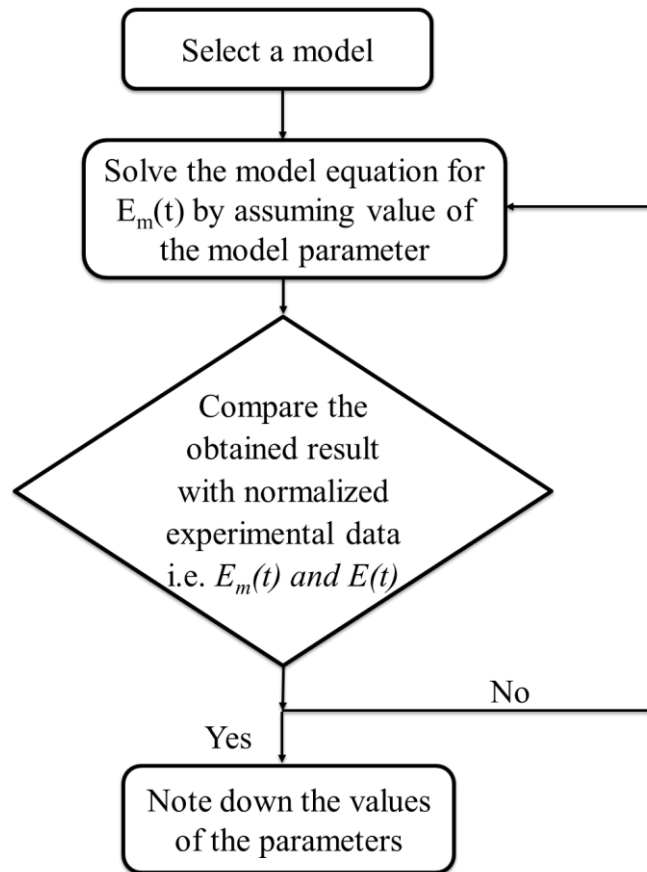
Where, V: volume of the system,  $Q_o$ : flow rate of waste water into the system,  $Q_R$ : recycle flow rate of wastewater. Under normal operating conditions, the theoretical and experimentally measured MHRT should be equal (Levenspiel, 2001). If the theoretical MHRT is lesser than the experimental MHRT, this either implies that a fraction of the radiotracer is held back in the system and is released gradually or the values of flow rates and volume used for determination of  $\tau$  are erroneous. Thus, the values of flow rates and volume need to be rechecked. In case, if the value of experimentally determined MHRT is lesser than the theoretical MRT, then their exist presence of dead volume i.e. the volume which is not utilized for flow of wastewater in the system. The presence of the dead volume decreases the hydraulic efficiency of the system. The extent of dead zone in the system can be estimated by comparing the experimental and theoretical MHRTs of the system (IAEA, 2008), given by Equation (3.9).

$$\% V_d = \left(1 - \frac{\bar{t}}{\tau}\right) \times 100 \quad (3.9)$$

### 3.4.2 RTD modeling

RTD experimental data can be further analyzed to predict the hydrodynamic behavior of the reactor by applying various available flow models. Modeling of RTD experimental data means to represent the behavior of the system in terms of ideal reactors and calculate the possible deviation. RTD flow models provides a quantitative description of hydrodynamic characteristics of the transported material and helps in understanding of a process and its prediction for additional conditions. Modeling of the experimental RTD curve with theoretical representations of different flow patterns can performed using different software. The arrangements of basic flow elements are used to provide a proper model that gives a response

identical to the tracer data obtained from the tracer experiment from the system under study. RTD models quantifies the deviation of the system from the ideal nature. The selection of a RTD flow model depends on the type of reactor, extent of mixing, resemblance to ideal reactor, reactor design parameter etc. There are various RTD models available in literature, which include ideal model such as plug flow model, perfect mixers model, one parameter models such as tank-in-series model, axial dispersion model and more complex multi parameter models like with tank –in-series with recycling model, tank-in-series with exchange model, etc. We have assumed that the real wastewater treatment reactors behaves as intermediates between pure convection (plug flow) and pure mixing (perfect mixer). A generic procedure of RTD modelling has been presented in Figure 3.8. Several models for the description of hydraulic behavior have been proposed in the present study have be described below.



**Figure 3.8 A generic procedure of RTD modelling ( $E_m(t)$  and  $E(t)$ : model and experimental RTD function respectively)**

### 3.4.2.1 Tank in series (TIS) model

In case of a perfect mixer (or perfect mixing cell), tracer is assumed to be mixed instantaneously and uniformly in the whole volume of system. As in aeration tank the mechanical aerators and diffusers are employed to maintain homogeneous mixing in the tank, TIS model was the first choice during model selection. This model has one parameter, i.e. number of tanks,  $N$ . The model equation is given by Equation (3.10)

$$E_m(t) = (t)^{N-1} \frac{N^N}{\tau^N (N-1)!} \exp\left(\frac{-tN}{\tau}\right) \quad (3.10)$$

The parameter  $t$  is any time,  $\tau$  is the theoretical MRT and  $N$  signifies the number of mixing tanks the system can be represented. As the value of  $N$  gets large, impulse response gets closer and closer to the axial dispersion flow model. For large values of  $N$ , following equivalence relationship is often applied to Peclet number,  $Pe$  and  $N$ , given by Equation (3.11)

$$Pe \approx 2. (N-1) \quad (3.11)$$

### 3.4.2.2 Tank-in-series with back-mixing model (TISBM)

The tanks-in-series with back-mixing model (TISBM) was found suitable for modelling of aeration tank to account for the extent of mixing in the aeration tank. For  $N$ , number of tanks in series and  $\alpha$  being the back-mixing ratio, for the  $N^{\text{th}}$  tank (Equation 3.12),

$$\frac{dC_n}{dt} = [(1 - \alpha)C_{n-1} + \alpha C_{n+1} - (1 + 2\alpha)C_n] / \tau_1 \quad (3.12)$$

For  $N^{\text{th}}$  tank,

$$\frac{dC_N}{dt} = (1 + \alpha)(C_{N-1} - C_N) \quad (3.13)$$

$n$  is a whole number, i.e.  $n = 2, 3, 4, \dots, (N-1)$ ,  $\alpha$  the back flow ratio is defined as the ratio of exchange flowrate between two tanks ( $q$ ) and total flow rate entering the system ( $Q$ ),  $\tau$ : mean residence time in each tank,  $(V/NQ)$ ,  $V$  is total volume of each tank.

### 3.4.2.3 Axial dispersion model (ADM)

In plug flow reactors, it is assumed that matter flows without any dispersion. In other words this flow is pure convection. However, industrial system that are supposed to act as ideal plug flow reactor deviates from its behavior. This deviation can be quantified by axial dispersion model (ADM). In ADM the flow is considered to be superimposition of convection and some amount of dispersion. The model has two parameters, time constant  $\tau = L/U$ , where  $L$  is the length of the system, and non-dimensional Peclet number,  $Pe = (U.L)/D$ , that represents the ratio of the convective to dispersive effects. In other words, dispersion is predominant when  $Pe$  is low and negligible when it is large. The inverse of  $Pe$  number,  $D/U.L$ , called the vessel dispersion number is also used to characterize the system parameters. ADM model was suitable to characterize the deviation of secondary clarifier. The open-open boundary conditions were chosen for modeling since there was no abrupt change in the flow conditions at the inlet point of tracer introduction and outlet boundaries for each of the tubes wherein the detectors were placed. The equation used to obtain model RTD function  $E_m(t)$  given by Equation (3.14)

$$E_m(t) = \frac{1}{2} \left( \frac{Pe}{\pi\tau t} \right)^{\frac{1}{2}} \exp \left( - \frac{Pe(\tau - t)^2}{4\tau t} \right) \quad (3.14)$$

Axial dispersion model was used in combination with TIS model to characterize the behaviour of the complete ASP. ADM model was also used along with convoluted signals for non-ideal pulse tracer inputs to subsequent reactors.

#### 3.4.2.4 Compartment model

Compartment models are based on the combination of ideal CSTR and PFR arranged in different configurations. The structure of the compartment model resembles as closely as possible the actual flow pattern of the reactor. The parameters of the compartment model are adjustable and can be predicted following multiple simulations with the experimentally obtained RTD data. The parameters can be used to predict the macroscopic flow model and mixing pattern in complex systems as the ASP.

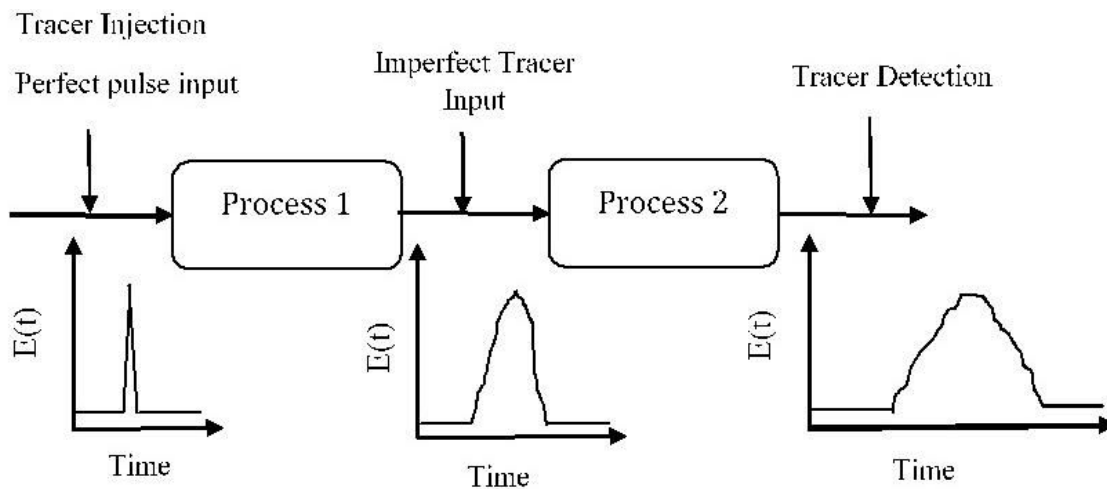
### 3.5 Convolution Technique

In industries, the wastewater treatment systems are interconnected and process material leaving from one reactor served as input ( $\tau$ ) to the succeeding one (Figure 3.9). The tracer input to

the subsequent systems acts non-ideal impulse input and can't be directly evaluated for further RTD analysis. Convolution technique is applied for the RTD analysis of non-ideal impulse tracer input.

### 3.5.1 Benefits of convolution technique for non-ideal tracer input

Mathematically convolution can be defined as the operation between two functions or signals, that produces a third function that is correlated to the original function. The radiotracer entering into a process from the previous reactor serves as an imperfect input impulse and the output signal gets modified. Convolution method may be used to obtain non-distorted E-curves from RTD curves of the reactors when input and output signals of the system are unknown.



**Figure 3.9 Interference with input signal due to a non-ideal impulse input**

### 3.5.2 Convolution integral

Convolution is the mathematical transformation that allows to predict the response to any injection history outlet concentration ( $C_0(t)$ ) from the known  $E(t)$  data. This method makes it possible to express the response to the whole injection sequence outlet concentrations as the sum of the responses to each individual injection step. The radiotracer entering into a process

from previous reactor serves as an imperfect input impulse and the output signal gets modified. This signal modification is related to the E-curve of this vessel as in the convolution integral, is given in Equation (3.15).

$$C_{out}(t) = \int_0^t C_{in}(t - t')E(t')dt' \quad (3.15)$$

This equation can be used to obtain non-distorted E- curve for the system when the input and output RTD signal of the system are known. The radiotracer experiments performed on the industrial ASP, the treatment reactors were connected in series. Hence, tracer output from one reactor served as the input the successive reactors. The tracer input to these reactors were then imperfect impulse input and the RTD models suggested for them were convoluted to obtain optimized model curves.

### 3.5.3 Estimation of parameters

To determine the model parameters of a convoluted model, the model suggested for the particular reactor was convoluted with the input tracer signal to the reactor. The output of this convoluted model was optimized with the output obtained from the RTD experiment. The model fitting was done by least square method of curve fitting and the model parameters were calculated for the model that minimizes the sum of the squares of the differences between the original obtained experimental RTD data and suggested RTD model.

## 3.6 Optimization procedure — Curve fitting method

The models selected to represent the hydrodynamics of wastewater treatment reactors generate model  $E_m(t)$  as a function of time with unknown variable parameters. Modeling requires to match to the  $E_m(t)$  to the experimental RTD curve and obtain the corresponding model parameters. The optimization (curve fitting) of the experimental RTD  $E_{exp}(t)$  with the model  $E_m(t)$  evaluates the model parameters. The model RTD function is fitted with the experimental RTD curve by using the least square curve fitting method given in Equation (3.16).

$$\varepsilon = \int_0^{\infty} [E_{exp}(t) - E_m(t)]^2 dt = \text{minimum} \quad (3.16)$$

The values of the model parameters corresponding to the minimum value of the squares of the differences are chosen as the best fitting. The superiority of the curve fit is judged by selecting the model parameters to minimize the sum of the squares of the differences between the RTD function of experimental  $E_{\text{exp}}(t)$  and model  $E_{\text{m}}(t)$  data.

### 3.7 RTD Software

There are commercial and homemade RTD software for modeling experimental RTD curve. The RTD software DTSPRO-V4.21 developed by PROGEPI was used for the modeling of experimental RTD curves. The objective of the software is to provide means to simulate the outlet of flow from a reactor using conventional as well as compartment models technique. The software support preliminary treatment of the tracer curves including background correction, exponential extrapolation, and normalization and automatically estimates moments of both experimental and theoretical curves. The software offers the possibility for the users to build any physically representative model for the system under investigation and subsequently use the same to simulate the experimentally measured data. A software comprises of three windows, the model (project) window to create the model for simulations, the curves (project) window that allows representation of both experimental  $E_{\text{exp}}(t)$  and model  $E_{\text{m}}(t)$  results and the table (project) window which allows to import experiment RTD data for preliminary treatment and save the results of simulation. The model window provides eight elementary reactor modules (perfect mixing cell, perfect mixing cells in series, perfect mixing cells in series exchanging with dead volume, perfect mixing cells in series with back mixing, plug flow reactor, plug flow reactor with axial dispersion open to the diffusion, plug flow reactor with axial dispersion closed to the diffusion and plug flow reactor with axial dispersion half open to the diffusion) that can be used as individual units or in combination to form a compartment model. These reactor modules are connected to each other with help of nodes and branches. The branch is oriented in the direction of the flow from the nodes to the reactor. This software is very useful in determining model parameters in complex reactors and in simulating and estimating the mass balance with multiple recirculation, bypassing and recycle systems.

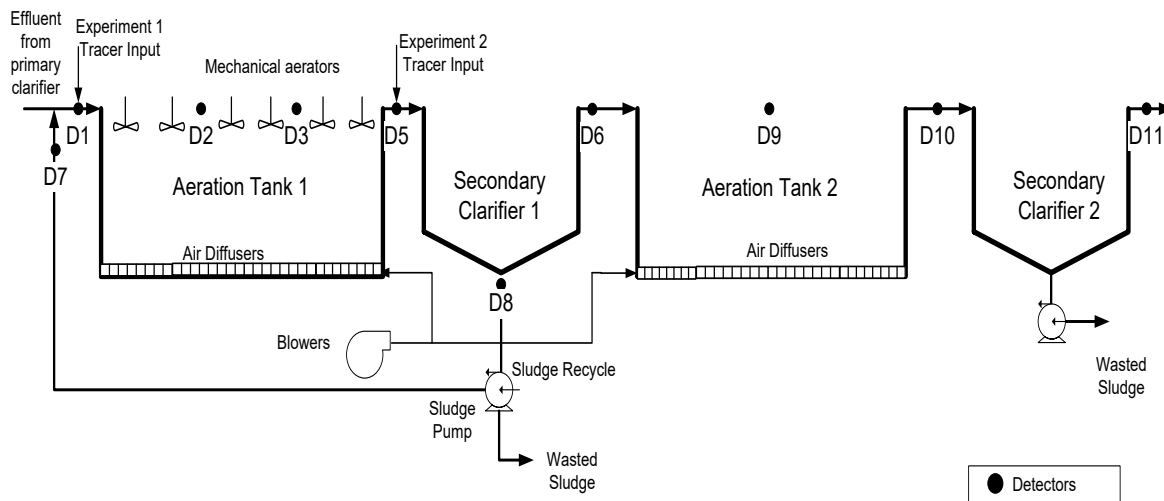


## INDUSTRIAL SCALE RTD EXPERIMENTS AND MODELING

Industrial-scale residence time distribution (RTD) measurement and analysis were performed for a full scale activated sludge process, consisting a combination of the aeration tanks and secondary clarifiers and hydraulic performance of the process was evaluated.

### 4.1 RTD Experiment on the Industrial scale wastewater treatment plant

The industrial ETP was spread out in a large area, so the RTD analysis was distributed into two separate radiotracer experiments (Run -1 and Run-2) to evaluate the complete secondary wastewater treatment system. The first radiotracer experiment (Run 1) was performed to evaluate the aeration tank-1 and secondary clarifier-1 and complete ASP (aeration tank 1+ secondary clarifier-1). For Run-1, the radiotracer was injected at the inlet of the aeration tank 1 (D1) and monitored at the outlet of aeration tank-1 (D5) and secondary clarifier-1 (D6). For Run-2, the radiotracer was injected at the inlet of secondary clarifier-1 (D5) and monitored at the outlet of secondary clarifier-1 (D6), aeration tank-2 (D10) and secondary clarifier-2 (D11). The schematic diagram of the complete setup of the secondary treatment unit has been given in Figure 4.1.



**Figure 4.1 Schematic diagram (front view) of the complete industrial scale secondary wastewater treatment system.**

#### **4.1.1 Modification and refining of data obtained from radiotracer experiments**

In the radiotracer experiment performed, the signal obtained from the scintillation detectors contained statistical disturbances and was not fit to be directly used for data modeling and simulation. Hence, various pre-treatment methods were applied on the raw experimental RTD data to refine it for further analysis.

##### **4.1.1.1 Pre-treatment of experimental data**

In order to eliminate these experimental errors and noises, basic signal modifications like dead-time correction, background correction, and data extrapolation were performed on the experimental RTD data. It was observed that the experimental RTD data had some unnecessary peaks, fluctuations, background radiations data and long tails that has to be eliminated before further analysis. Some other signal modification referred to in the literature like decay correction and data smoothing were not applied for the particular experiment. The radiotracer used for the experiment was I-131, have a half-life of 8.4 days. The RTD experiments were conducted for a period of 6 days and hence decay correction was not necessary. Moreover, the major fluctuations in the experimental RTD curves were due to the anomalies and were a part of the analysis. Hence, data smoothing was not applied and only unusual peaks were eliminated.

The original and modified experimental RTD curves obtained at the end of the aeration tank-1, secondary clarifier-1, aeration tank-2, and secondary clarifier-2 have been shown in Figure 4.2 (a-h), respectively.

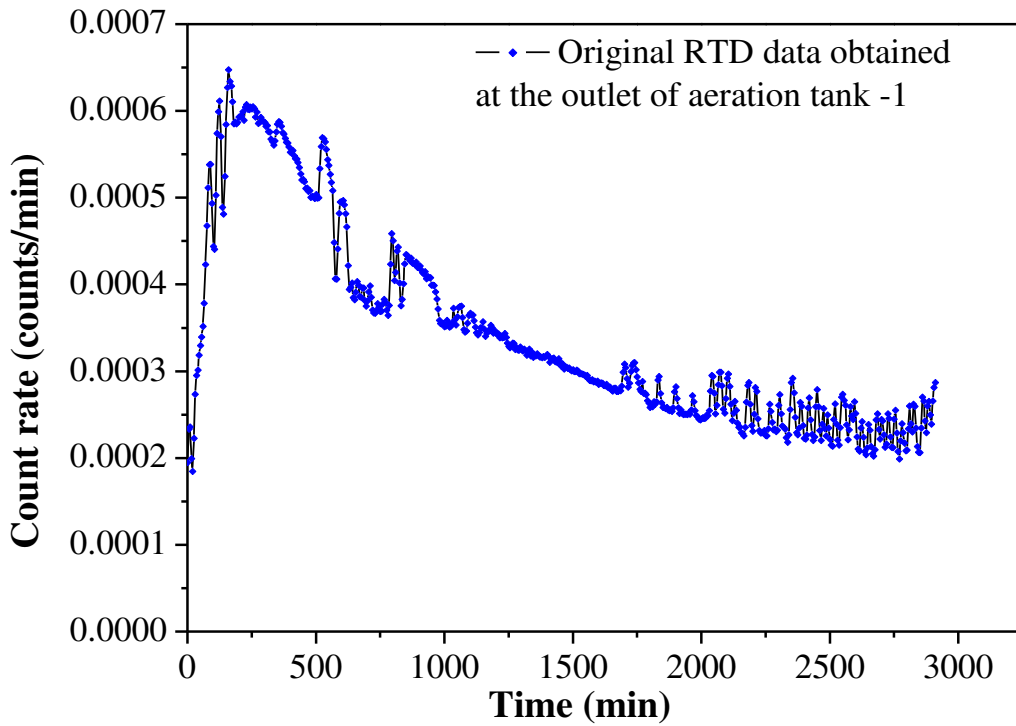


Figure 4.2 (a) Radiotracer signal obtained from aeration tank-1.

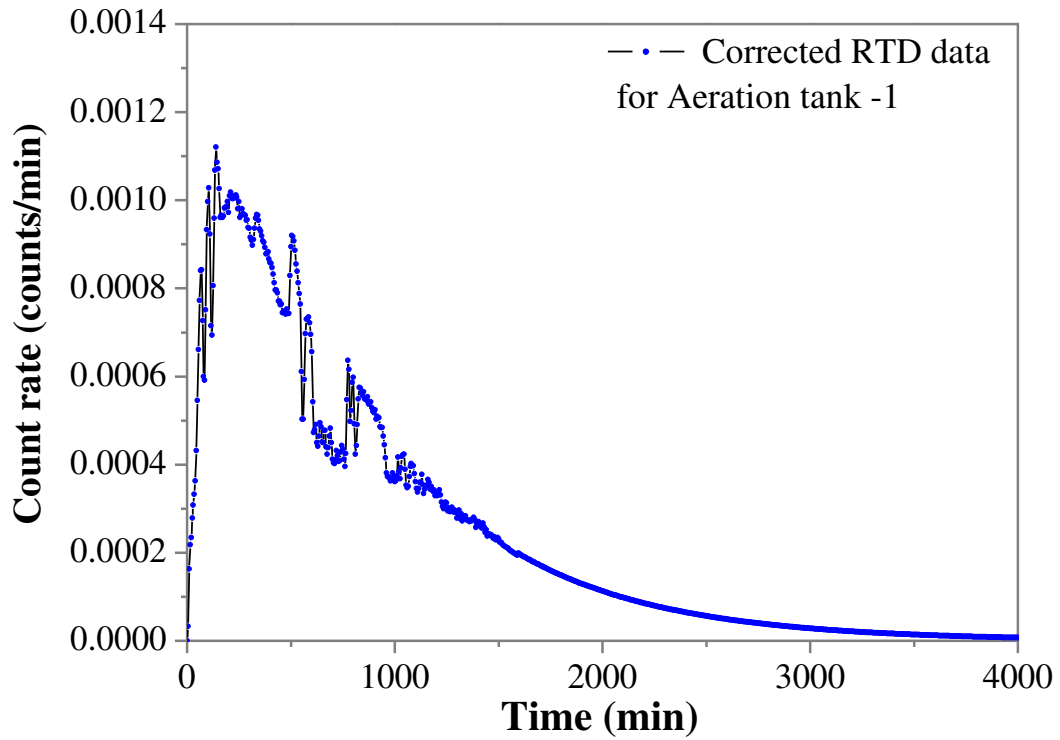


Figure 4.2 (b) Corrected radiotracer signal obtained from aeration tank-1.

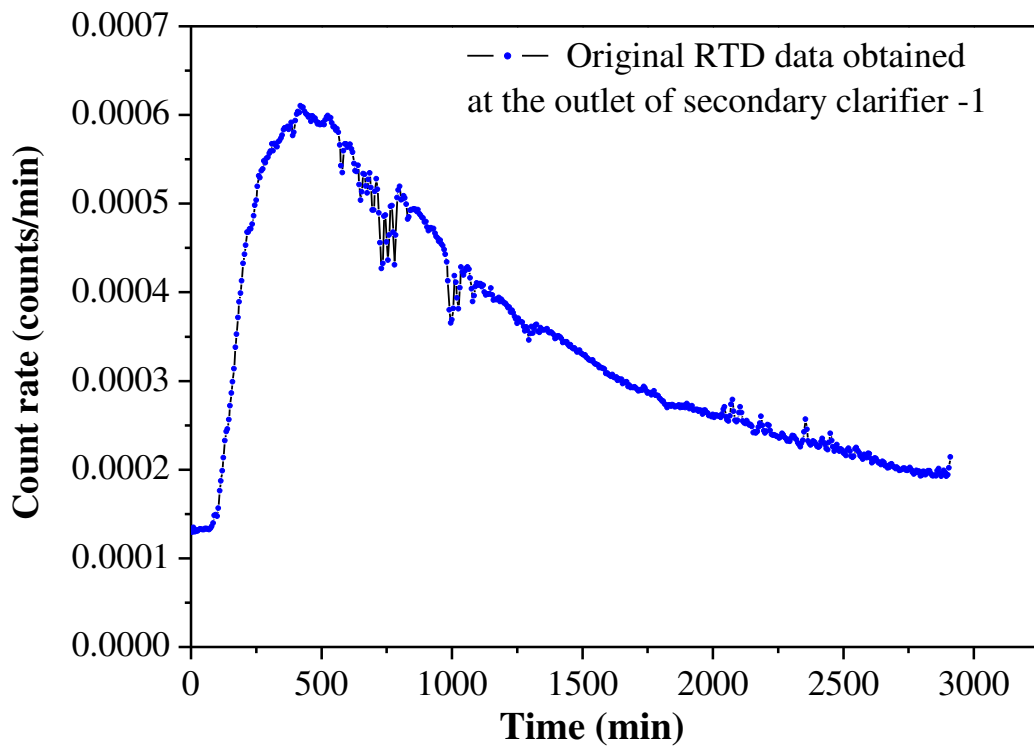


Figure 4.2 (c) Radiotracer signal obtained from secondary clarifier-1.

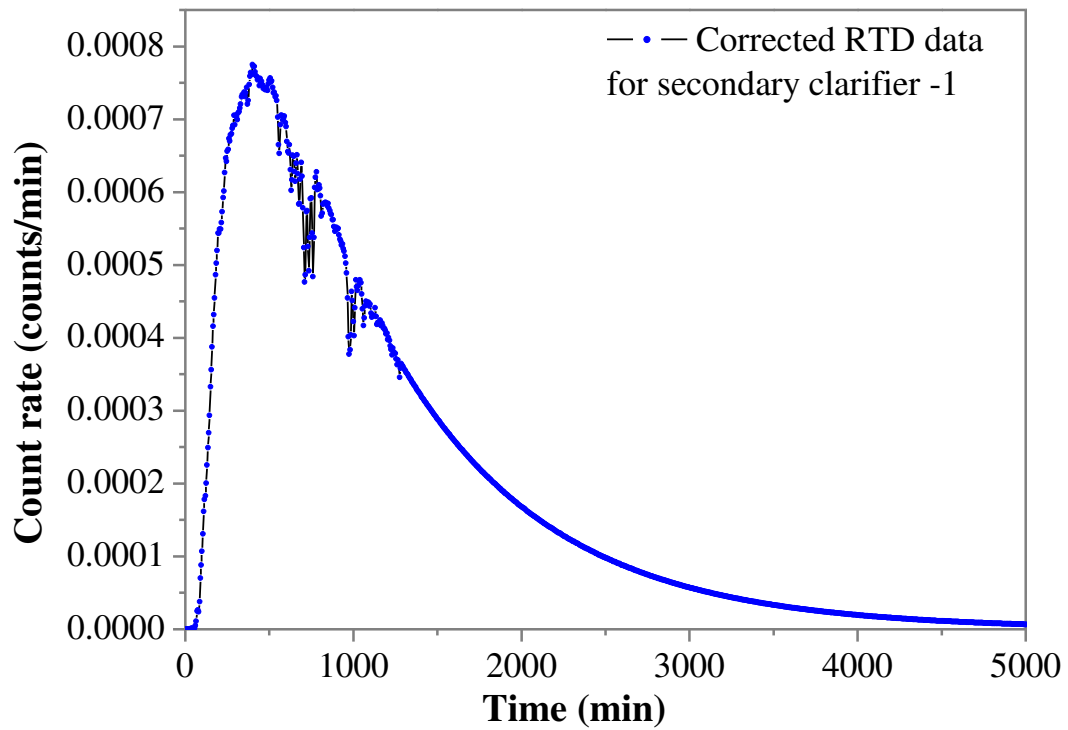


Figure 4.2 (d) Corrected radiotracer signal obtained from secondary clarifier-1.

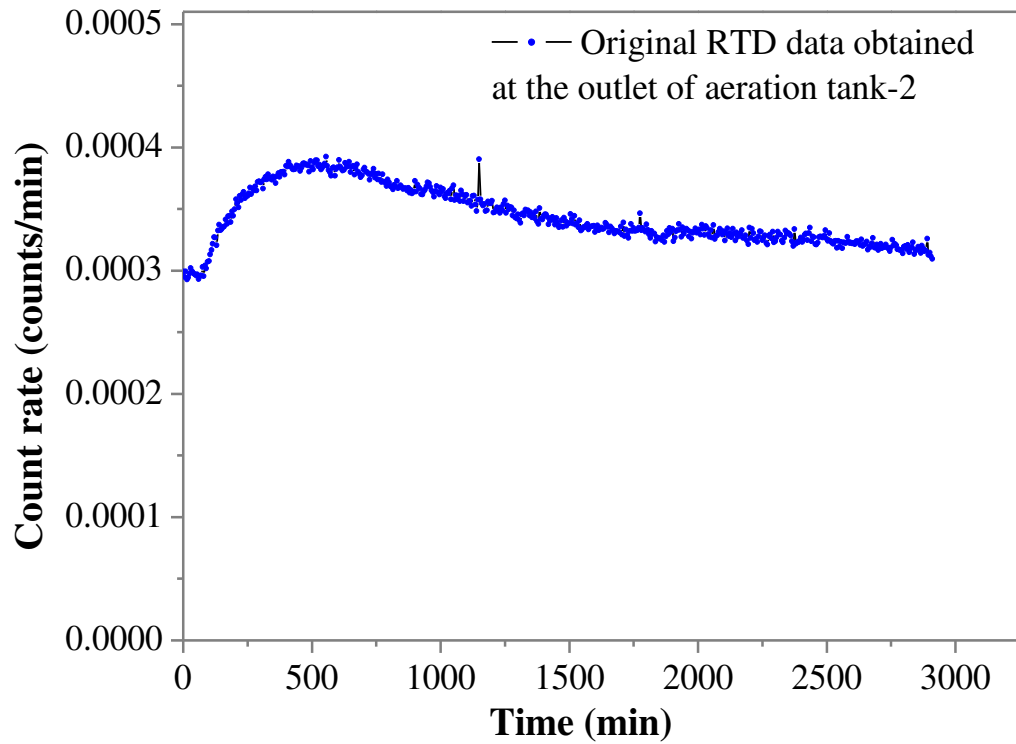


Figure 4.2 (e) Radiotracer signal obtained from aeration tank-2

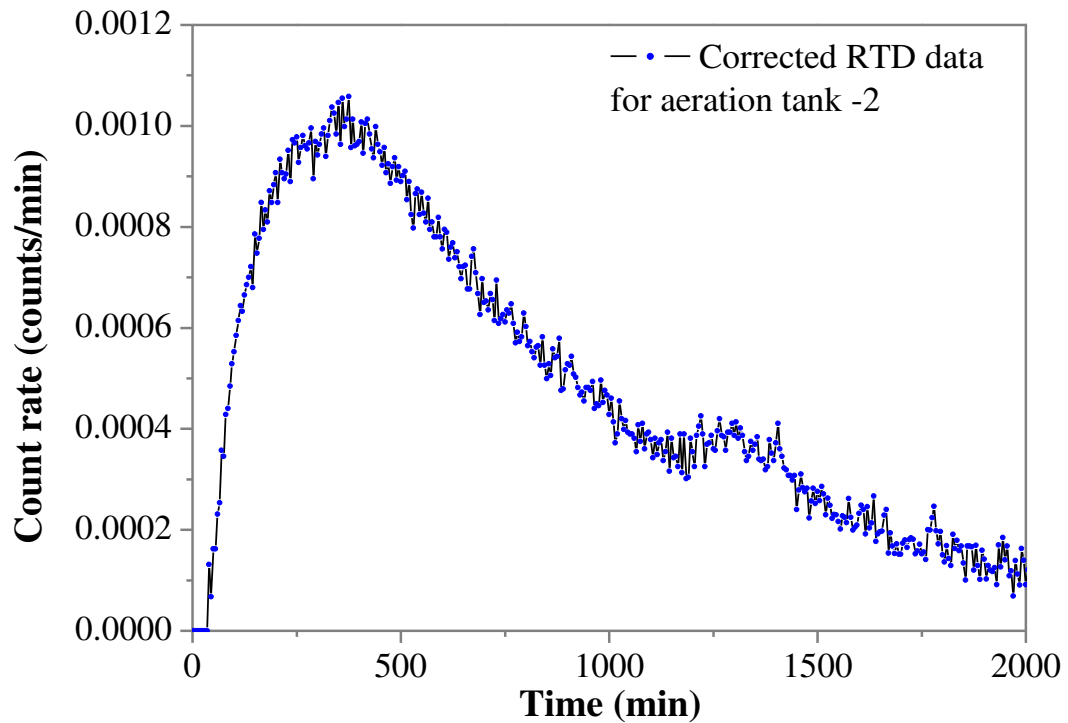


Figure 4.2 (f) Corrected radiotracer signal obtained from aeration tank-2

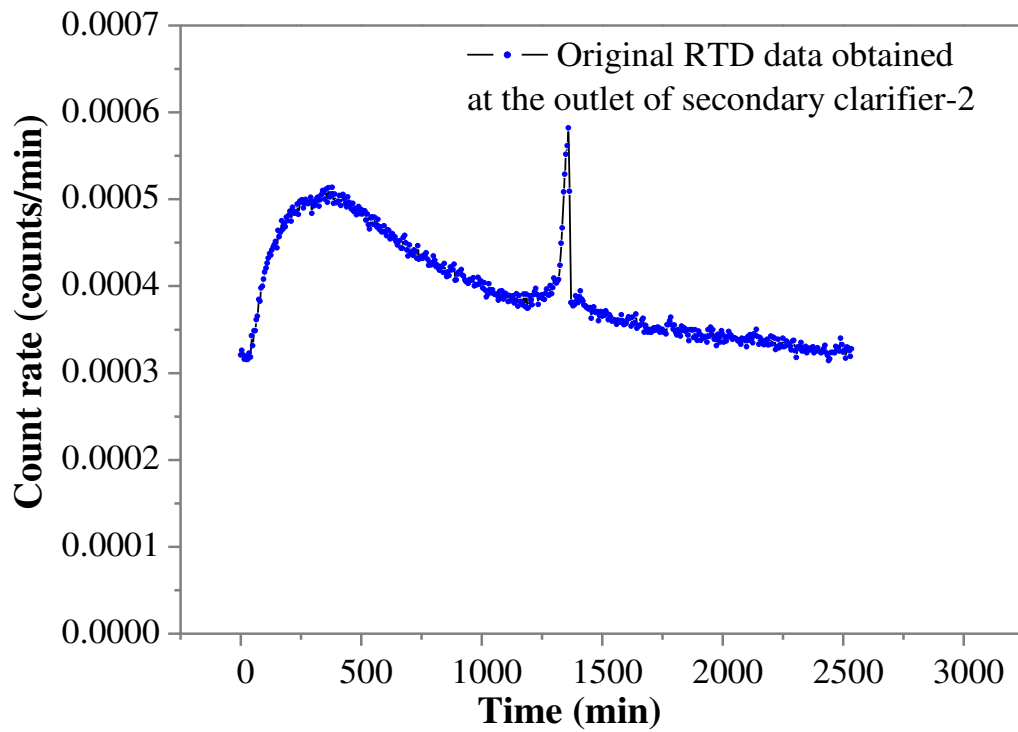


Figure 4.2 (g) Radiotracer signal obtained from secondary clarifier-2

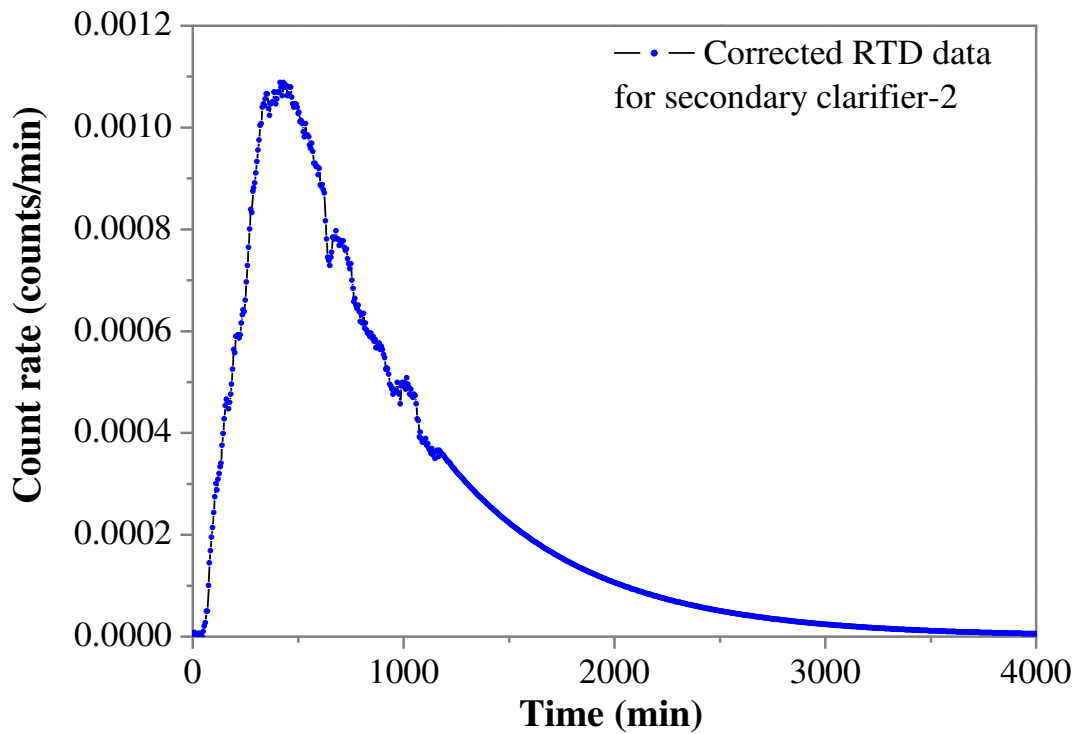


Figure 4.2 (h) Corrected radiotracer signal obtained from secondary clarifier-2

#### 4.1.1.2 Calculation of Moments

The experimental RTD data was obtained in the form of counts  $C(t)$  vs time  $(t)$  curves that were changed to RTD function  $E(t)$ , which denotes the zero<sup>th</sup> moment of RTD. The RTD function  $E(t)$  is also referred to as exit age distribution function or the washout function represents a probability function that represents in a quantitative manner, the amount of time every individual fluid matter spends inside the reactor. The second moment of RTD is the mean residence time (MRT), or in our study is the mean hydraulic residence time (MHRT) as only the liquid phase was traced. The experimental mean hydraulic residence time ( $\tau$ ) for each reactor was calculated from the  $E(t)$  data and was compared to the theoretical MHRT ( $\bar{t}$ ). The theoretical and experimentally calculated MHRTs of the reactors has been given in Table 4.1.

**Table 4.1 Experimental and theoretical mean hydraulic retention times (MHRT)**

Sr. No.	System	V (m <sup>3</sup> )	Q (m <sup>3</sup> / min.)	$\bar{t}$ (Min.)	$\tau$ (Min.)
1	Aeration tank-1	5472	6.2	881	855
2	Secondary clarifier-1	944	6.2	152	145
3	Entire activated sludge process system	6655	6.2	1073	1175
4	Aeration Tank-2	3240	5.21	622	618
5	Secondary Clarifier-2	890	5.21	171	70

#### 4.2 RTD Modeling

RTD models provide physical representation of the system under study. The RTD data obtained for individual systems as well as entire ASP (aeration tank + secondary clarifier) were fitted with appropriate RTD models to understand the flow structure in the system.

#### 4.2.1 Diagnosis and modeling of RTD data for Aeration Tank -1

The RTD data obtained from the first RTD experiment at the outlet of the aeration tank-1 was evaluated to identify the anomalies in the system. From the shape of the RTD curve monitored at the outlet of the aeration tank-1 (D5), no parallel flow paths were found to exist within the tank. The curve contained three distinct peaks superimposed on the RTD curve that could be due to undesired minor lumped flow produced by the mechanical aerators. However, the extent of lumped flow is so low that does not affect the aeration process. The experimental MHRT of wastewater in the aeration tank-1 was determined to be 855 min. The volume of the aeration tank-1 was 5472 m<sup>3</sup> was and the volumetric flow rate entering the aeration tank during the experiment was 5.21±0.3 m<sup>3</sup>/min. In addition to this, a recycle stream from the secondary clarifier also entered the reactor (Figure 4.3) having a flow rate of 1.04 m<sup>3</sup>/min. Therefore, the theoretical MHRT was estimated to be 881 min. The values of MHRTs are given in Table 4.1. Based on the comparison of theoretical and experimentally measured MHRTs, the dead volume present within the aeration tank was estimated to be about 3%. The amount of dead volume present is negligibly small and within the experimental error. This implies that all most entire geometric volume of the aeration tank-1 is utilized for flow and hence available for the wastewater treatment.

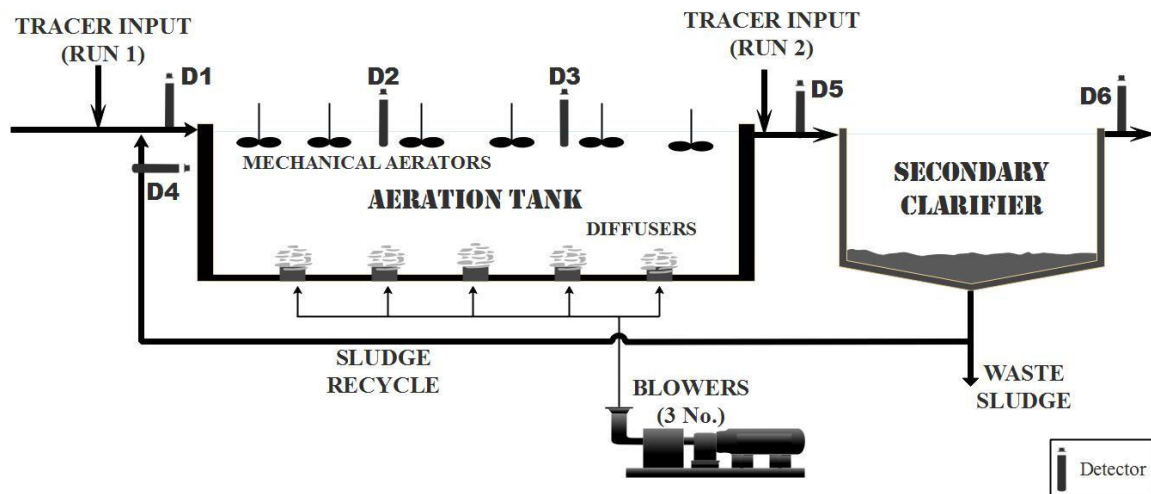


Figure 4.3 Schematic diagram (front view) for first radiotracer experiment (Run-1) in ASP System.

The RTD curve measured in Run 1 at the outlet of the aeration tank (D5) was simulated using a model, whose physical representation has been given in Figure 4.4. The model consists of three building blocks i.e. plug flow component (PFR) connected in series with a tanks-in-series with back-mixing (TISBM) and a recycle stream. The PFR, in the beginning, represents initial shift or time taken by radiotracer to appear at the outlet of the tank without any mixing, whereas the TISBM model represents mixing regime within the aeration tank. The recycle stream is added as a fraction of outlet stream is recycled back to the inlet of the tank and the same is represented by a plug flow component. The radiotracer starts appearing in the recycle stream after about 60 minutes of the injection of the radiotracer. The proposed model was used to simulate the RTD measured at the outlet of the aeration tank (D5) and the comparison of experimental and model simulated RTD curves corresponding to the best fit as shown in Figure 4.5. Several standard models were fitted from the literature using DTS Pro V-4.21. The TIS with backmixing + plug flow model was best fit for Aeration tank 1. The aerator was considered a CSTR and back-mixing was added to account for the backflow of water due to 6 surface aerators installed in the reactor. The plug flow unit in the model accounted for the delay in response obtained during the radiotracer experiment. Several standard models like tank-in-series and ADM and other combination of these models were applied and fitted from the literature and the best fitted model was presented. The model parameters corresponding to the best fits were obtained. The MHRTs predicted by the model in the plug flow component, in the beginning, TISBM component (aeration tank) and the plug flow ( $\tau_p$ ) component in the recycle loop ( $\tau_r$ ) were found to be 10 minutes, 858 minutes and 20 minutes, respectively. The value of tank number (N) was estimated to be 2 indicating that the aeration tank behaved equivalently to two ideal continuously stirred tank reactor (CSTR) with back-mixing (flow in backward as well as forward direction) between them. The back-mixing ratio ( $\alpha$ ) i.e. the ratio of forward to backward flow was estimated to be 2, which signifies that a good degree of axial mixing was occurring in the aeration tank-1. For intense back-mixing, the value of back-mixing ratio tends to be infinity. The MHRT predicted by the model is in good agreement with the experimentally measured MHRT. In order to know the local mixing patterns, the radiotracer concentration was also measured at two axial locations (D2 and D3) within the aeration tank-1.

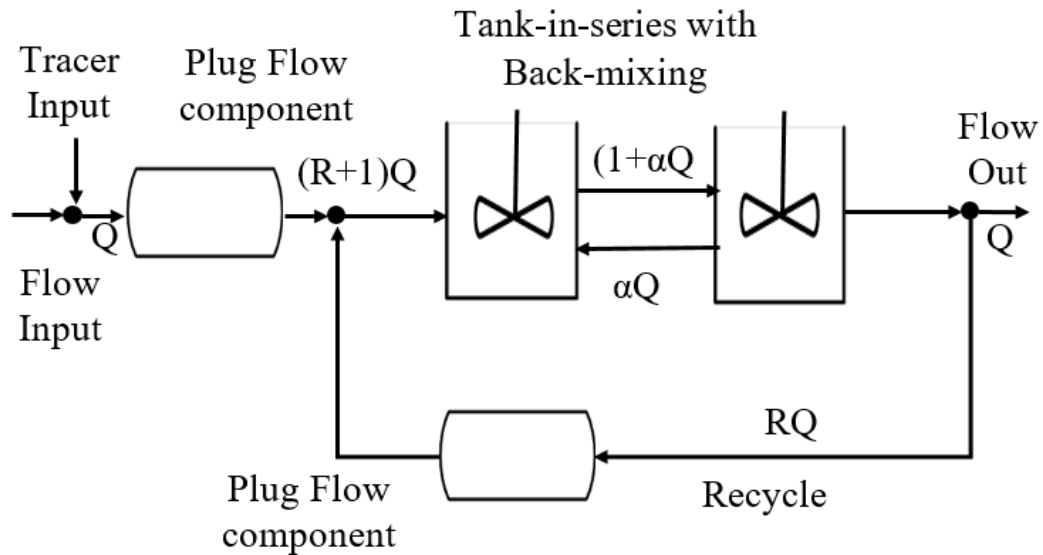


Figure 4.4 Conceptual physical model representation of aeration tank-1

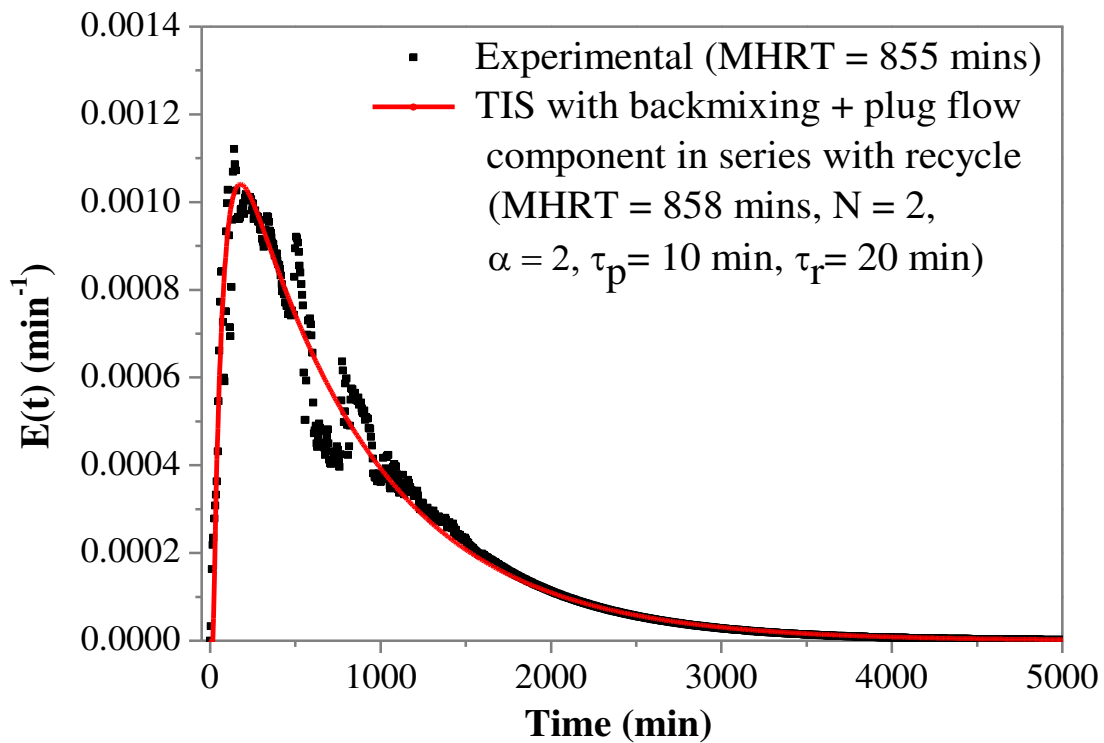


Figure 4.5 Comparison of experimental (D5) and model simulated RTD curves at the outlet of the aeration tank-1 (Run 1)

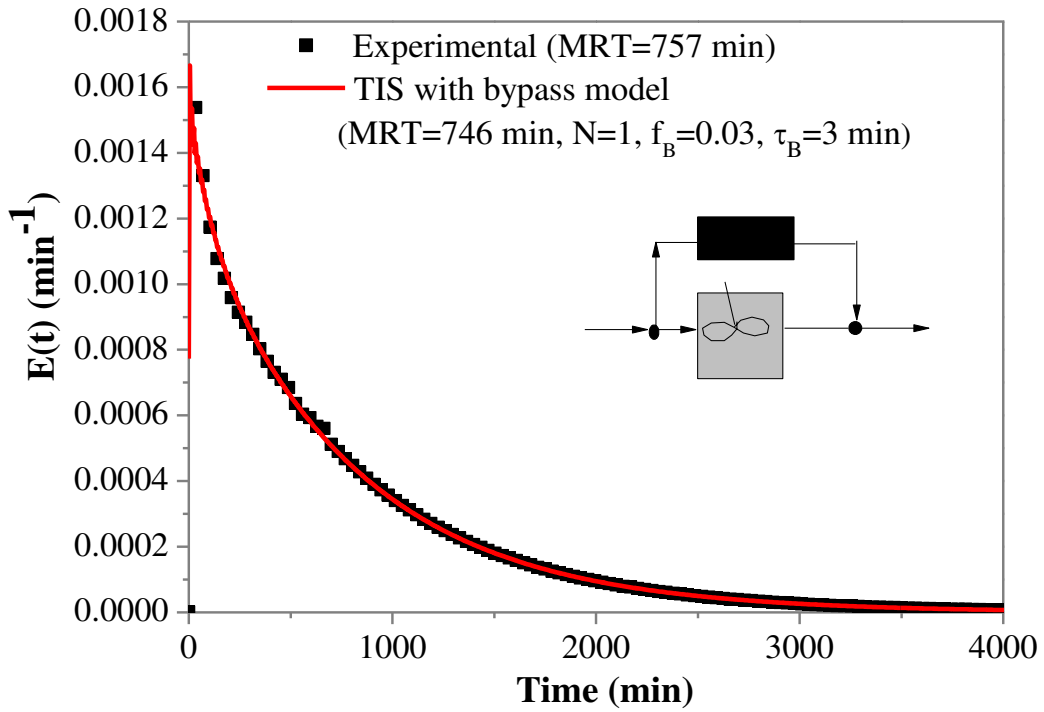
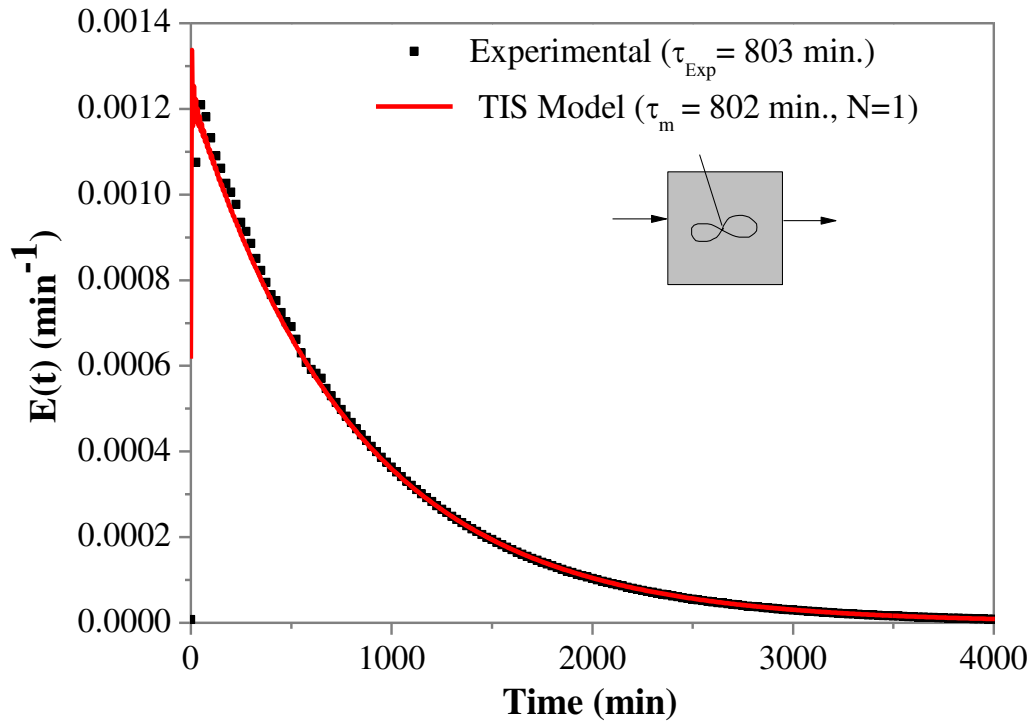


Figure 4.6 Comparison of experimentally measured and model simulated RTDs at location D2 within the aeration tank-1.



**Figure 4.7 Comparison of experimentally measured and model simulated RTDs at location D3 within the aeration tank-1**

The RTD curve measured at first axial location (D2) was simulated using a tank-in-series model with bypassing and the results of the model simulation has been Figure 4.6. At location D2, a small fraction (3 %) of wastewater bypasses the main flow within a very short time i.e. 3 minutes. The RTD curve measured at the second axial location (D3) was simulated using a simple tank in the series model and the results of model simulations has been shown in Figure. 4.7.

**4.2.2 Modeling of Secondary clarifier-1**

During Run 1, the radiotracer concentration was also monitored at the outlet of the secondary clarifier-1, but the measured tracer concentration curves at the inlet (outlet of the aeration tank-1) and outlet of the secondary clarifier-1 were found to be much dispersed and thus not considered analysis as they might cause significant errors in the determination of MHRT and model parameters. Therefore, to characterize the flow of the secondary clarifier-1, second RTD experiment (Run 2) was independently carried out. The radiotracer was instantaneously injected (impulse input) at the inlet of the secondary clarifier-1 (D5) and monitored at the outlet of the secondary clarifier-1 (D6) and the treated RTD curve has been shown in Figure 4.2(d). From the measured RTD, the MHRT of wastewater in secondary clarifier-1 was calculated to be 145 minutes. Since the volume of the secondary clarifier-1 was  $944 \text{ m}^3$  and the total volumetric flow rate entering the secondary clarifier-1 was  $6.21 \pm 0.3 \text{ m}^3/\text{min}$ , the theoretical MHRT was determined to be 152 min. The comparison of experimental and model simulated RTDs indicates that about 4.6 % of the geometric volume of the secondary clarifier is stagnant. This is justified as the sludge (biomass) settles down at the bottom and reduces the active volume of the clarifier. The secondary clarifier should ideally act as a perfect plug flow reactor with some degree of axial mixing and the simple axial dispersion model with recycle was chosen to simulate the RTD data and has been shown in Figure 4.8. The MHRT of the wastewater in the clarifier is much lesser than the residence of wastewater in the aeration tank. Thus, the recycle fraction of the radiotracer had little affect on the RTD curve of the secondary clarifier-1 and thus could be neglected for a practical purpose. Thus the simple axial dispersion model was used to simulate the measured RTD of the clarifier. The

model parameter i.e. Peclet number ( $Pe$ ) corresponding to the best fit was found to be 1.3, indicating a significant degree of back-mixing or non-symmetrical dispersion in the clarifier. This could be due to the diffusion of radiotracer into the stagnant volume (sludge) at the bottom of the clarifier. Figure 4.9 shows the comparison of the experimental and model simulated curves corresponding to the optimum model parameters.

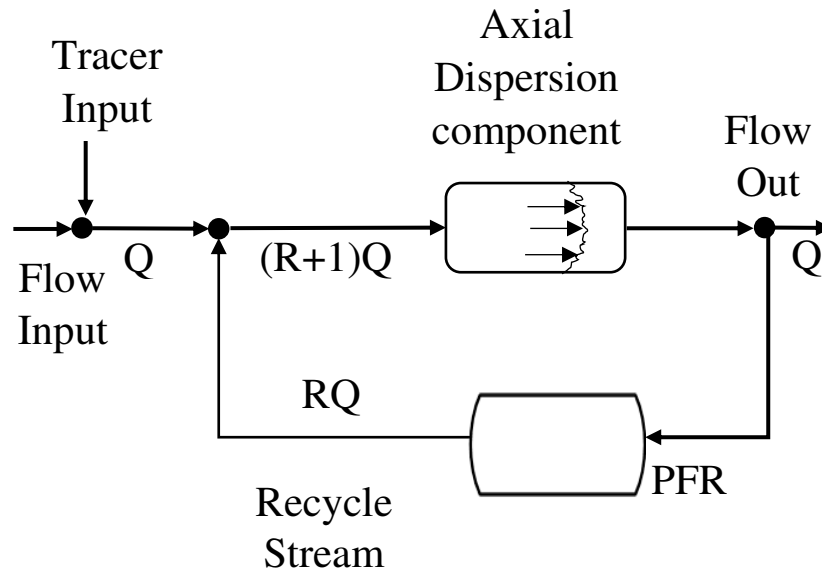
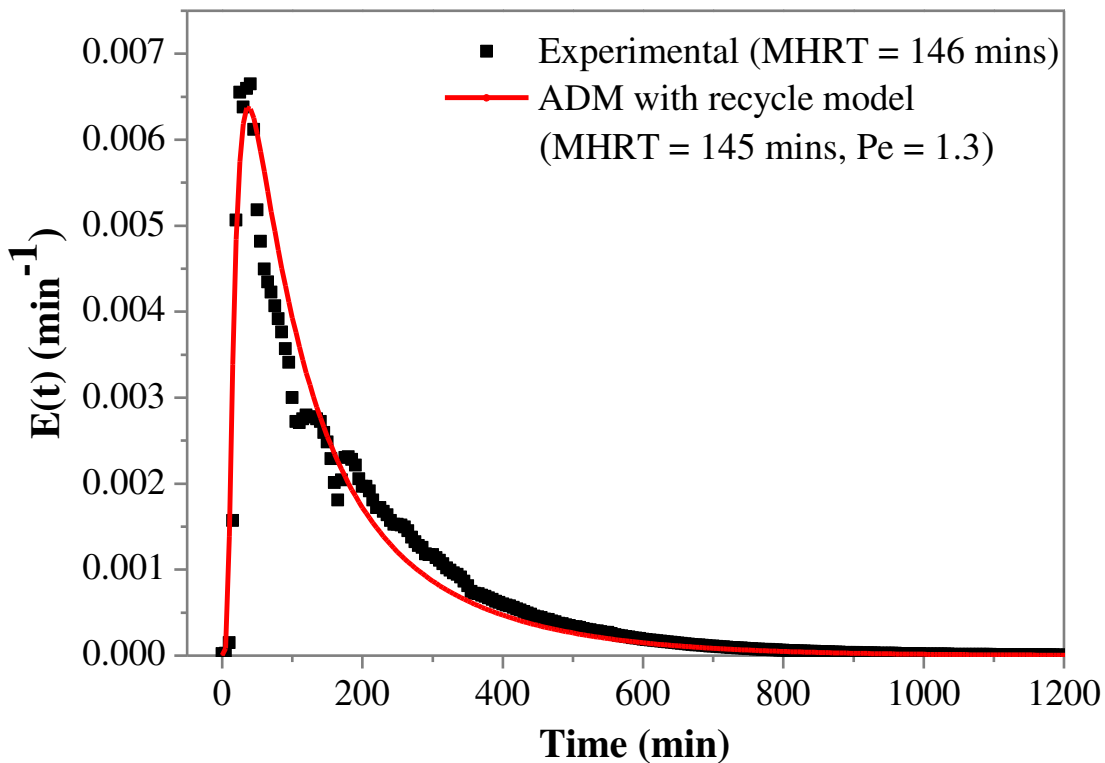


Figure 4.8 Conceptual physical model for the secondary clarifier-1



**Figure 4.9 Experimental and model simulated RTD curves at the outlet of the secondary clarifier-1 (Run 2)**

#### 4.2.3 Modeling of complete Activated sludge process

An attempt was also made to model the entire activated sludge process (aeration tank+secondary clarifier-1). During Run 1, the radiotracer was also measured at the outlet of the secondary clarifier-1 using detector D6 and the measured curve provides RTD of the entire ASP and was independently modeled. The volume of the ASP (aeration tank and secondary clarifier) was  $6655 \text{ m}^3$  and the total volumetric flow rate entering the aeration tank, including the recycle stream during the experiment was  $6.21 \pm 0.3 \text{ m}^3/\text{min}$ . The theoretical MHRT of wastewater in the entire ASP was estimated to be 1073 mins. However, the experimental MHRT obtained for the entire system was 1175 minutes. As the secondary clarifier-1 is a settling tank with negligible mixing, the biomass collected at the bottom of the tank can act as a stagnant zone. A fraction of the radiotracer may get trapped in this stagnant zone and thus will be unable to flow with the active volume (Kim et al., 2005; Pant et al., 2012). Also since the role of recycle stream becomes more prominent while studying the complete ASP, the

recirculation of tracer in the system can be the reason for the higher value of experimental MHRT than the theoretical MHRT. Based on the results of the modeling of the aeration tank-1 and the secondary clarifier-1, a physically representative model as shown in Figure 4.10 was conceptualized and used for modeling of the RTD measured at the outlet of the entire system. The model comprises of a tank-in-series component connected in series with a axial dispersion model component along with a recycle line. The model shows that the secondary clarifier-1 acts as an axial dispersion component with a Peclet no. of 1.1 which is extremely close to the Peclet no. 1.3 obtained when modeled independently. A recycle stream was added to the ASP system, with a recycling rate of 20% of the total flow rate. The model MHRT was found to be 1145 min which is very close to the experimentally obtained MHRT. Figure 4.11 shows a plot showing a comparison of the experimental and model simulated RTD curves corresponding to the optimum model parameters.

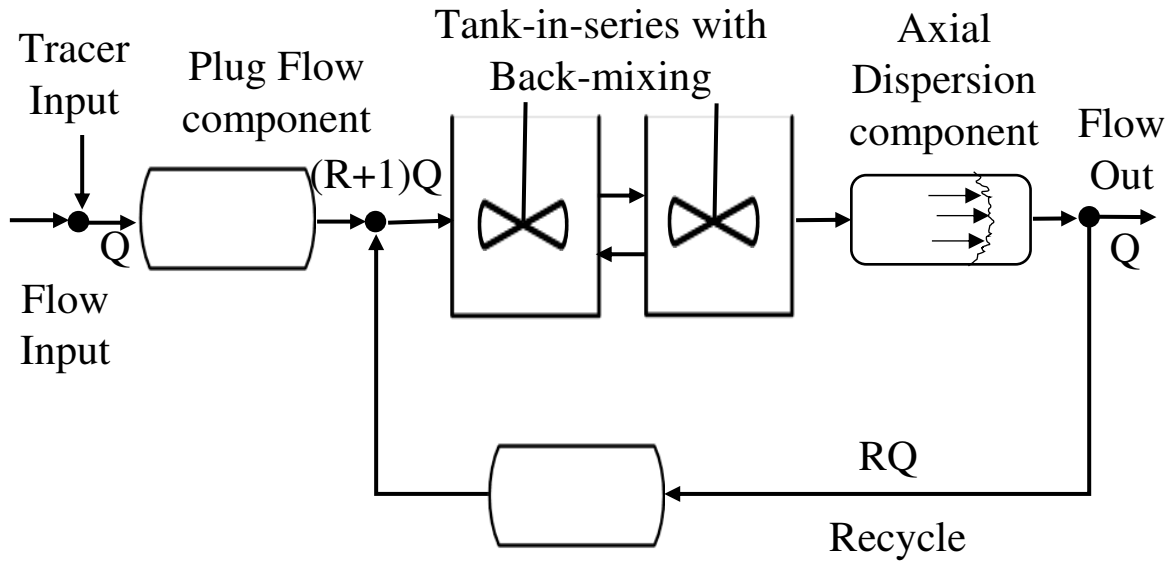
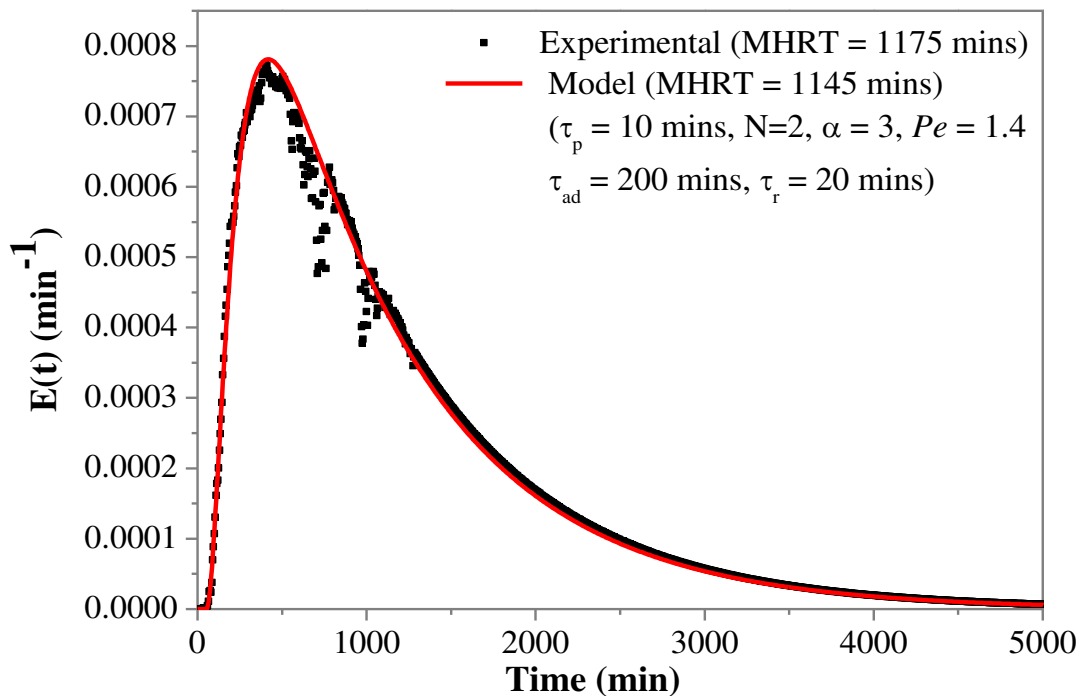


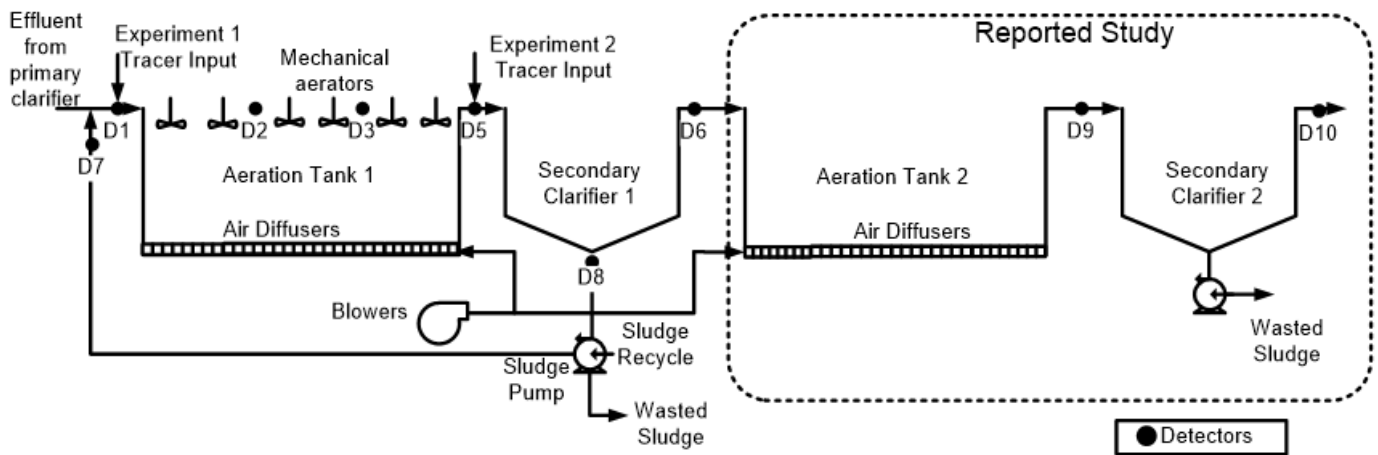
Figure 4.10 Phenomenological model for the entire activated sludge processing system



**Figure 4.11 Experimental (D6) and model simulated RTD curves at the outlet of the ASP system (Run 1)**

### 4.3 Modeling of non-ideal tracer input

The radiotracer injected in the system using pulse input method, directly provided a RTD curve which can be conveniently simulated using the RTD models present in literature (Levenspiel, 2001; Fogler, 2010). However industrial systems are interconnected and the output radiotracer signal from one system acts as the input to the subsequent system. These inputs for continuous connected systems are non-ideal impulse signals that cannot be directly evaluated and only be studied using numerical convolution along with the preferred RTD model. In the second RTD experiment (Run 2), the radiotracer was injected at the inlet of secondary clarifier-1 and was monitored at the outlet of secondary clarifier-1 (D6), aeration tank-2 (D9) and secondary clarifier-2 (D10). The tracer input to aeration tank-2 and secondary clarifier-2 was non-ideal impulse input, and the RTD data was evaluated using convolution of input signal. In this section hydrodynamic study of aeration tank-2 and secondary clarifier-2 has been discussed and the schematic diagram of the reported study has been given in Figure 4.12.



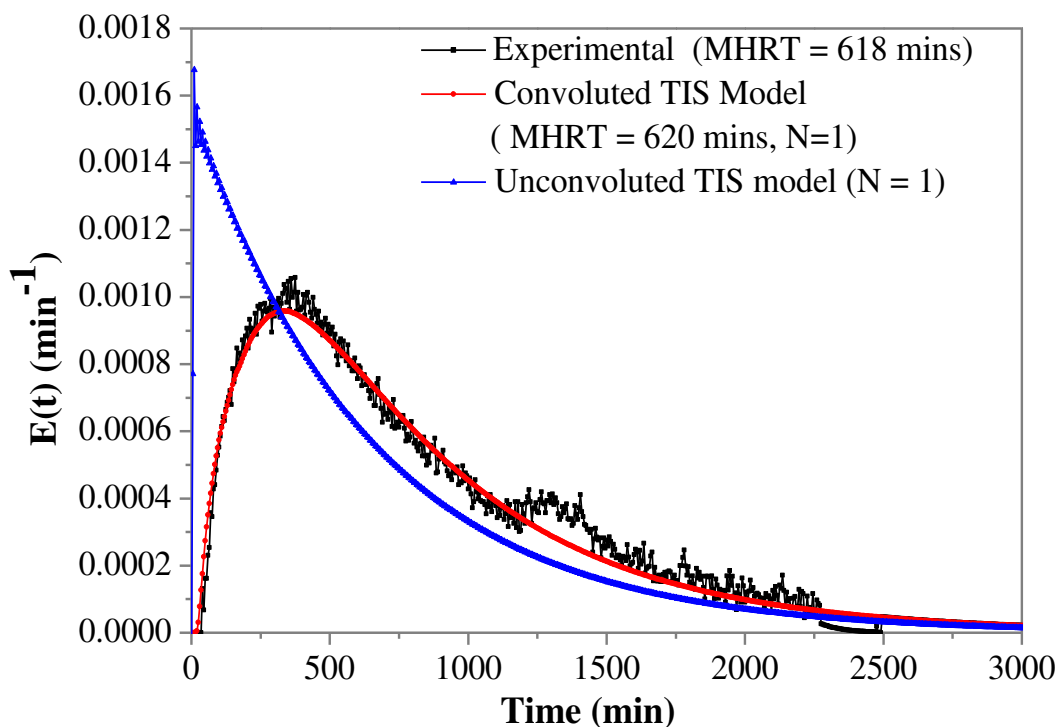
**Figure 4.12 Schematic diagram of system reported for non-ideal impulse input.**

### 4.3.1 Modeling of Aeration Tank-2

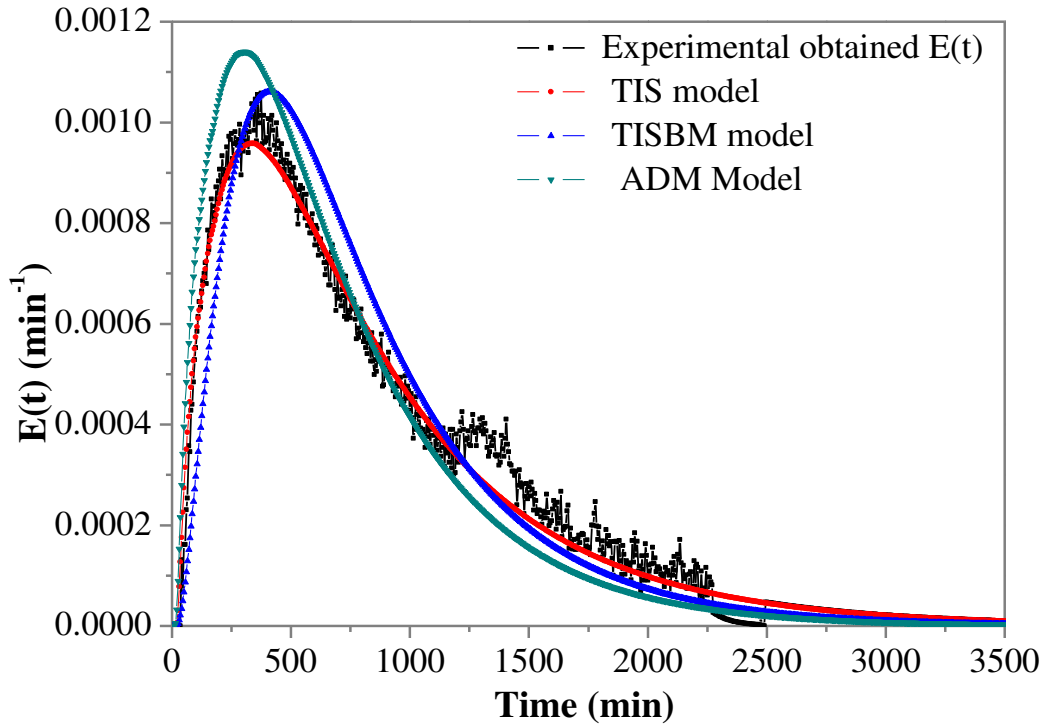
The second experiment performed on the two-stage ASP was monitored at the outlet of aeration tank-2 (D9). The RTD curve obtained at the outlet of aeration tank-2, was pre-corrected to remove any signal disturbances as shown in Figure 4.2(f). As it can be perceived from the Figure 4.12, no equalization tank exists prior to the aeration tank-2, hence a temporary variation of flowrate in the system may have caused the sudden disturbance in signal was observed at the later part of experiment. The RTD curve did not show any multiple peaks which also indicates no specific bypassing and parallel flow paths exist in the system. The volume of the aeration tank-2 was  $3240 \text{ m}^3$  and the volumetric flow rate entering the aeration tank during the experiment was  $5.21 \pm 1\% \text{ m}^3/\text{min}$ . Therefore, the theoretical MHRT was estimated to be 622 min.

The non-ideal impulse response i.e. RTD curve measured at the outlet of the aeration tank-2 was normalized to obtain  $E(t)$  curves. As ideally aeration tank is supposed to act as a perfect CSTR. Tank-in-series and Tank-in-series with back-mixing are the most suitable models to represent aeration tanks (Chmielewski and Selecki, 1979; Le Moullec *et al.*, 2010a). Systems with surface aerators or baffles, often showed significant back-mixing in the tank. Often complex aeration system are also represented by compartment models that consist of combination of CSTR and PFR, to account for dead zone, bypassing and recycle line (Farooq

et al., 2003). Tank in series model was chosen to simulate the tank. The model normalised RTD,  $E_m(t)$  was obtained from the model equation and convoluted against the non-ideal input signal of the aeration tank-2. The model parameter i.e number of tanks (N) was varied to acquire the curve that fits well with the experimental  $E(t)$  curve. The reactor was best represented with single mixing tank (N=1), implying that the aeration tank-1 is acting as an ideal mixing tank with insignificant flow anomalies. The model MHRT was calculated from the model parameters and was found to be 620 min. The comparison of experimental and convoluted model have been shown in Figure 4.13. As the theoretical and experimentally measured MHRTs, are approximately equal, no dead volume is present within the aeration tank. This implies that all most entire geometric volume of the tank is available for the aeration process. Three different RTD models available in literature (i.e TIS, TISBM and ADM) were simulated for the aeration tank as shown in Figure 4.14 and the model parameters have been reported in Table 4.2. The model comparison showed that tank-in-series model was the most suitable fit for the experimental RTD data obtained for aeration tank -2.



**Figure 4.13 RTD curve monitored at the outlet of aeration tank-2 (D9) and model adjustment by convolution.**

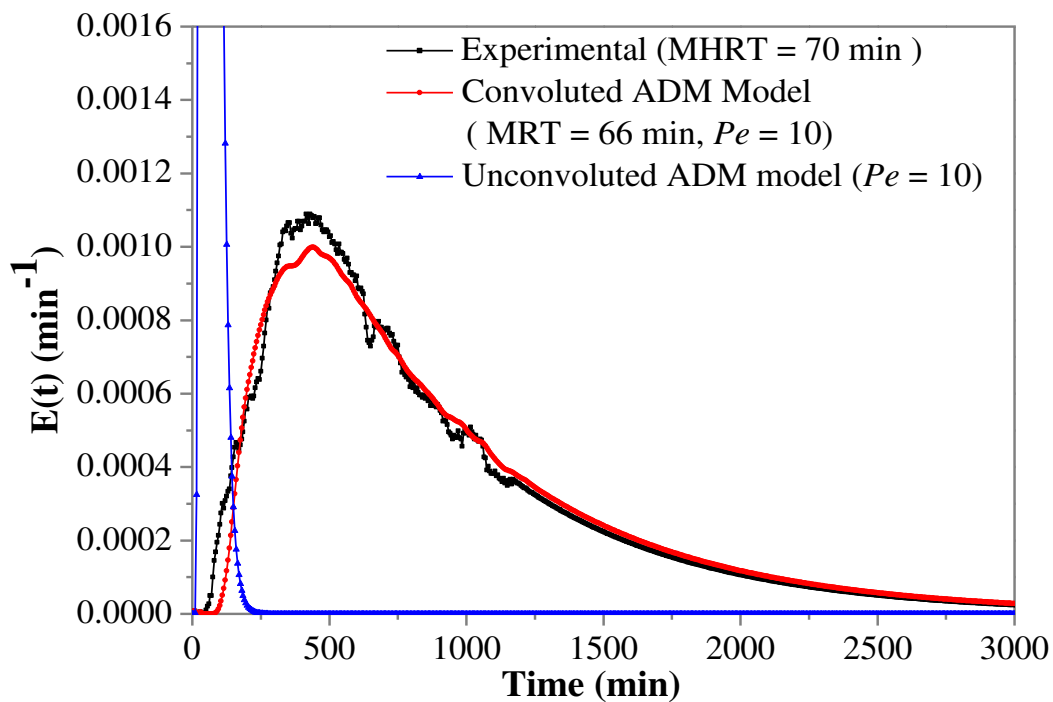


**Figure 4.14 Comparison of RTD models for aeration tank-2**

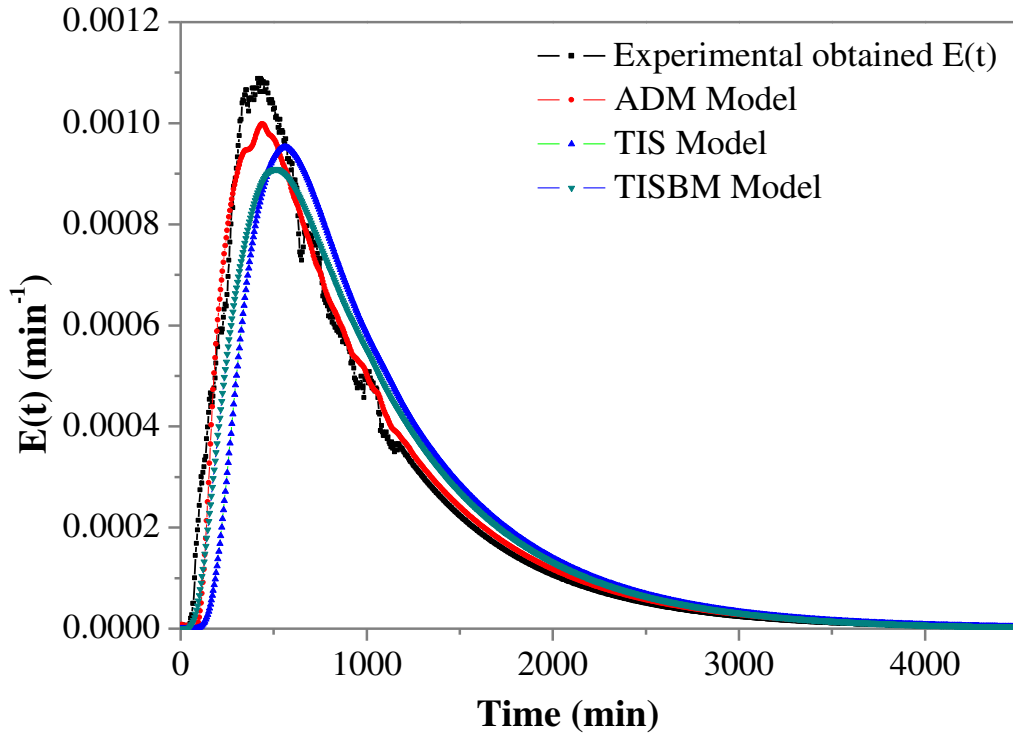
### 4.3.2 Modeling of Secondary Clarifier-2

The aeration tank-2 and secondary clarifier-2 were connected in series, the radiotracer concentration at the outlet of the aeration tank (D9) served as input signal for the clarifier. Since, the volume of the secondary clarifier-2 was 890 m<sup>3</sup> and the total volumetric flow rate entering the secondary clarifier was 5.21 m<sup>3</sup>/min, the theoretical mean hydraulic residence time (MHRT) was determined to be 174 min. The secondary clarifier should ideally act as a perfect plug flow reactor, however deviation from the same can be suitably displayed by axial dispersion model. The axially dispersion model was used to simulate the normalized RTD signal at the outlet of secondary clarifier (D10) and model  $E_m(t)$  was convoluted with the input radiotracer signal of the secondary clarifier (D9). The obtained Peclet number for the axial dispersion component was 10. The high value of Peclet Number indicates that the clarifier is acting as a plug flow reactor. The model MHRT was calculated from the model parameters and was found to be 66 mins. The comparison of the theoretical and experimental MHRT predicts

that more than half of the reactor volume is inactive and unavailable for hydraulic flow. The dead zone in the reactor can be explained due to the accumulation of biomass at the bottom of the tank. The Figure 4.15 shows the comparison of the experimental and model simulated curves corresponding to the optimum model parameters. The comparison of TIS, TISBM and ADM model for the secondary clarifier has been shown in Figure 4.16 and the model parameters have been reported in Table 4.2. The model comparison showed that axial dispersion model was the best fit for the experimental RTD data obtained for secondary clarifier -2.



**Figure 4.15** RTD curve monitored at the outlet of secondary clarifier-2 (D10) and model adjustment by convolution.



**Figure 4.16** Comparison of RTD models for secondary clarifier-2.

**Table 4.2** Comparison of different models and their parameters for aeration tank and secondary clarifier

RTD Models	Aeration Tank-2			Secondary Clarifier-2		
	MHRT (min)	Parameters	R <sup>2</sup>	MHRT (min)	Parameters	R <sup>2</sup>
Tank-in-series Model (TIS)	620	N=1	0.99	71	N=6	0.93
Tank-in-series with back-mixing Model (TISBM)	600	N=2, $\alpha=1$	0.97	72	N=6, $\alpha=0.4$	0.97
Axial Dispersion Model (ADM)	610	Pe =0.1	0.98	70	Pe =10	0.99

The radiotracer technique was found to be suitable to study the hydraulic behaviour of the ASP system in an integrated pulp and paper industry in India. The study revealed that the behaviour of complete ASP is associated with the hydrodynamics of the individual reactors (aeration tank and secondary clarifier) and study of individual systems is necessary to predict the performance of ASP effectively. The results of the investigation also helped the industry to take a decision to selectively operate the aerators within the aeration tank with an optimum speed. This led to significant economic benefit due to reduced power consumption.

## PILOT SCALE RTD EXPERIMENT AND MODELING

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### 5.1 Design objectives of pilot scale ASP

The operating parameters of integrated paper and pulp mill ASP on which the industrial scale RTD experiments were performed, were fixed according to the industry's needs and can't be varied, as it might disrupt the entire WWTP operation. Hence, it was impractical to change the operating parameters in a full-scale industrial system. The objective of designing the pilot scale ASP was to analyse the hydrodynamic as well as the biological treatment efficiency of the ASP at different MHRT and recycle rates. The wastewater was collected from the overflow of the primary clarifier of the same industry (Shreyans Industries Ltd., Ahmedgarh, Punjab, India) where the industrial scale experiment was performed. The wastewater was characterized and the parameter obtained has been presented in Table 5.1.

**Table 5.1 Characteristics of the influent of treatment system**

Parameters	Value
BOD (mg/L)	580
COD (mg/L)	1050
TDS (mg/L)	2350
TSS (mg/L)	1090
pH	7.53

### 5.2 Microorganism growth and acclimatization

Initially the pilot scale ASP was operated on batch mode to facilitate acclimatization of microorganisms in the aeration tank. The batch mode operation was conducted for a period of 40 days until the concentration of microorganism was found to stabilize in the system. In the preliminary stage of ASP start up, excessive foaming in the aeration tank was found during the initial test runs. The rate of air supplied to the reactors was sufficient to keep the sludge flocs

suspended in the aerator. The trend analysis of pH, MLSS and COD during the acclimatization process has been discussed in subsequent sections.

### 5.2.1 Change in pH

The trends in the variation of pH during the entire period of acclimatization are almost similar. The pH was found to be within a narrow range of 7.53 – 8.54. The above pH range was favorable for acclimatization process as it falls in the alkaline pH range. The variation of pH during the acclimatization of microorganisms in the wastewater operating under batch mode has been shown Figure 5.1. The initial pH noted for the wastewater was 7.53 and tend to fall to 8.54 after addition of inoculums and gradually became constant after 15 days to 7.84.

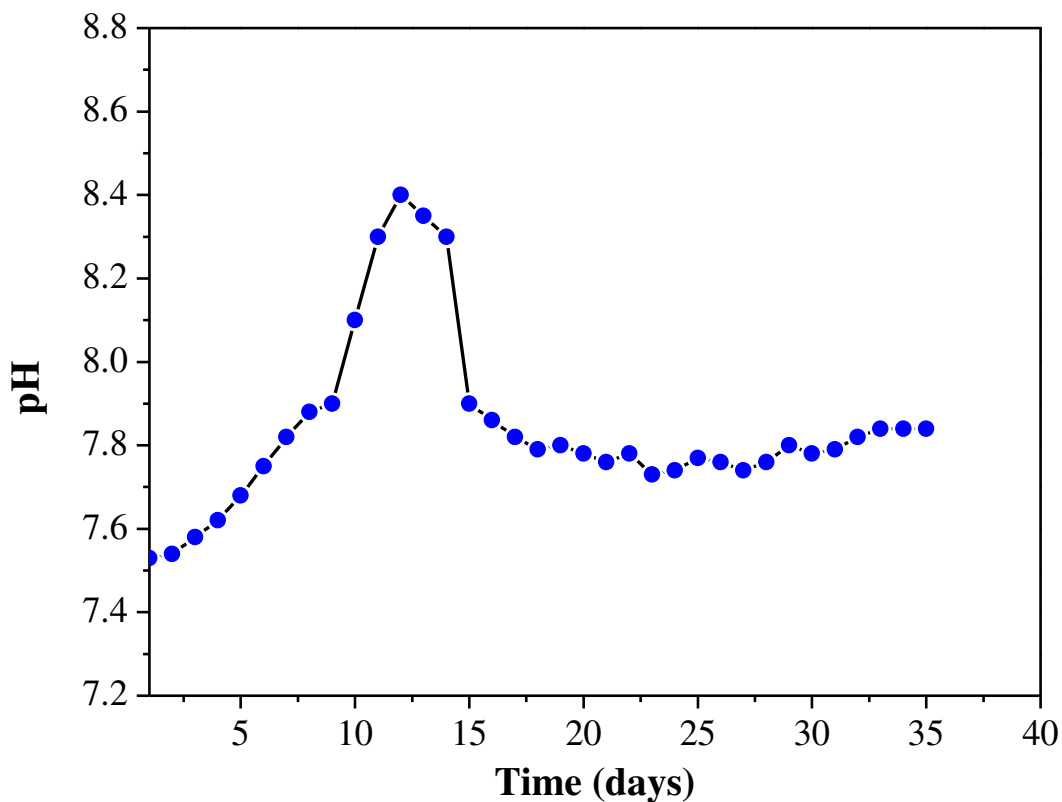


Figure 5.1 pH change of wastewater during the acclimatization process.

### 5.2.2 Change in average MLSS concentration

The initial MLSS concentration after addition of inoculum to the wastewater was 1100 mg/L. Initially the growth of microorganisms in the system was slow but starts to increase gradually from the 7<sup>th</sup> day. It was observed that the microorganisms required approximately 40 days to acclimatize in the wastewater (Figure 5.2). The plot of MLSS vs time revealed that on day 34<sup>th</sup>, the microorganisms started to stabilize and their concentration did not change significantly until day 40<sup>th</sup>. The maximum biomass concentration was recorded was 3500 mg/L. The biomass started to increase gradually from the 7<sup>th</sup> day and the final MLSS concentration on the 35<sup>th</sup> day was 3 times the initial MLSS concentration. The acclimatization period was within the range of that found in the literature (Rao *et al.*, 2005; Zawani *et al.*, 2013).

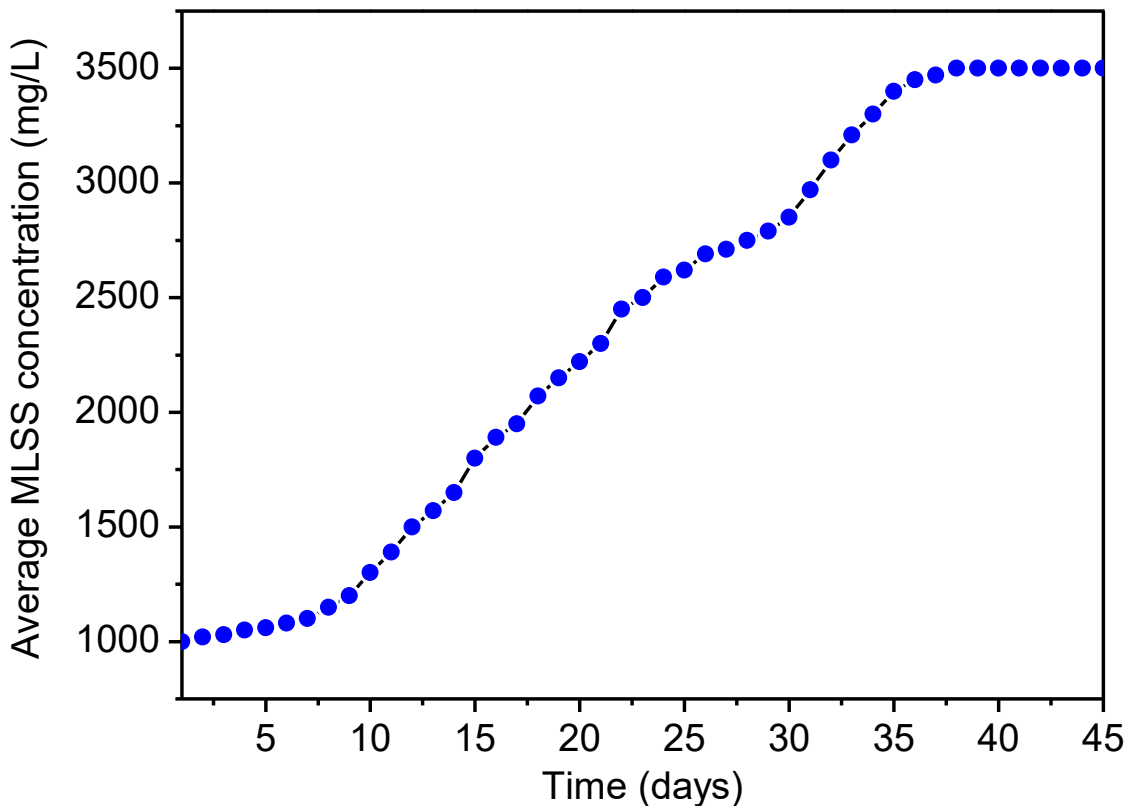


Figure 5.2 The change in average MLSS during the acclimatization process.

### 5.2.3 Change in Chemical Oxygen Demand (COD)

The COD of the influent wastewater was 1050 mg/L and the reduction of COD was observed on everyday basis during the acclimatization process. The periodic drops in the influent COD, during acclimatization as shown in Figure 5.3. The plot indicates biodegradation of effluent started right from the beginning of the batch process. The highest removal of COD was recorded during the acclimatization process was 91 %, as shown in figure 5.3. It was also observed that the degradation process was relative to the MLSS concentration in the system and the COD concentration decreased with gradual increase in the concentration of microorganisms. However, after 28 days, the degradation process became sluggish and showed no significant change was found in concentration of COD.

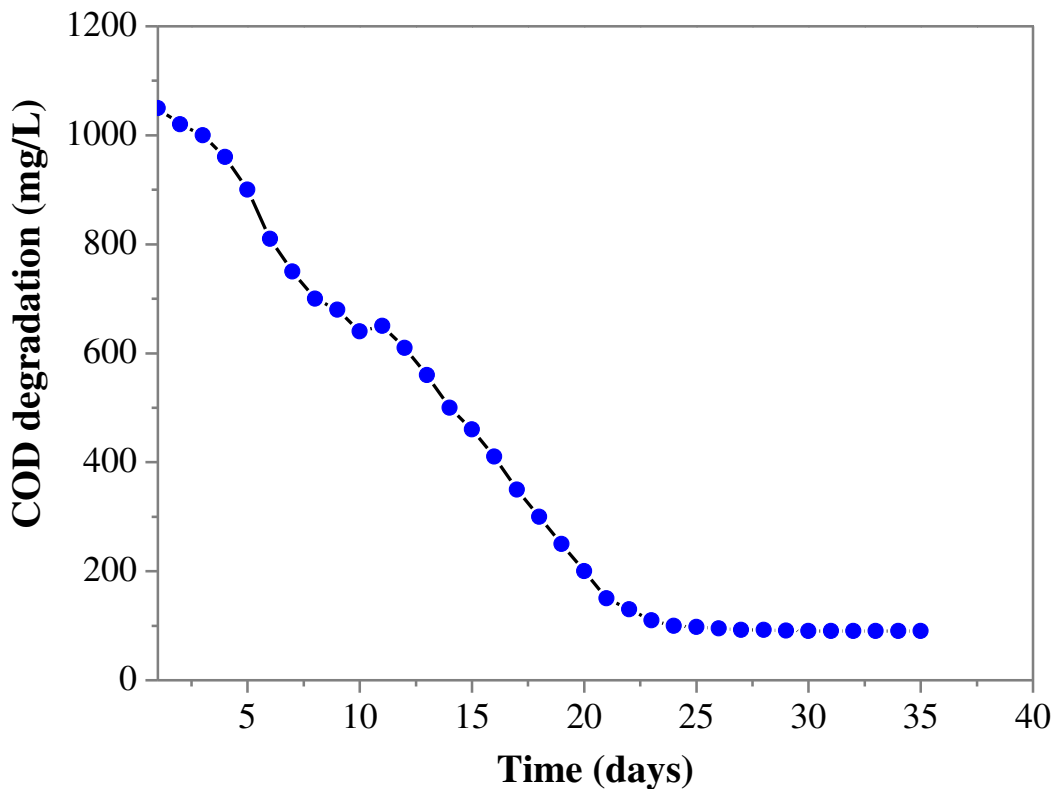


Figure 5.3 The substrate degradation (COD) during the acclimatization process.

### 5.3 RTD study of pilot scale ASP

The hydraulic performance of pilot-scale aeration tank and complete ASP was evaluated for a steady state performance. The experimental setup for pilot scale ASP has been shown in Figure 5.4. The DO concentration in the aeration tank was maintained at 1.8-2.0 mg/L, considering difficulty to maintain DO level above 2 mg/L at industrial operating condition. The set of different MHRTs were chosen to establish a correlation between operating parameters and treatment efficiency. The RTD experiments were carried out on the aeration tank at hydraulic MHRT of 14, 16, 20 and 24 hours without any sludge recycle. RTD experiments were also performed by varying the sludge recycle rate from the bottom of secondary clarifier to the aeration tank to 10% and 20% of the inlet wastewater flowrate, for each set of MHRT. The background LiCl concentration was noted and pre-calculated amount of tracer was injected at the inlet of the aeration tank and monitored at the outlet of the aeration tank ( $S_1$ ) and the overflow of secondary clarifier ( $S_2$ ).

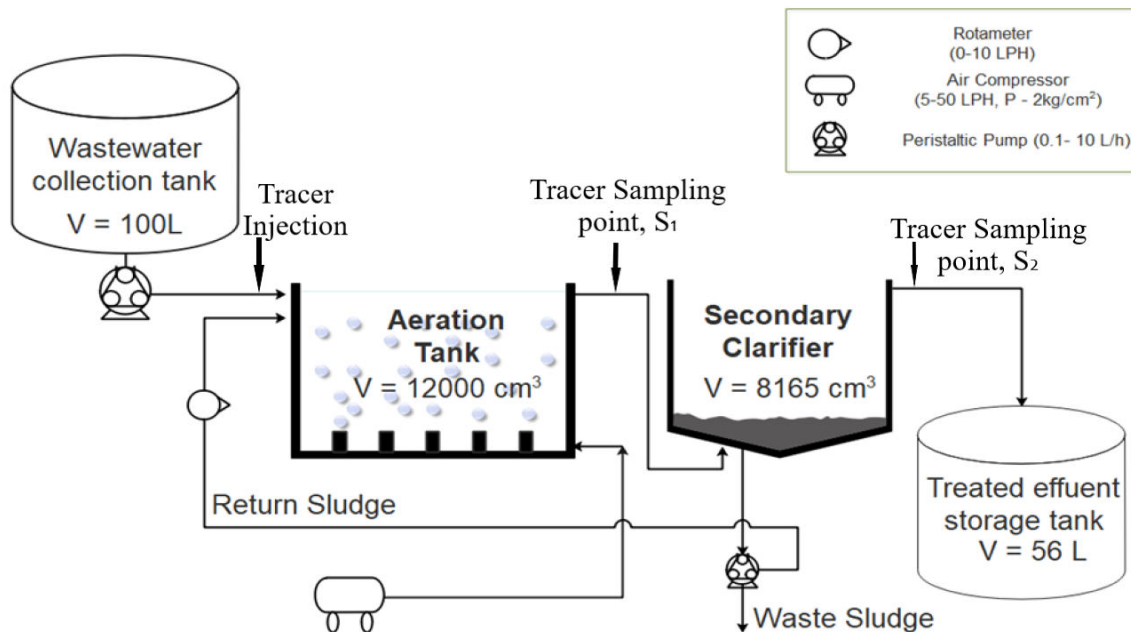


Figure 5.4 Experimental setup of pilot scale ASP

### 5.3.1 Calculation of Moments

The experimental RTD data evaluated at the outlet of aeration tank and secondary clarifier was obtained in the form of LiCl concentration vs time ( $C_{Li^+}(t)$  vs  $t$ ) curves that were transformed to RTD function  $E_{Li^+}(t)$  vs  $t$  curves.  $E_{Li^+}(t)$  denotes the zero<sup>th</sup> moment of RTD was calculated according to Equation 3.2. The  $E_{Li^+}(t)$  vs  $t$  curves obtained for the aeration tank and complete ASP has been shown in Figure 5.5 (a) and Figure 5.5 (b) respectively. For the aeration tank, the analysis of the RTD curves (Figure 5.5 (a)) indicates a distinguish peak at MHRT 14 h. This peak indicates the existence of bypassing streams in the aeration tank at 14 h MHRT. As the MHRT of the wastewater is increased in the reactor, no significant peaks were observed, hence the bypassing streams disappears for MHRTs higher than 14 h. The experimental RTD data for the aeration tank also indicated the non-existence of parallel and recirculation steams in the aeration tank. The LiCl tracer concentration was also measured at the overflow of the secondary clarifier (sampling point S<sub>2</sub>). This considers the flow in complete ASP as tracer passes through both aeration tank and secondary clarifier. The  $E_{Li^+}(t)$  vs  $t$  curves obtained at different MHRT for complete ASP did not show any significant peaks. No bypassing, parallel and recirculation streams were observed in any of the RTD curves. The experimental MHRT for aeration tank and complete ASP was calculated from the  $E_{Li^+}(t)$  curves (Table 5.2 and Table 5.3). The fraction of stagnant zones present in the aeration tank and complete ASP was determined by comparing the theoretical MHRT and experimentally obtained MHRT (Table 5.2 and Table 5.3).

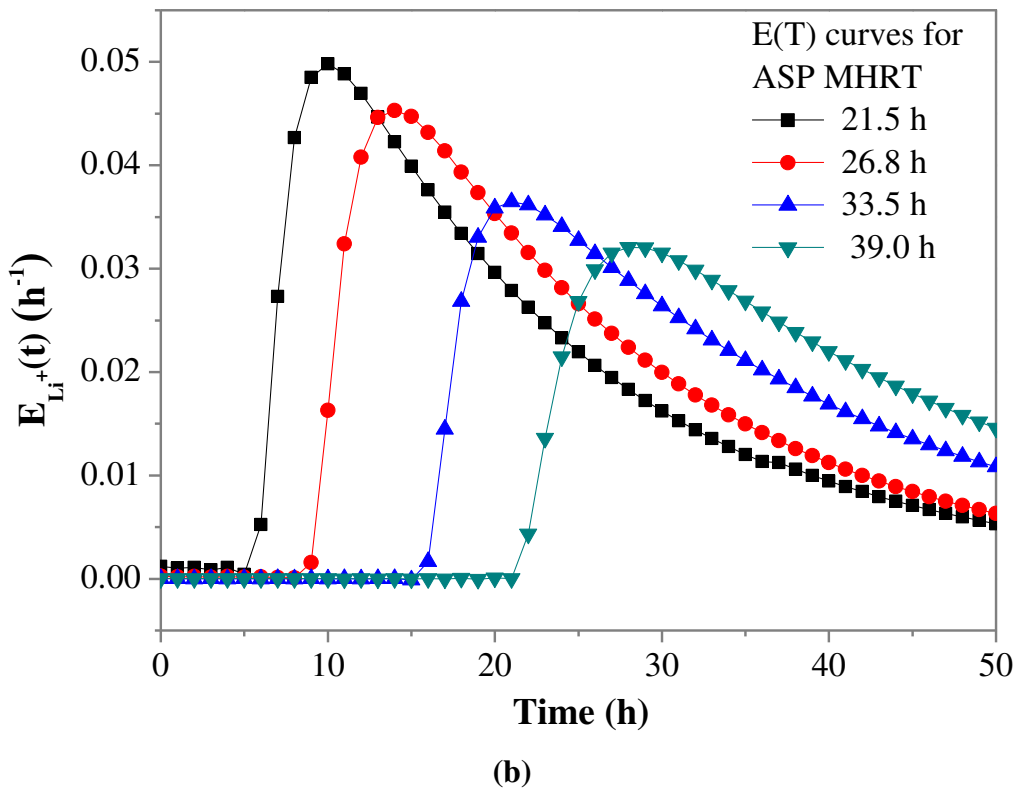
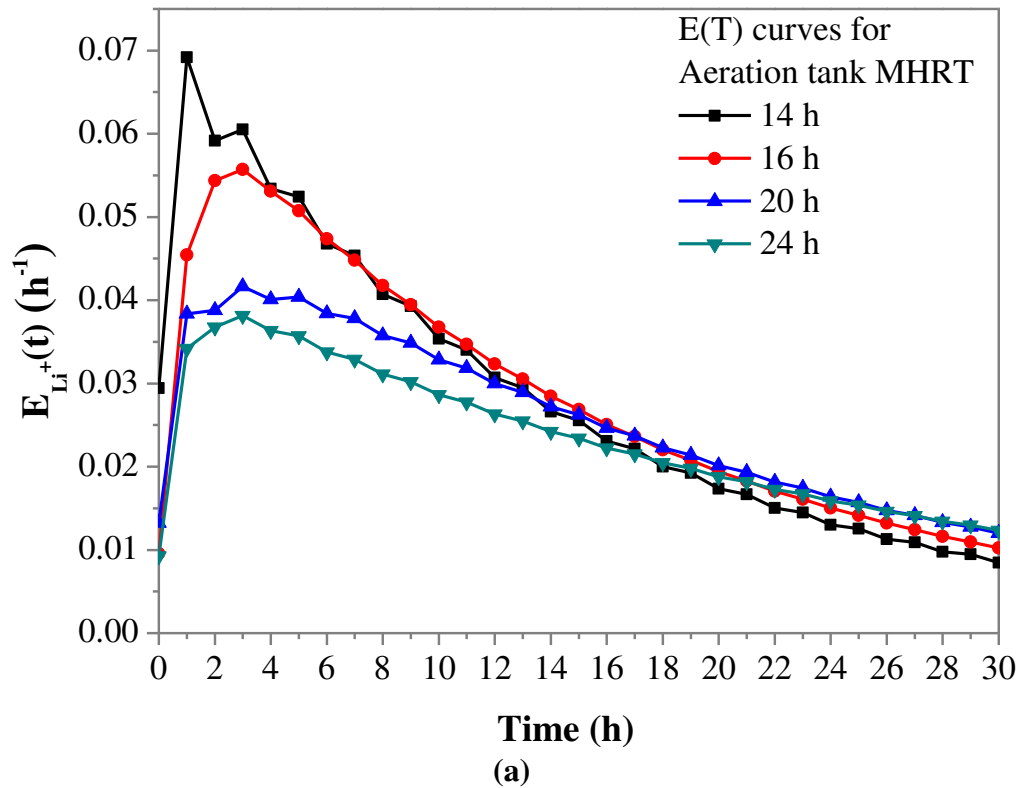


Figure 5.5 The RTD curves obtained at different MHRT for (a) aeration tank and (b) ASP.

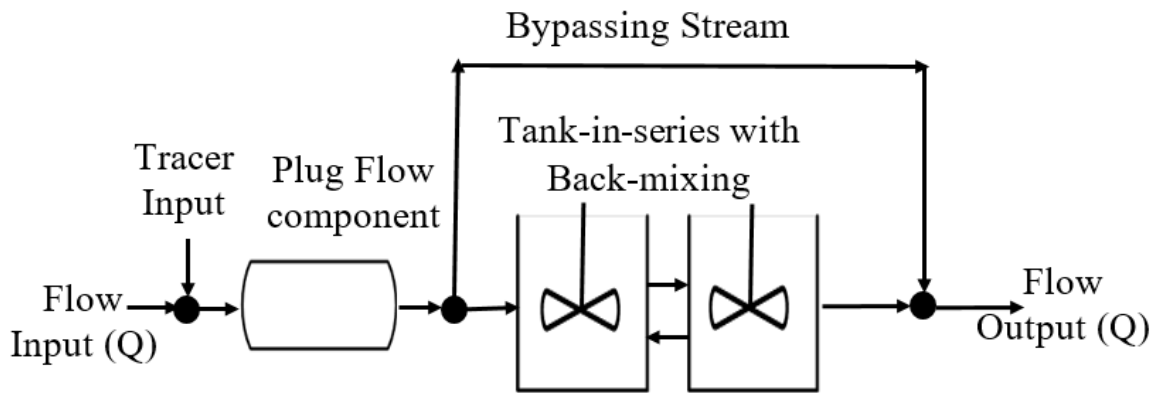
## 5.4 RTD Modeling

### 5.4.1 Modeling of pilot scale Aeration tank at varying MHRT

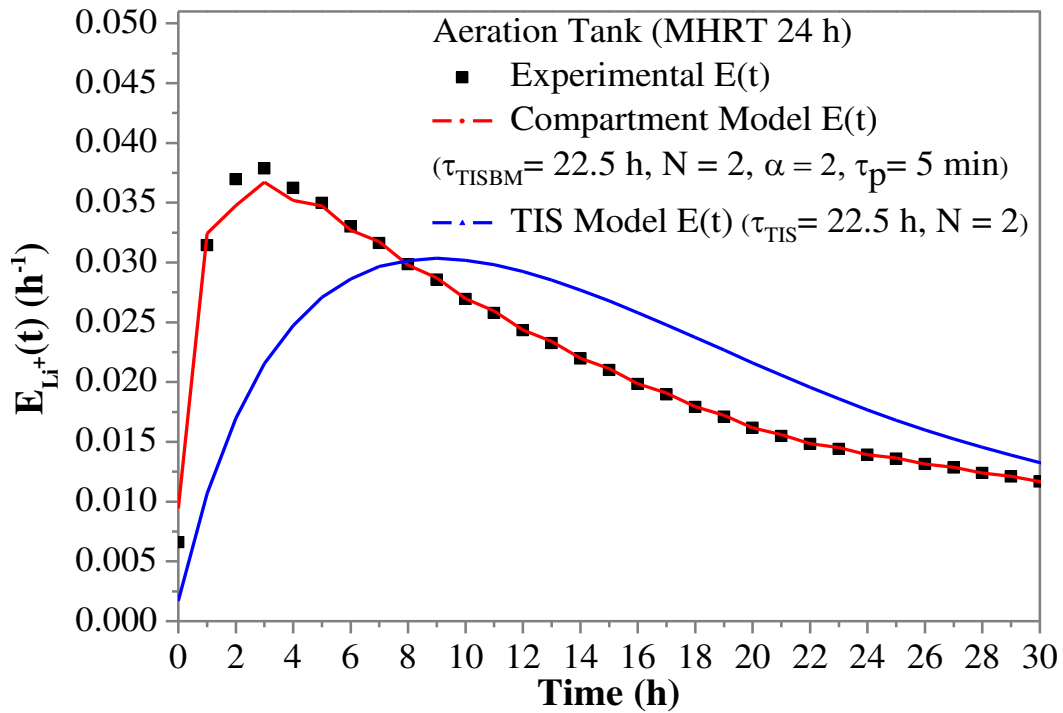
To obtain a more extensive view of the behaviour of the aeration tank, the RTD curves measured at the outlet of aeration tank at different MHRT (no recycle) were simulated using RTD models. Appropriate empirical models were proposed by applying compartment modeling technique. The model proposed for aeration tank have been show in Figure 5.6, consists of three building blocks i.e. plug flow component (parameter, plug flow component MHRT,  $\tau_p$ ) connected in series with a tanks-in-series with back-mixing component (parameters tank-in-series with back-mixing MHRT ( $\tau_{TISBM}$ ), number of tanks (N) and back-mixing ratio ( $\alpha$ )) and a bypassing stream. The plug flow component in the beginning, represents the delay in appearance of LiCl tracer at the outlet of the aeration tank, whereas the TISBM model represents extent of mixing within the aeration tank. The bypassing stream is added to account for the percentage bypassing at lower MHRT. It was found that, at 14 h MHRT of aeration tank, 7 % of wastewater bypasses the main flow during first 30 minutes. Several standard models were fitted from the literature using DTS Pro V-4.21. The compartment models are a combination of multiple reactors that provides close to exact representations of the actual reactors. The compartment model assumes anomalies as small reactors acting inside the main reactor. These reactors can be taken in various commination along with the main reactor and curve fitting can be obtained.

In the prior RTD studies performed on the aeration tank, it was observed that the hydrodynamic behaviour was represented by a series of mixing tank (Makinia and Wells, 2005; Olivet *et al.*, 2005). Conventional model as tank-in-series and axial dispersion models were unable to satisfactorily portray the RTD curves (De Clercq *et al.*, 2005; Makinia and Wells, 2005; Karches and Buzas, 2013). Hence, the suggested compartment modeling was compared to conventional tank-in-series model. The modeling of aeration tank at 14 h MHRT predicted that reactor was working approximately as a complete mixing tank with no back-mixing. The TIS model was also well fitting for the 16 h MHRT, but showed large deviation at higher MHRT of 20 h and 24 h. The back-mixing ratio obtained for the aeration tank at 16 h, 20 h and 24 h MHRT indicates that the diffused aeration system was providing sufficient aeration and the mixing intensity in the aeration tank provided homogeneity to the aeration tank maintaining the

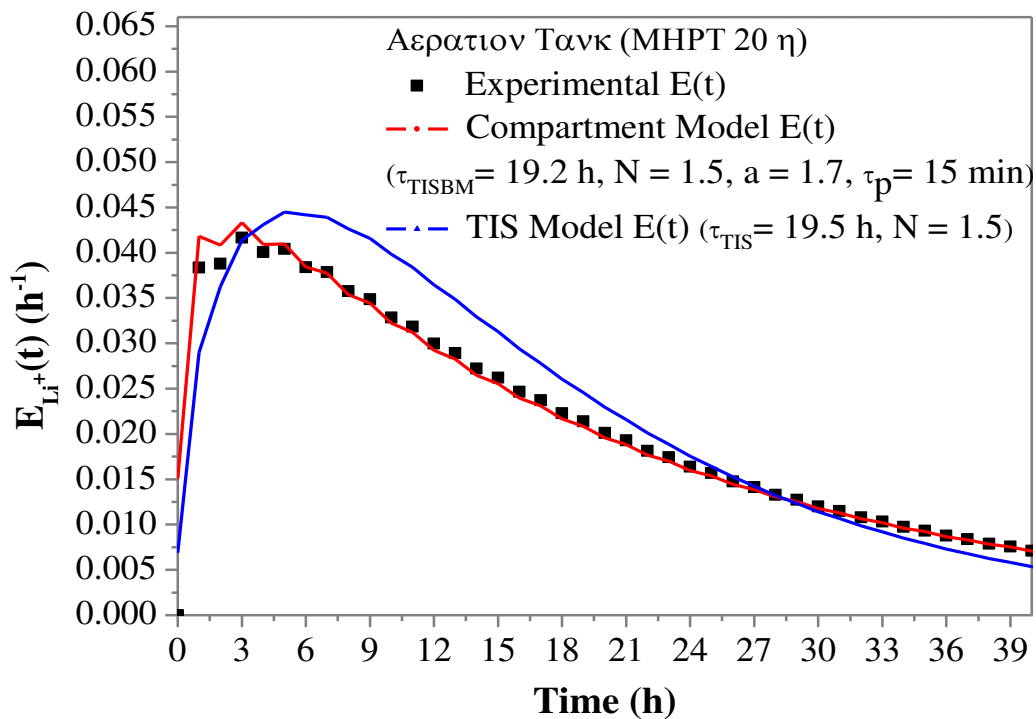
microorganisms well suspended in the tank. Hence, at the lower MHRT, the aeration tank was behaving more as perfect mixing tank. The compartment model parameters for the aeration tank, the number of tanks,  $N$ , and back-mixing ratio  $\alpha$ , for each experiment were determined and has been listed in Table 5.2. The model stimulated data for aeration tank at different MHRT has been shown in Figure 5.7 (a-d).



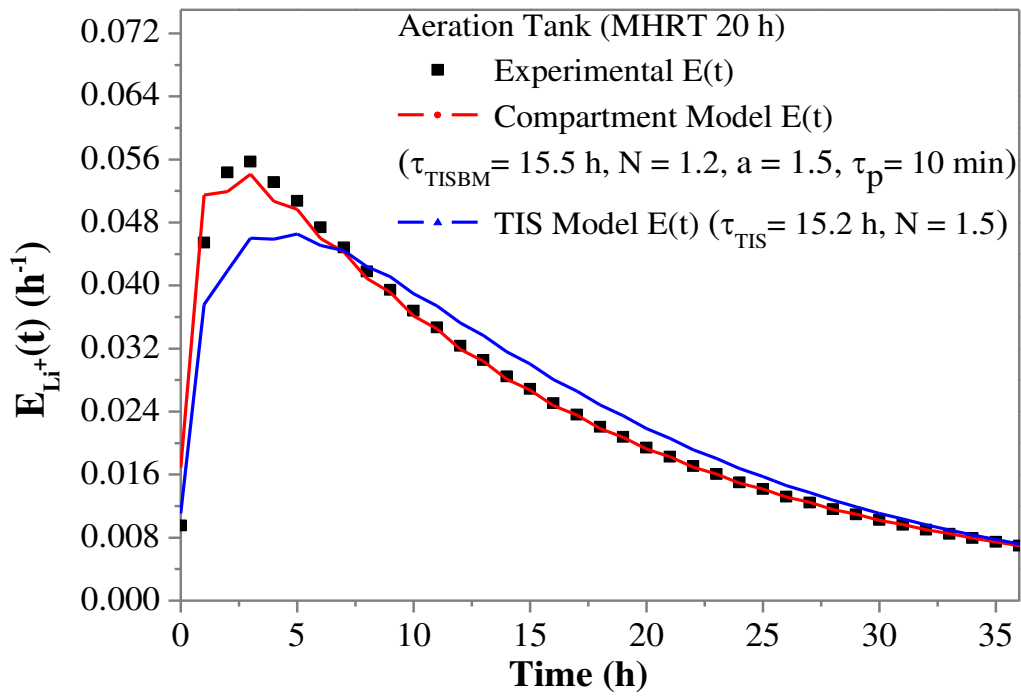
**Figure 5.6** Compartment model for the hydraulic representation of Aeration Tank at varying MHRT without recycle.



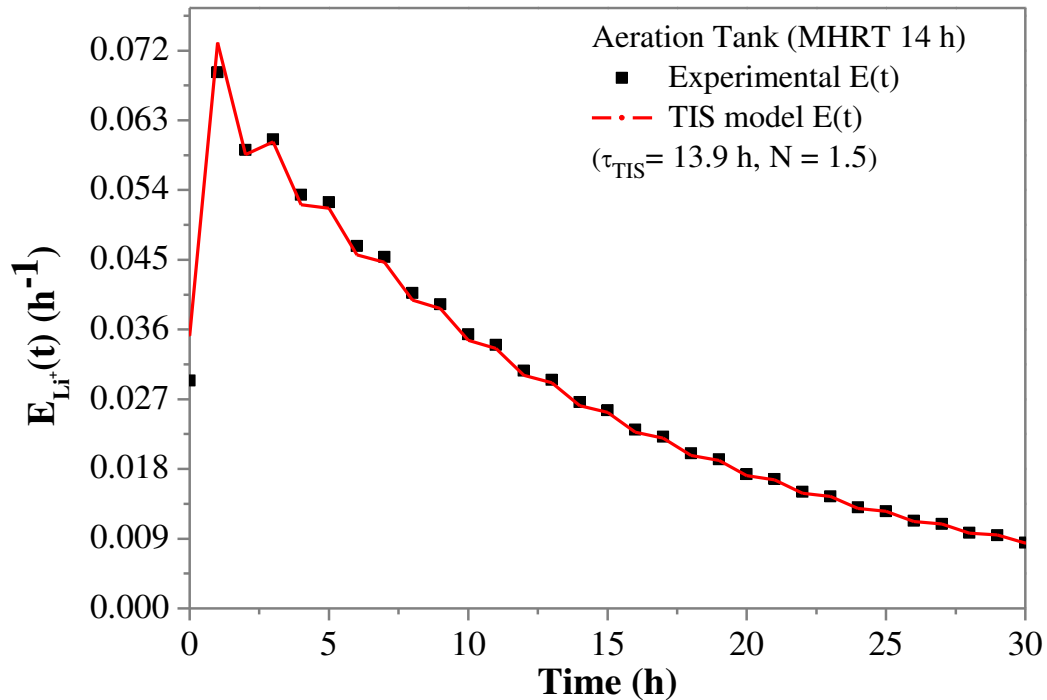
(a)



(b)



(c)

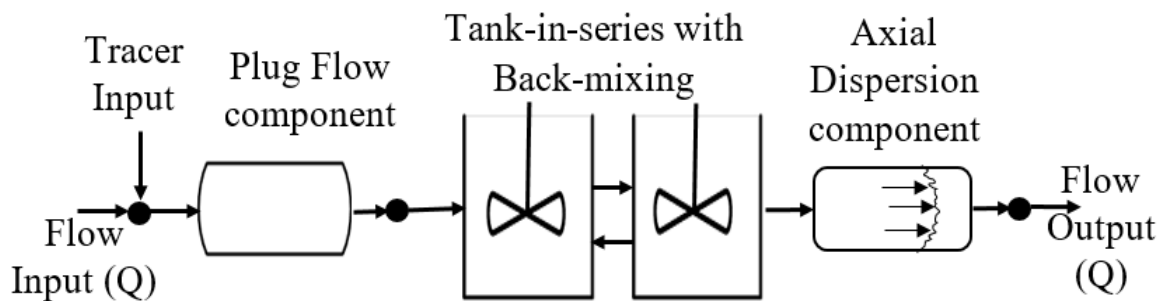


(d)

**Figure 5.7 Comparison of experimentally measured and model-simulated RTD curves at the outlet of the aeration tank for MHRT (a) 24 h (b) 20 h (c) 16 h and (d) 14 h.**

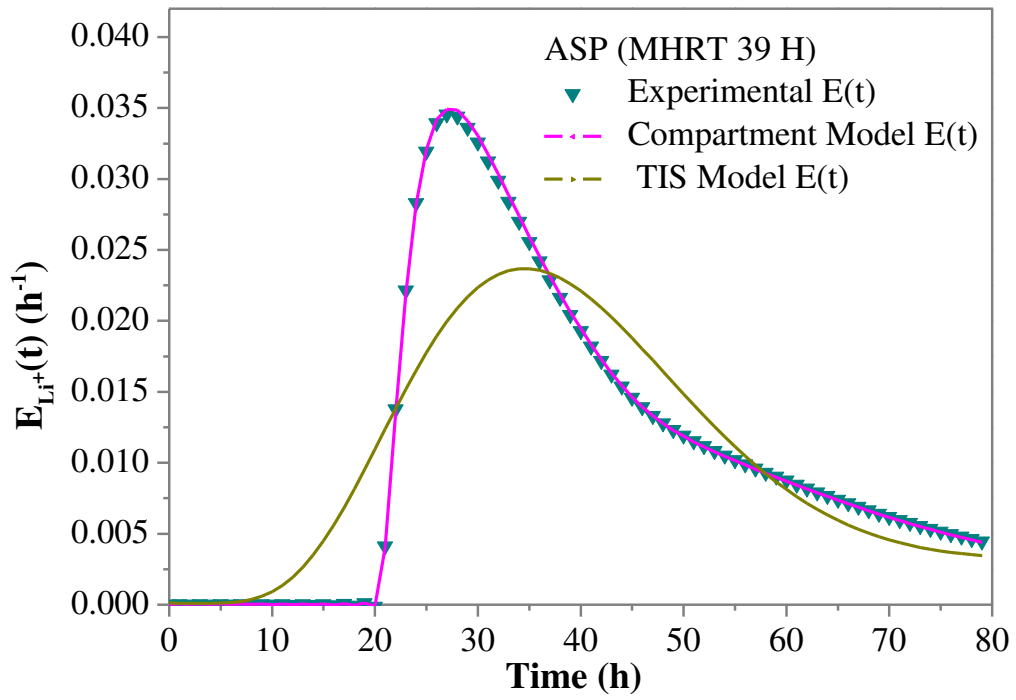
### 5.4.2 Modeling of pilot scale ASP at varying MHRT

The hydrodynamics of complete ASP was more complex as it was a combination of the aeration tank and secondary clarifier. Based on the model fitting for aeration tank a compartment model was suggested for complete ASP (Figure 5.8). The model consists of three building blocks i.e. plug flow component (parameter, plug flow component MHRT,  $\tau_p$ ), a tanks-in-series with back-mixing component (parameters tank-in-series with back-mixing MHRT ( $\tau_{TISBM}$ ), number of tanks (N) and back-mixing ratio ( $\alpha$ )) and axial dispersion component (parameter pecelet number ( $Pe$ )) all connected in series.

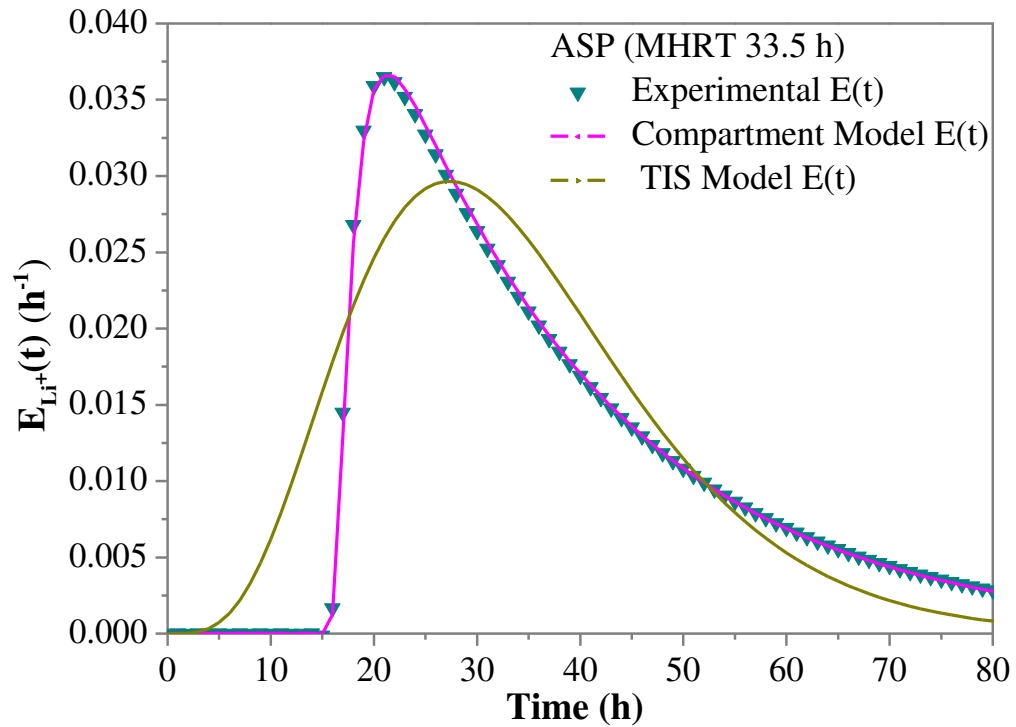


**Figure 5.8 Compartment model for the hydraulic representation of ASP at varying MHRT without recycle.**

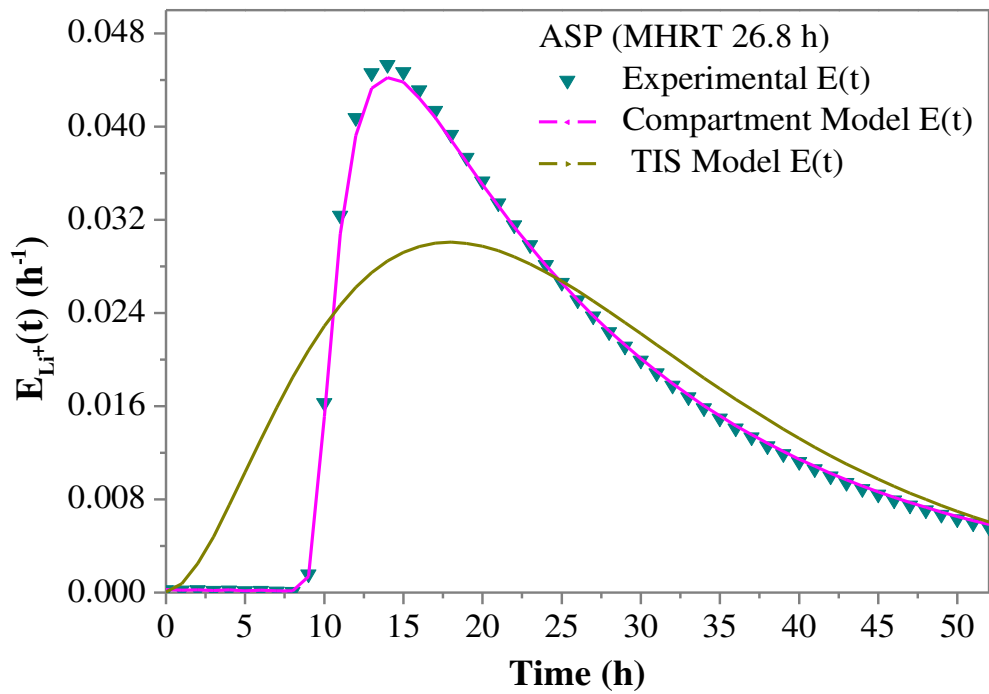
The tank-in-series model is a single parameter model that only accounts for the mixing intensity of the reactor in terms of the number of reactors, N. If the number of tanks, N equals to 1, the reactor behaves as a perfect mixing tank. As the value of N increases the reactor tends to behave as a plug flow reactor. As the ASP is a complex reactor, conventional TIS model fails to take into account the effect back-mixing, recycle streams and dead zones on the flow behaviour of the aeration tank (Burrows *et al.*, 1999; Kjellstrand *et al.*, 2005; Collivignarelli *et al.*, 2018). The compartment model was able to better represent the entire ASP than the conventional tank-in-series (TIS) model.



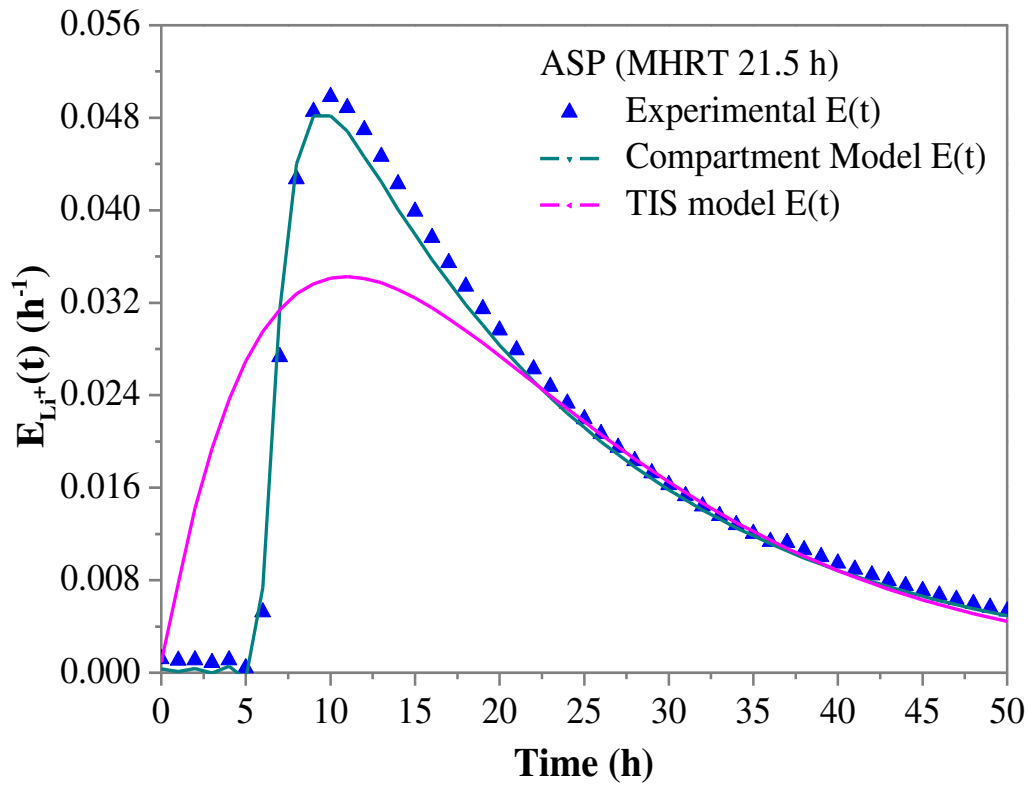
(a)



(b)



(c)



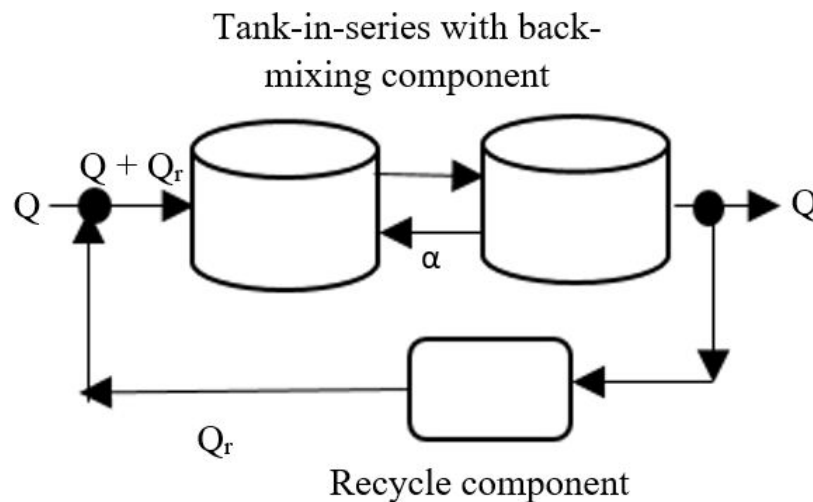
(d)

**Figure 5.9 Comparison of experimentally measured and model-simulated RTD curves at the outlet of the ASP MHRT (a) 39 h (b) 33.5 h (c) 26.8 h (d) 21.5 h.**

The model parameters, the number of tanks,  $N$ , and back-mixing ratio  $\alpha$ , and Peclet no ( $Pe$ ) for each experiment were determined. The results of the RTD experimental data and model-simulated data for the aeration tank and ASP have been shown in Figures 5.9 (a-d). The possibility of the dead-zones in the ASP was also calculated and has been reported in Table 5.3. The dead-zone fraction for all the set of experiments remained between the ranges of 0.5- 4%, which was well below the experimental limits.

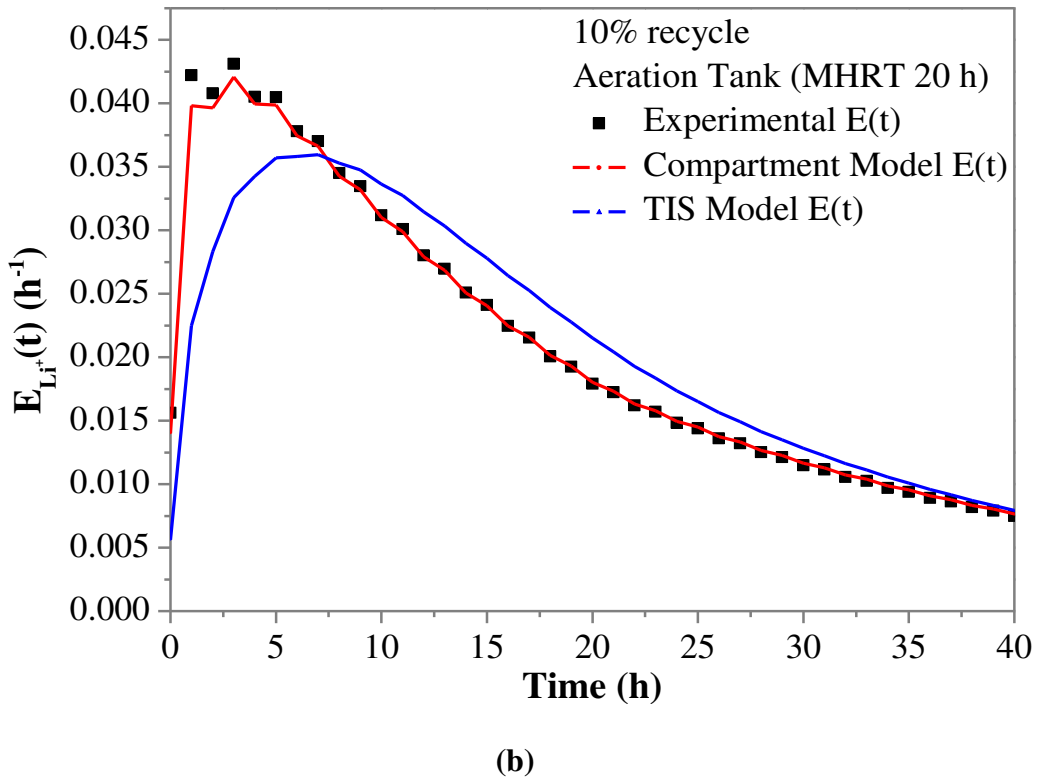
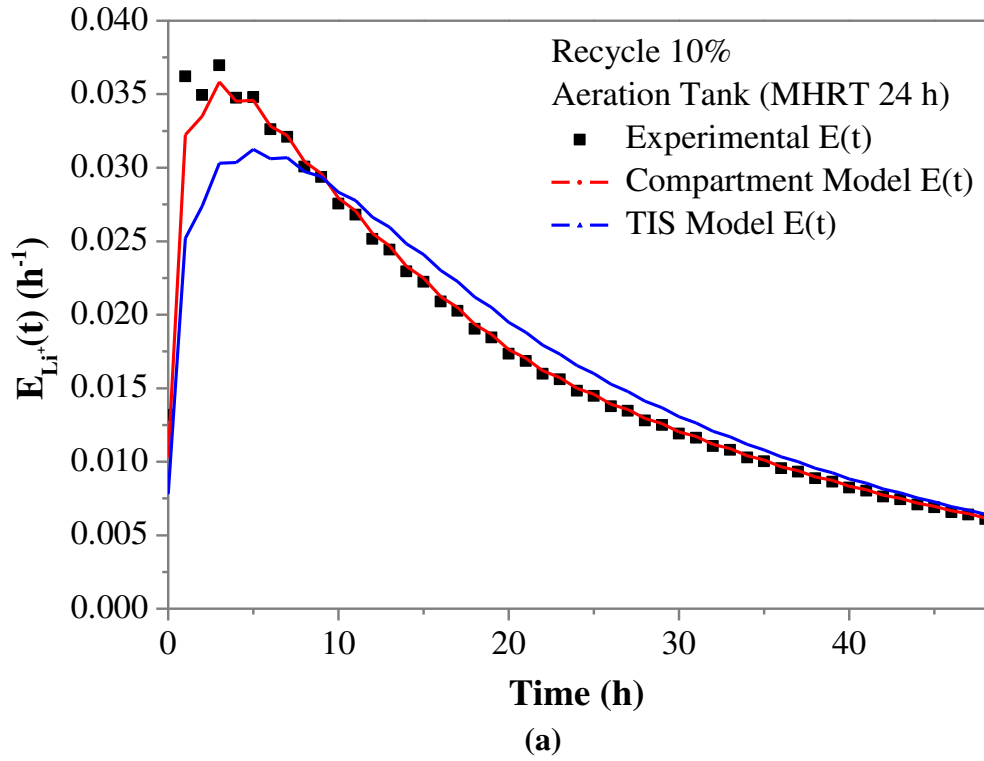
### 5.4.3 Modeling of Aeration Tank at varying MHRT with sludge recirculation

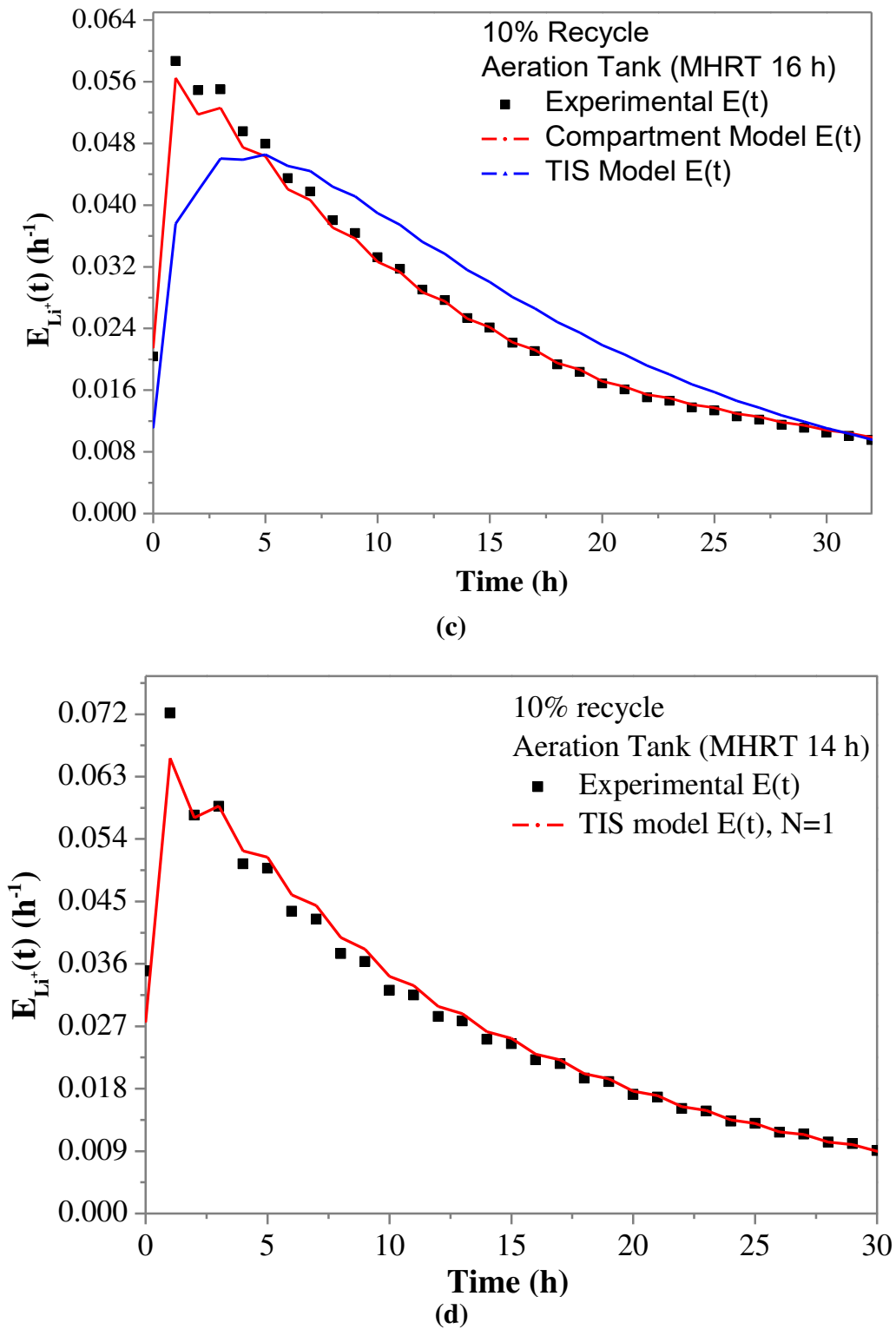
The biomass formed in the aeration and settled at the bottom of the clarifier is recycled back to the aeration tank. The effect of the rate of sludge recirculation on the hydrodynamics of the aeration tank was analysed. For each set of MHRT the sludge recycle ratio was set to 10% and 20% of the wastewater inlet flow rate and RTD experiment was performed. The RTD curves were studied and modeled with appropriate compartment models. The compartment model proposed of the aeration tank includes back-mixing component (parameters tank-in-series with back-mixing MHRT ( $\tau_{TISBM}$ ), with a recycle line that was added to the model that to compensate the effect of recycling on the system (Figure 5.10).



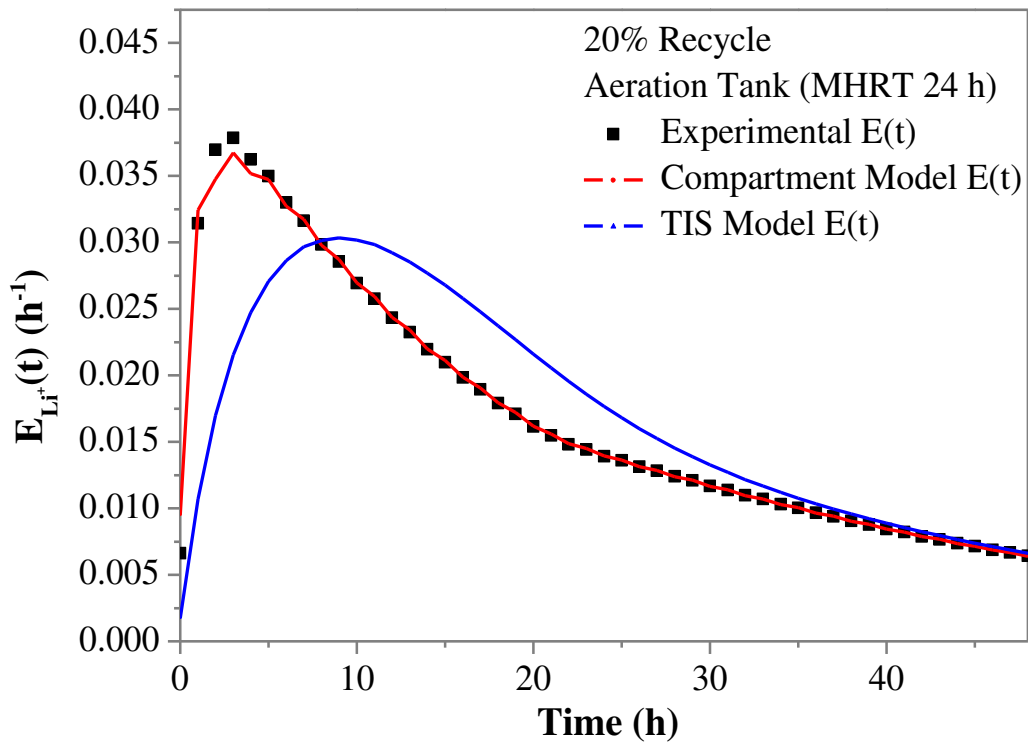
**Figure 5.10** Compartment model for the hydraulic representation of Aeration Tank with recycle.

The result of the RTD experimental data and model-simulated data has been presented in Figure 5.11 and Figure 5.12 for sludge recycle of 10% and 20%, respectively. It was observed that dispersion of tracer increased with addition of recycle line. The effect of recycling was found to be more prominent on the hydrodynamics of the aeration tank at higher residence time. The back-mixing ratio in the aeration tank tends to increase with the increase in the sludge recycle ration. The recycle stream did not cause any bypassing parallel paths in the reactor. In the aeration tank working at 14 hour MHRT, fraction of bypassing stream reduced as the recycled sludge percentage was increased. The percentage bypassing in aeration tank, working at MHRT 14 hours and 10% recycle was found 5%. The percentage bypassing reduced to mere 2% as the recycle sludge fraction was increased to 20%. The model parameters, number of tanks,  $N$ , and back-mixing ratio  $\alpha$ , and recycle time,  $\tau_R$  for each experiment was determined and has been given in Table 5.2. The fraction of dead- zones in the aeration tank intensified at high sludge recycle rate due to the high concentration of MLSS in system. However, the dead volume was less than 4% in all experiments which was well under the experimental limits. A slight increase in back-mixing ratio was observed as the recycle ratio was increased, but the fraction was insignificant and did not cause any major changes in the RTD modelling.

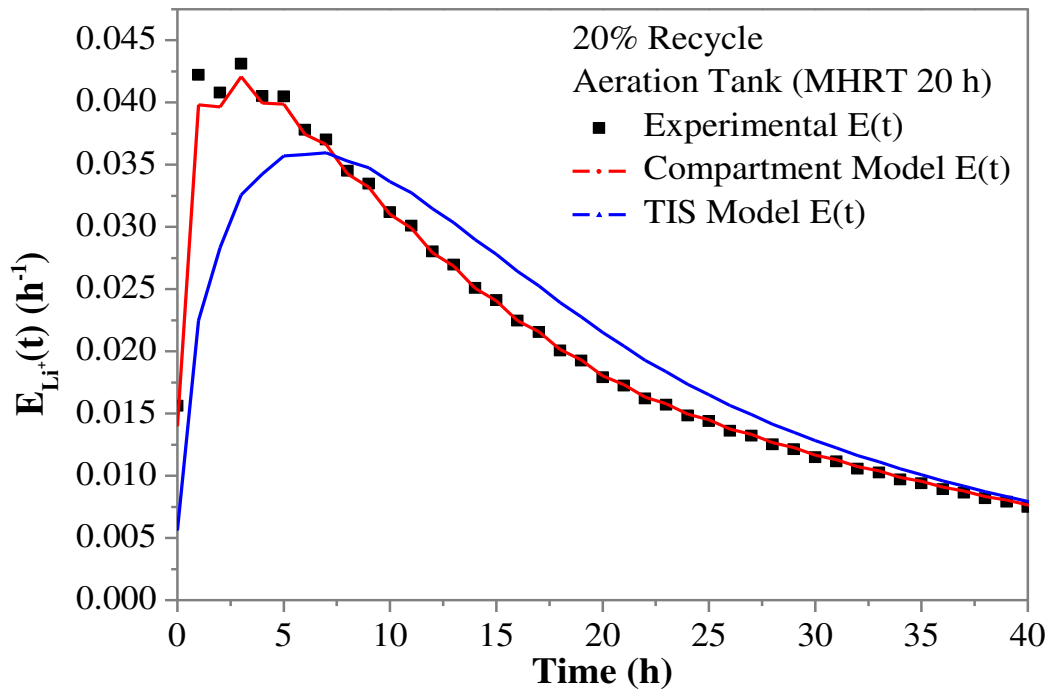




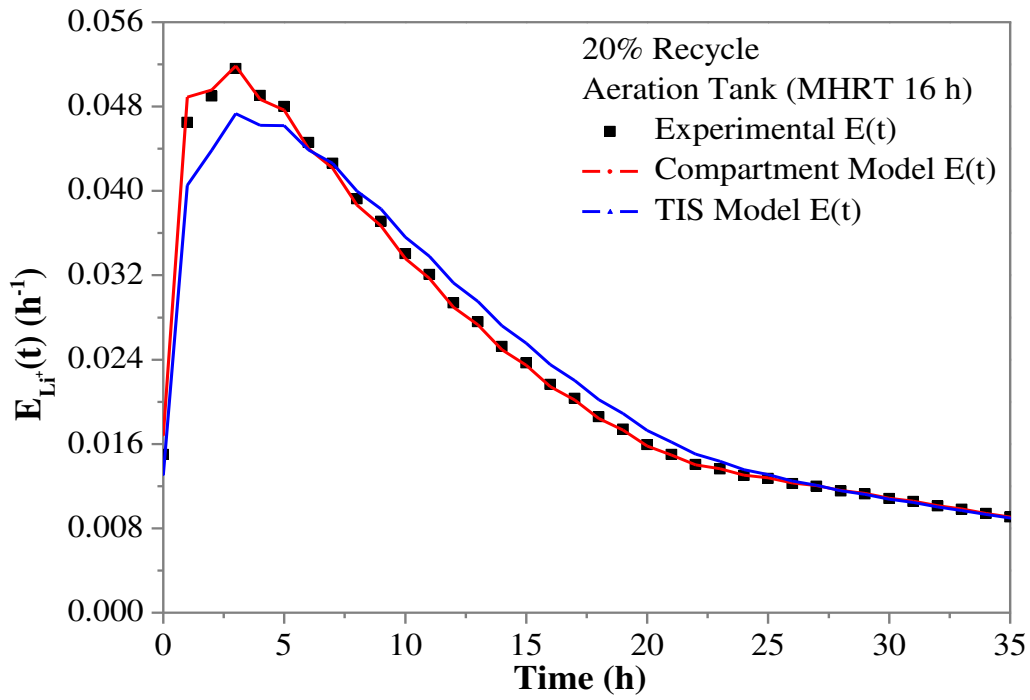
**Figure 5.11 Comparison of experimentally measured and model-simulated RTD curves aeration tank for 10% sludge recycle and MHRT (a) 24 h (b) 20 h (c) 16 h and (d) 14 h.**



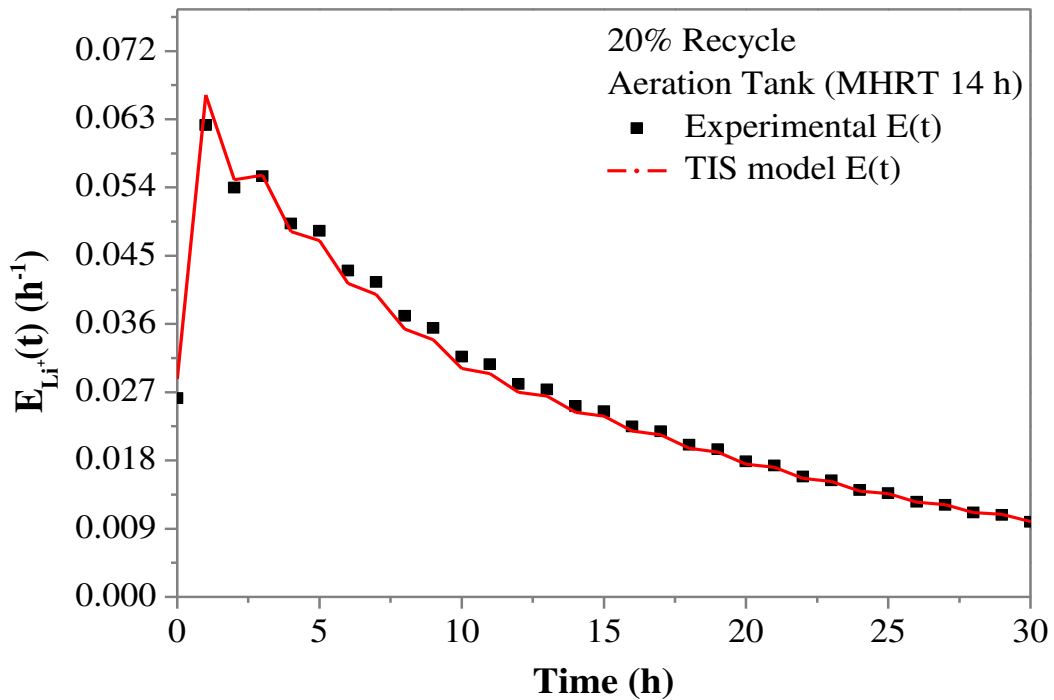
(a)



(b)



(c)

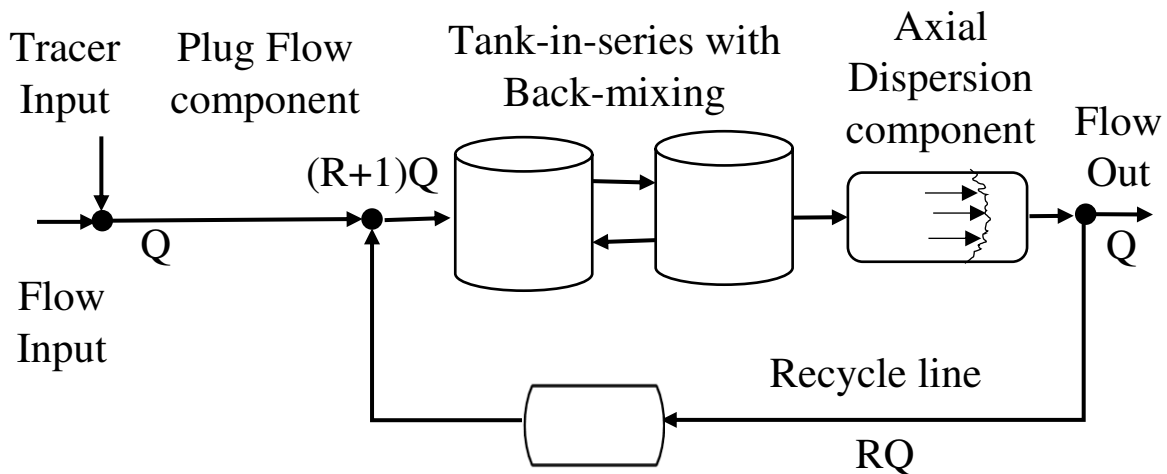


(d)

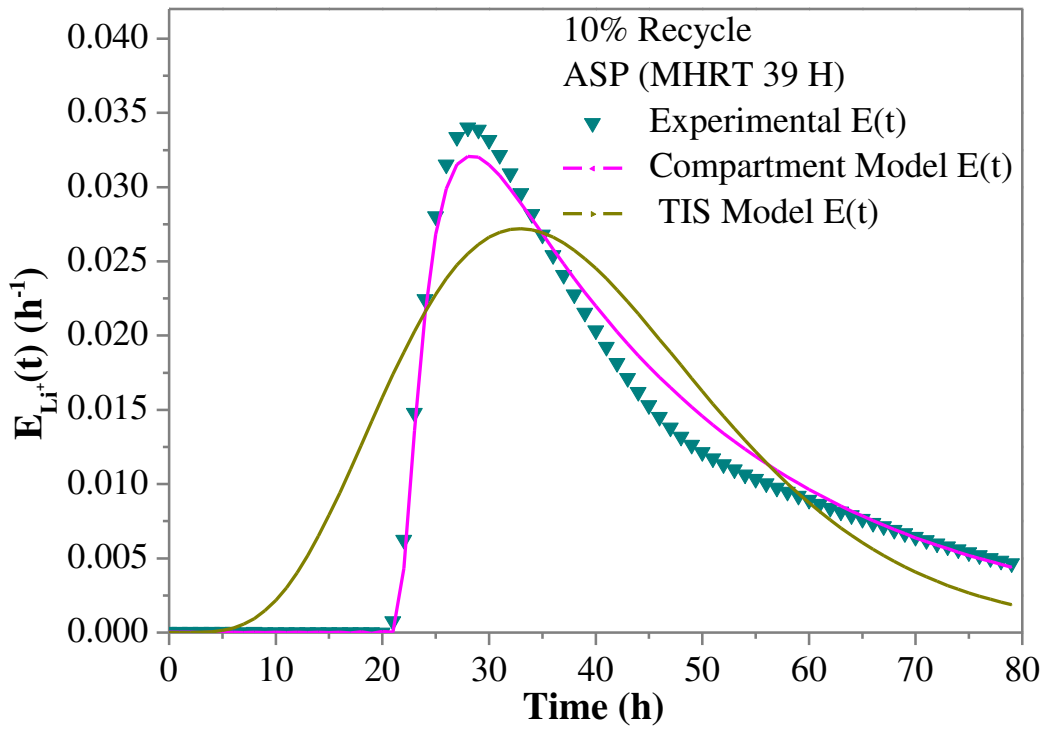
**Figure 5.12 Comparison of experimentally measured and model-simulated RTD curves aeration tank for 20% sludge recycle and MHRT (a) 24 h (b) 20 h (c) 16 h and (d) 14 h.**

#### 5.4.4 Modeling of complete ASP at varying MHRT with sludge recirculation

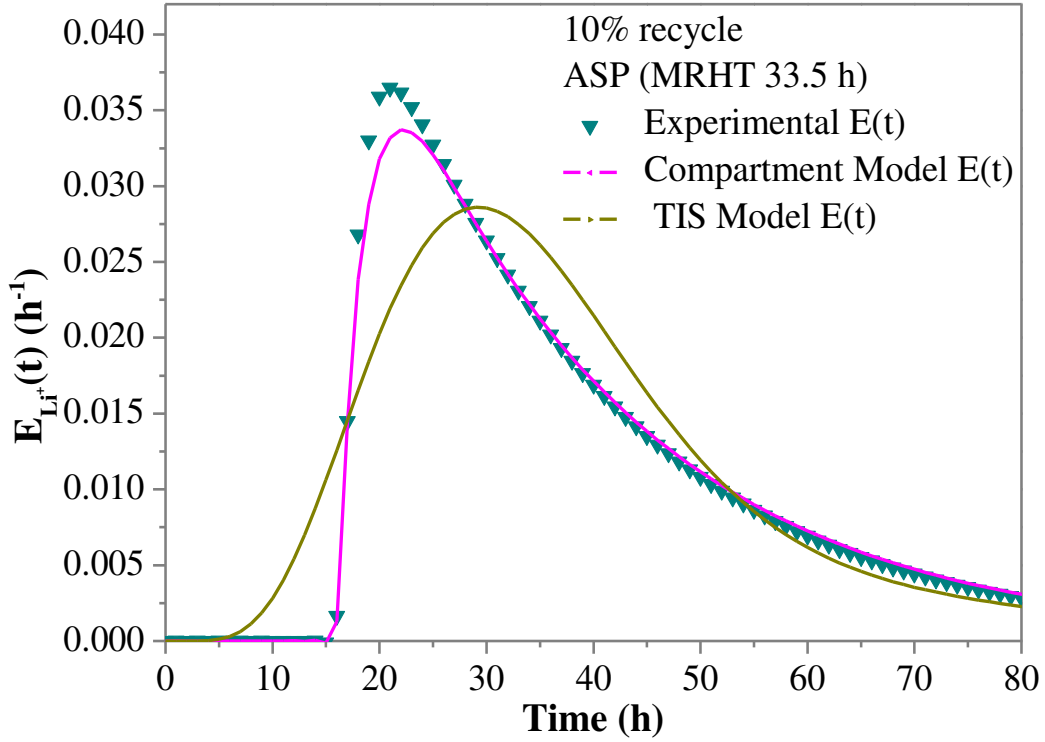
The influence of varying recycle rate was also analysed for complete ASP (aeration tank + clarifier). The RTD curves were studied and modeled with appropriate compartment models. The recycling stream didn't cause any significant fluctuations in the RTD curves. The building blocks of compartment model proposed for the complete ASP consisted of a tank in series with back-mixing component (parameters tank-in-series with back-mixing MHRT ( $\tau_{TISBM}$ ), number of tanks (N)) connected in series with axial dispersion model (Parameters: axial flow component MHRT( $\tau_{ADM}$ ), Peclet number, Pe) connected in series with recycling line with a plug flow component ( $\tau_R$ ) connected in parallel as shown in Figure 5.13. The model parameters, number of tanks, N, and back-mixing ratio  $\alpha$ , and Peclet number, Pe and recycle time,  $\tau_R$  for each experiment was determined (Table 5.3). The result of the RTD experimental data and model-simulated data has been presented in Figure 5.14 and Figure 5.15 for sludge recycle of 10% and 20%, respectively. The fraction of dead zone in the tank increases by approximate 10 - 15% with every 10% increase in recycle rate.



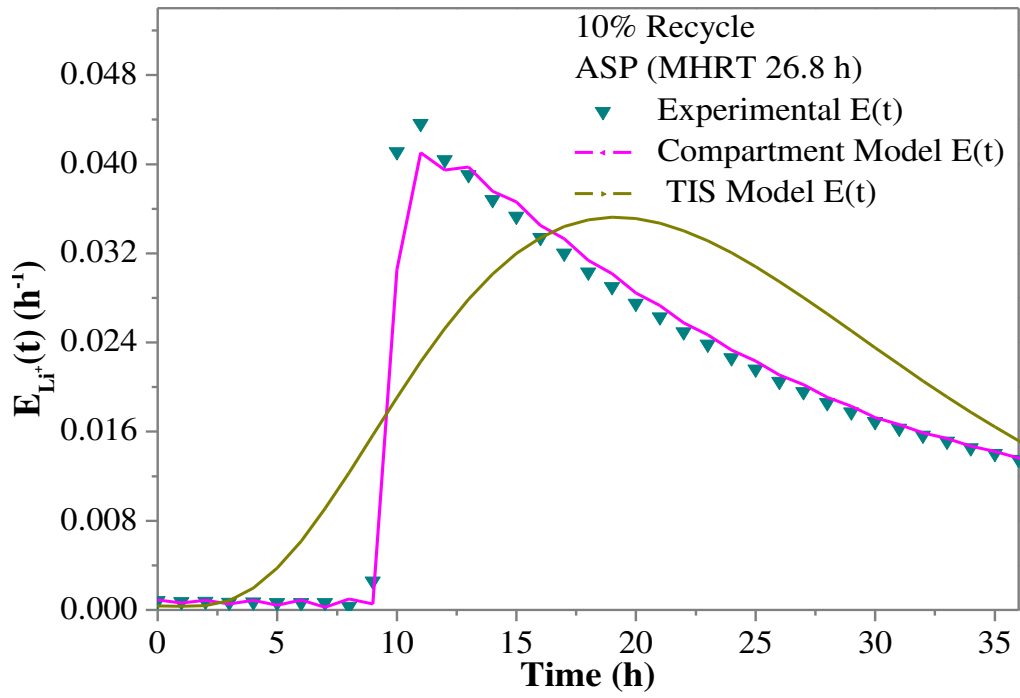
**Figure 5.13** Compartment model for the hydraulic representation of ASP with recycle.



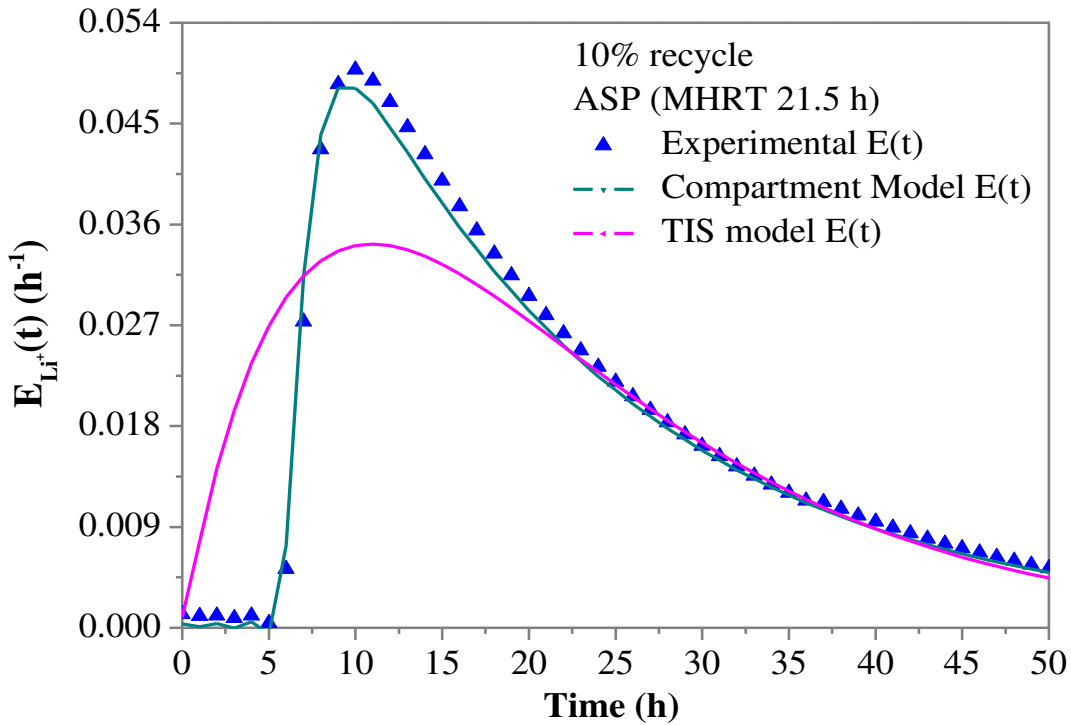
(a)



(b)

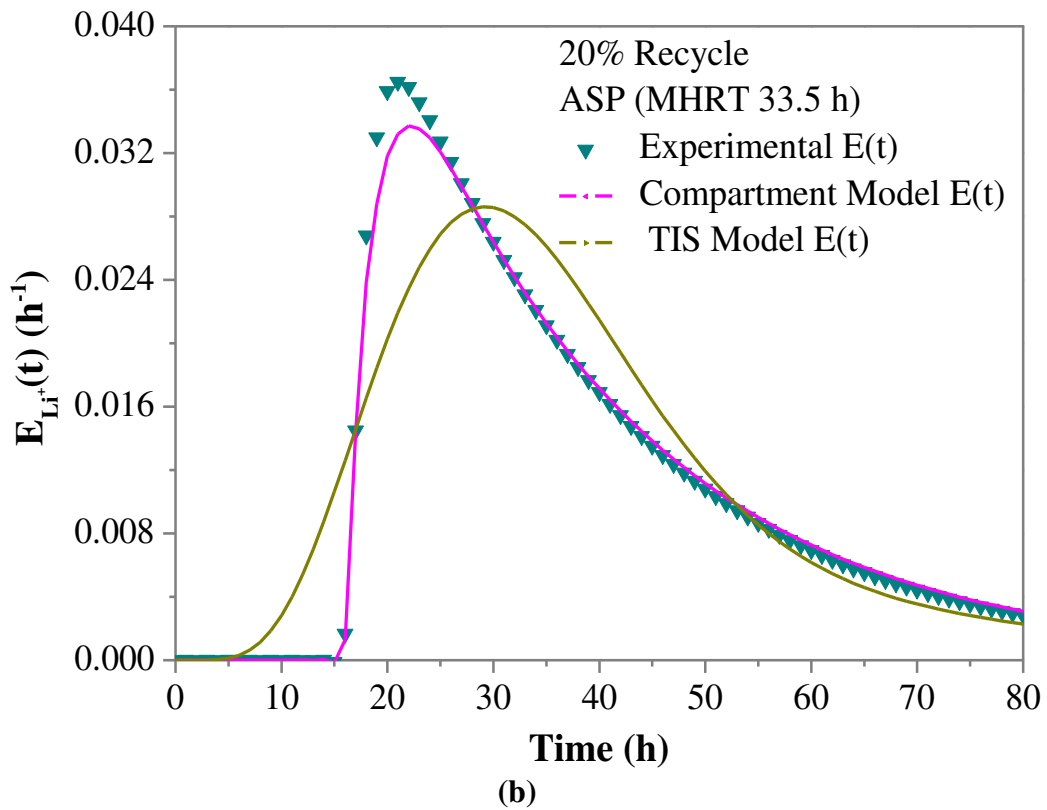
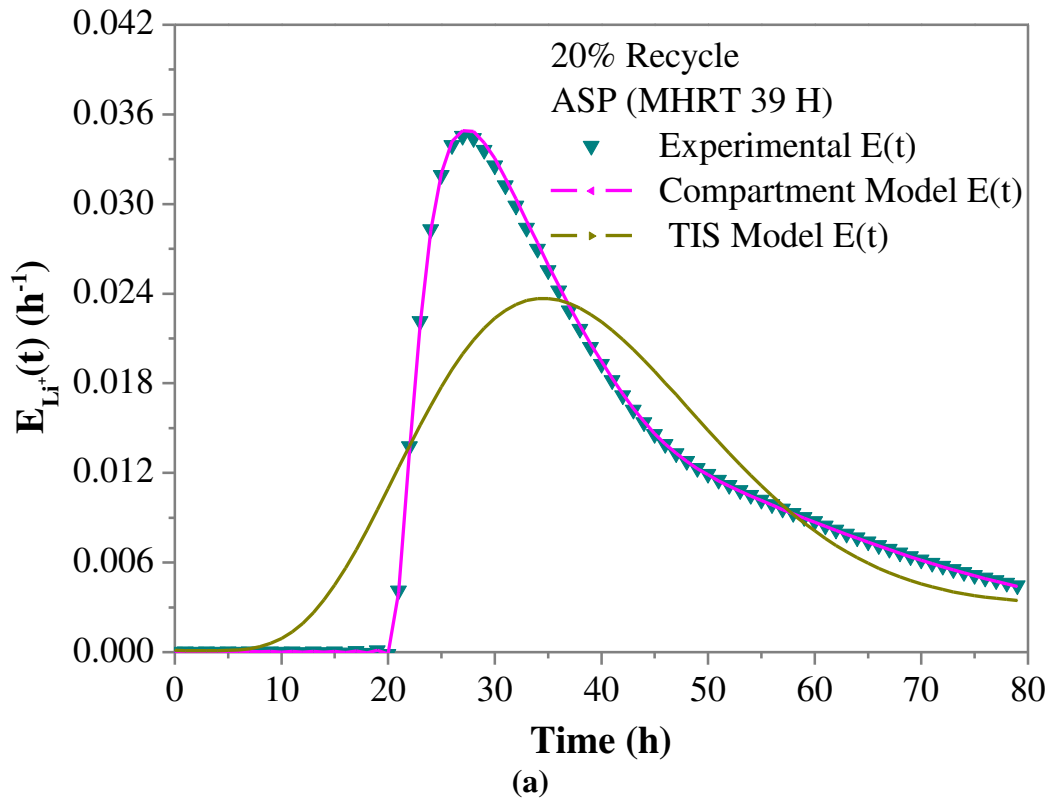


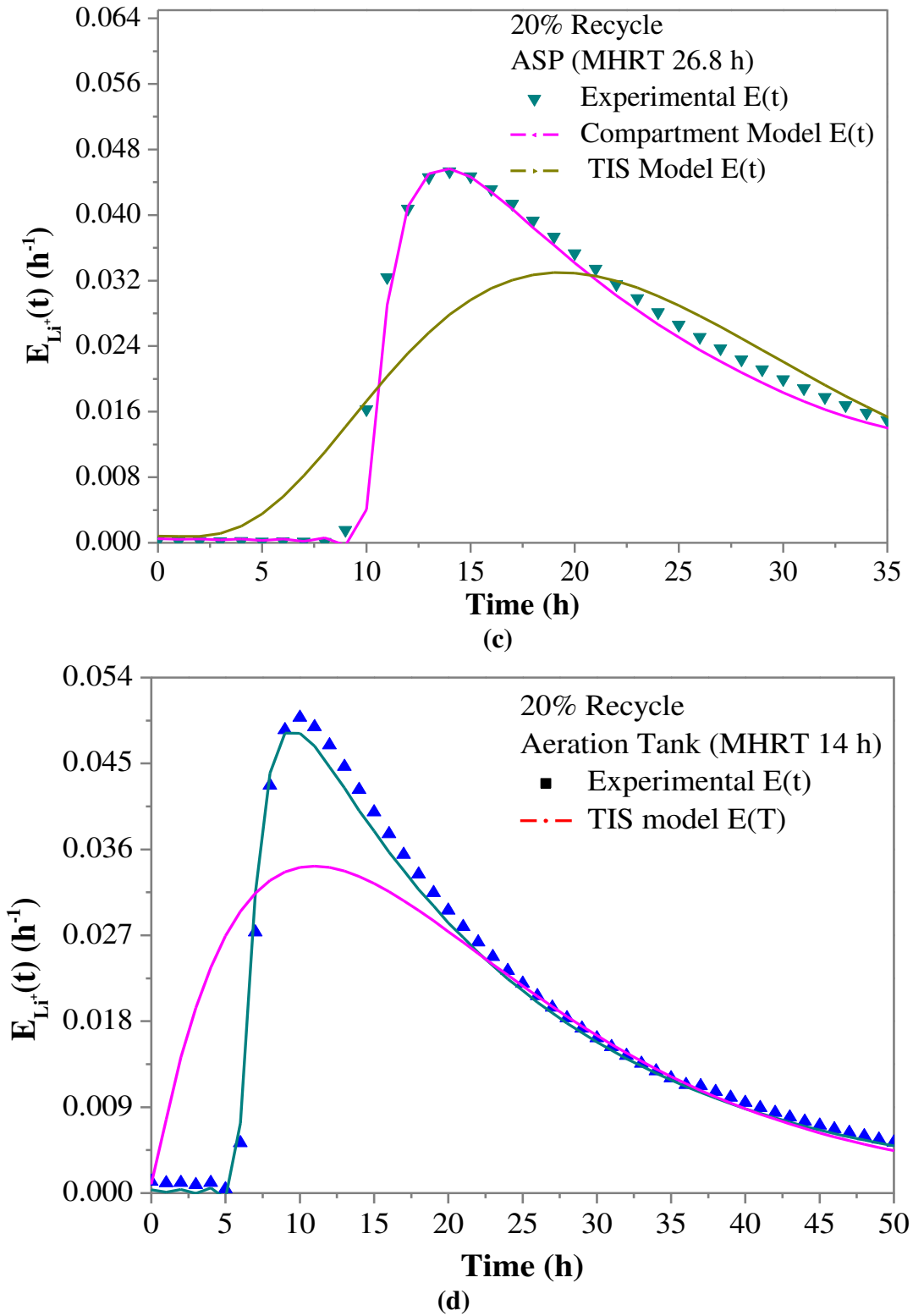
(c)



(d)

Figure 5.14 Comparison of experimentally measured and model-simulated RTD curves at the outlet of the ASP for 10% recycle and MHRT (a) 39 h (b) 33.5 h (c) 26.8 h (d) 21.5 h.





**Figure 5.15** Comparison of experimentally measured and model-simulated RTD curves at the outlet of the ASP for 20% recycle and MHRT (a) 39 h (b) 33.5 h (c) 26.8 h (d) 21.5 h.

**Table 5.2 Parameters of Aeration tank at different MHRT and sludge recycle rate**

Parameters	MHRT of Aeration Tank, (Volume = 12,000 cm <sup>3</sup> )											
	24 h			20 h			16 h			14 h		
	No Recycle	10% recycle	20% recycle	No Recycle	10% recycle	20% recycle	No Recycle	10% recycle	20% recycle	No Recycle	10% recycle	20% recycle
$\bar{t}$ (hr.)	23.77	23.72	23.65	19.82	19.86	19.89	15.89	15.91	15.95	13.88	13.92	13.97
% V <sub>d</sub>	0.95	1.16	1.4	1	0.7	0.5	0.5	0.45	0.25	0.85	0.57	0.2
$\tau_{TISBM}$	23.71	23.67	23.62	19.75	19.76	19.79	15.85	15.87	15.90	13.86	13.90	13.95
%Bypassing	-	-	-	-	-	-	2	1.5	-	7	5	2.1
N		2			1.5			1.2			1	
$\alpha$		2			1.7			1.5			-	
Q (L/hr.)		0.5			0.6			0.76			0.86	

**Table 5.3 Parameters of ASP at different MHRT and sludge recycle rate**

Parameters	MHRT of ASP (Volume = 20,165 cm <sup>3</sup> )											
	39 h			33.5 h			26.8 h			21.5 h		
	No recycle	10% recycle	20% recycle	No recycle	10% recycle	20% recycle	No recycle	10% recycle	20% recycle	No recycle	10% recycle	20% recycle
$\bar{t}$ (hr.)	38.77	23.72	23.65	19.82	19.86	19.89	15.89	15.91	15.95	13.88	13.92	13.97
% V <sub>d</sub>	3.90	5.26		1.7	1.89	2.5	0.8	1.95	2.25	0.75	0.87	1.1
$\tau_{TISBM}$ (h)	23.71	23.67	23.62	19.75	19.76	19.79	15.85	15.87	15.90	13.86	13.90	13.95
$\tau_{ADM}$ (h)	14.98	15.08	15.11	13.2	13.53	13.9	10.5	10.41	10.4	7.4	7.26	7.1
N		2			1.5			1.2			1	
$\alpha$		2			1.7			1.5			-	
Pe		20			15			10			4	
$\tau_R$ (h)		0.33			0.25			0.2			0.16	
Q (L/hr.)		0.5			0.6			0.76			0.86	

## 5.5 Treated effluent-pollution parameters

The comprehensive performance of ASP can only be obtained by taking into account the biological treatment efficiency along with the hydraulic performance. The hydrodynamics of the reactor directly influences the biological treatment efficiency of the tank (Sánchez *et al.*, 2016; Youssef *et al.*, 2017). The knowledge of actual reactor MHRT can prevent incomplete biochemical reactions in ASP and help in growth of desirable microorganisms (Teefy, 1996; Schraa *et al.*, 2016; Knap and Balbierz, 2017). With the optimal combination of operating parameters, optimized ASP performance can be achieved that results in maximum BOD and COD removal from the wastewater (Karches and Buzas, 2013). The intensity of mixing leads to enhanced mass exchange in the aeration tank, hence directly influencing the concentration of residual polluting matter in the wastewater.

The effect of MHRT on the biological treatment efficiency of the aeration tank was studied for MHRT 14, 16, 20 and 24 hours. The substrate removal efficiency of the organic load of the influent was measured in terms of BOD<sub>5</sub> and COD. For each set of MHRT the MLSS and DO concentration was maintained between 3500 - 3800 mg/l and 1.6 - 2 mg/l, respectively. The concentration of BOD and COD attained in the treated effluent at a different set of operating parameters has been given in Table 5.4. The effect of varying MHRT and recycle rate on the treatment efficiency of the aeration tank and ASP is shown in Figure 5.16- 5.19 respectively. The study revealed that the biological treatment efficiency of the effluent increased substantially with an increase in the residence time of the effluent. For the aeration tank operating at MHRT 14, 16, 20 and 24 h without recycle, the percentage substrate removal in terms of COD and BOD were found to be 48%, 54%, 72%, 84% and 53%, 63%, 74%, 86% respectively. The return sludge concentration to the aeration tank was varied for each set of MHRT to 10% and 20% and the subsequent effect on the wastewater treatment efficiency was observed. For the aeration tank operating MHRT of 14, 16, 20 and 24 hours and 10% sludge recycle from the secondary clarifier, the COD removal and BOD removal was found to be 52%, 60%, 77%, 88% and 60%, 68%, 79%, 91%, respectively. Similarly, for the aeration tank operating MHRT of 14, 16, 20 and 24 hours and 20% sludge recycle from the secondary clarifier, the COD removal and BOD removal was found to be 55%, 62%, 80%, 90% and 68%, 72%, 84%, 95%, respectively.

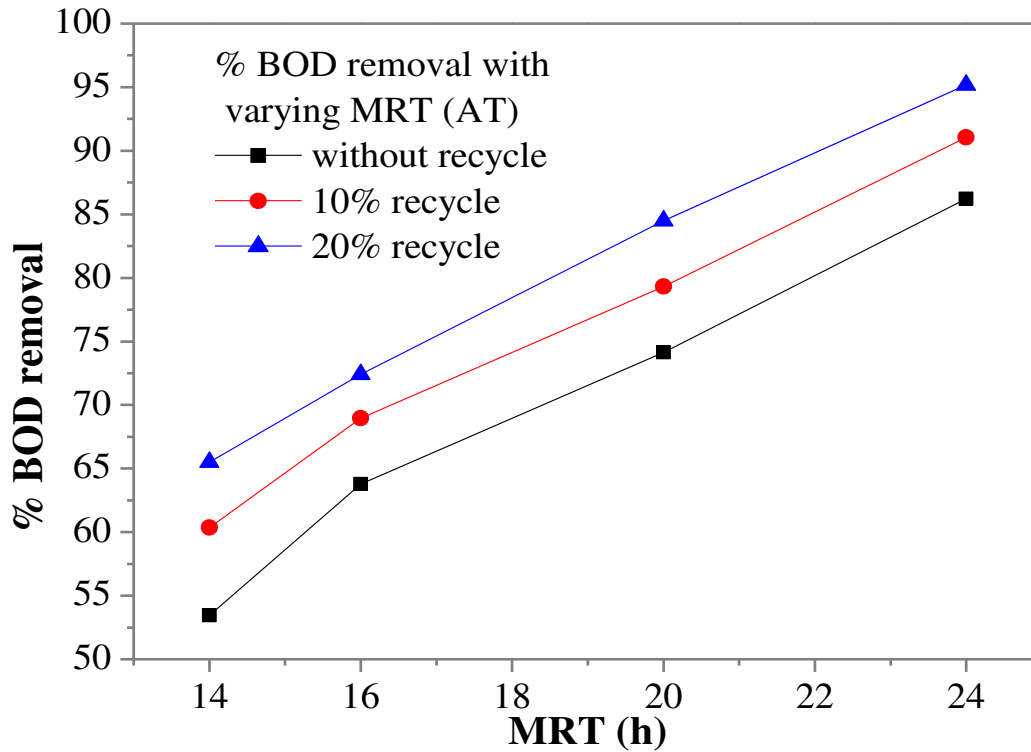


Figure 5.16 Degradation of BOD for aeration tank

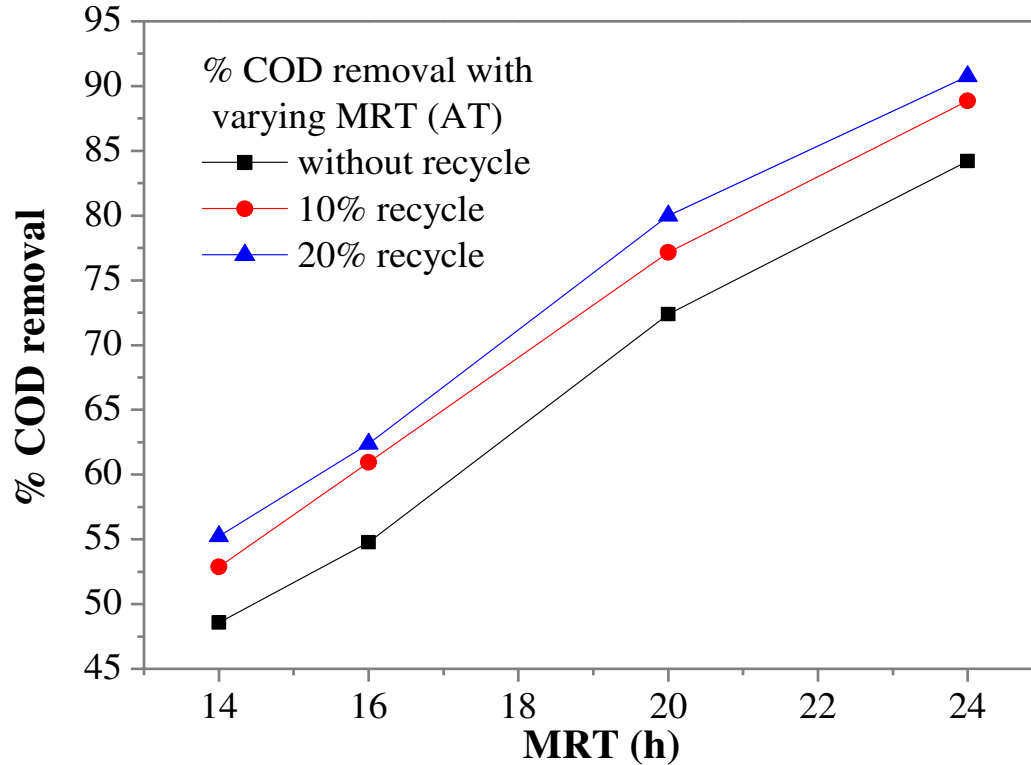


Figure 5.17 Degradation of COD for aeration tank.

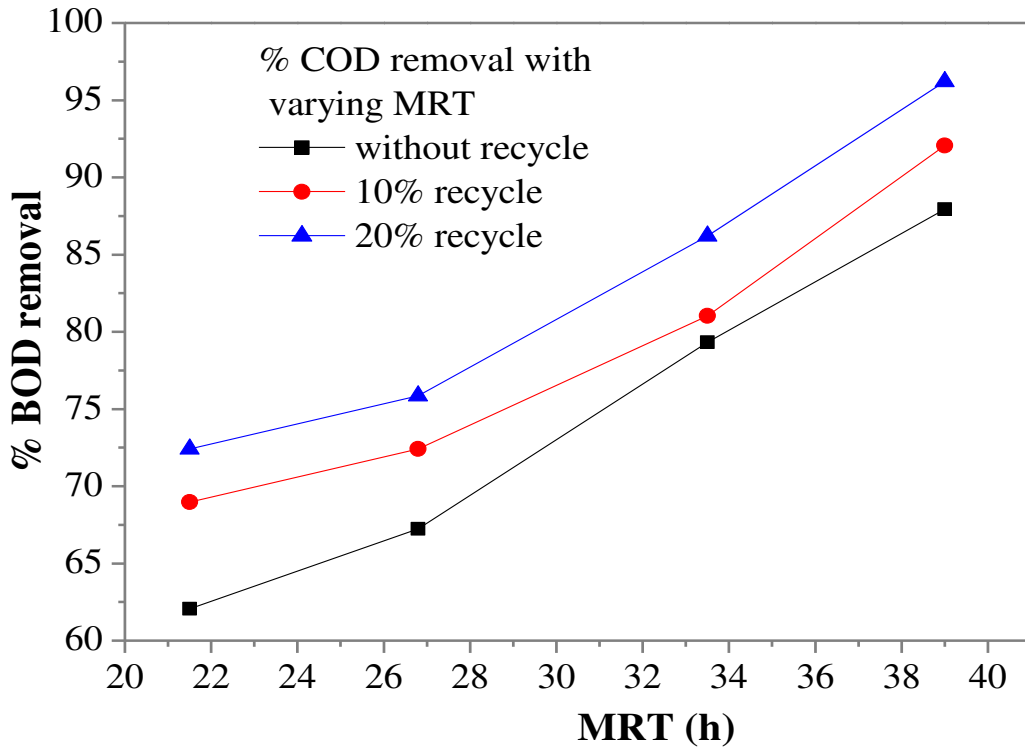


Figure 5.18 Degradation of BOD for ASP.

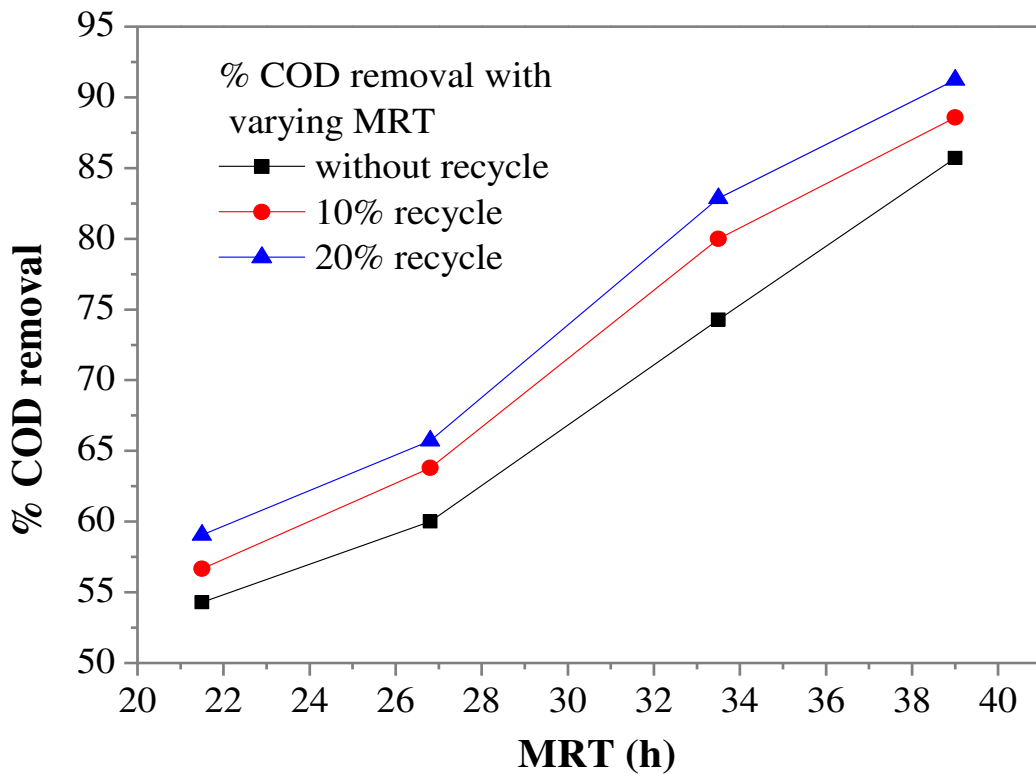


Figure 5.19 Degradation of COD for ASP.

**Table 5.4 Concentrations of BOD and COD obtained at varying MHRT and recycle rate**

	<b>Aeration tank</b>				<b>ASP</b>			
MHRT (h)	14	16	20	24	21.5	26.8	33.5	39
BOD concentration (mg/L) (corresponding to inlet concentration 580 mg/L)								
No recycle	270	210	150	80	220	190	120	70
10%	230	180	120	52	180	160	110	46
20%	200	160	90	28	160	140	80	22
COD concentration (mg/L) (corresponding to inlet concentration 1050 mg/L)								
No recycle	540	475	290	166	480	420	270	150
10%	495	410	240	117	455	380	210	120
20%	470	394	210	97	430	360	180	92
TDS concentration (corresponding to inlet concentration 2350 mg/L)								
No recycle	810	730	650	570	766	690	630	520
10%	758	688	578	510	726	656	566	487
20%	740	640	540	463	694	644	534	434
TSS concentration (corresponding to inlet concentration 1090 mg/L)								
No recycle	760	630	524	442	524	442	342	324
10%	800	690	557	473	557	473	363	357
20%	810	750	570	497	570	497	410	288

For complete ASP working at MHRT 21.5, 26.8, 33.5 and 39 h and no recycle from the clarifier, the COD removal and BOD removal was found to be 54%, 60%, 74%, 85% and 62%, 67%, 79%, 87% respectively. The BOD removal efficiency of the complete ASP was found to increase from 62% to 87% with the increase in residence time from 21.5 h to 39 h. Similarly, ASP working at MHRT 21.5, 26.8, 33.5 and 39 h and 10% recycle rate, the COD removal and BOD removal was found 56%, 63%, 80%, 88% and 68%, 72%, 81%, 92%, respectively. Increasing the recycle rate increased the treatment efficiency of the reactor and ASP working at MHRT 21.5, 26.8, 33.5 and 39 h and 20% recycle rate, the COD removal and BOD removal was found 59%, 65%, 82%, 91% and 72%, 75%, 86%, 96%, respectively. It can be concluded that the biological performance of the complete ASP improved with the increase of recycling rate and the maximum BOD and COD removal efficiency of 96% and 91% was achieved at the MHRT of ASP at 39 hours with 20% of sludge recycle, corresponding to COD and BOD of inlet effluent 1050 mg/L and 580 mg/L. The ASP working at 39 hours MHRT and 20% sludge recycle, the effluent parameter obtained from treating paper mill effluent meet the discharge standards.

The typical design parameters (substrate (BOD) loading rate, food to microorganism (F/M) ratio and solid retention time (SRT)) for ASP working at 39 hour MHRT and 20% sludge recycle were evaluated for each set of experiment and has been given in Table 5.5 . The substrate loading rate or organic volumetric rate is the amount of substrate (BOD or COD) feed into the aeration tank per unit volume of the aeration tank per day. The value of substrate loading rate varies 0.3 to 1.6 kg BOD/m<sup>3</sup>d for complete mixing aeration tank. The food to microorganism or F/M ratio is defined as the ratio of total amount of substrate feed in the aeration tank to total biomass present in the reactor and varies from 0.2 to 0.6 kg BOD/ kg MLSS.d. The SRT is the ratio of total mass of solids in the aeration tank to the amount of solid removed per day and varies from 3 to 40 days depending on the process conditions (Tchobanoglous, 2003). The optimal performance was obtained for aeration tank working at MHRT 24 h and 20% sludge recycle, as all treatment parameters (BOD, COD, TDS, TSS) met the discharge standards set by CPCP, India.

Table 5.5 Design parameters of ASP

MHRT of aeration tank (h)	Recycle rate	$V_L$ (kg BOD / m <sup>3</sup> .d)	F/M (kg BOD / kg MLSS.d)	SRT, $\Theta_c$ (d)
14	20%	0.60	0.11	17
16	20%	0.53	0.10	19
20	20%	0.40	0.09	22
24	20%	0.35	0.08	26

\*d-day

The design parameters calculated for ASP at this operating parameter were well within the range of operational parameters reported in previous literature indicating ideal design of the ASP (Tchobanoglous *et al.*, 2003). The performance of aeration tank and ASP over a range of operating parameters were successfully evaluated.

## **CONCLUSIONS AND FUTURE RECOMMENDATION**

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### **6.1 Conclusion**

The RTD study was performed on the industrial scale ASP as well as pilot scale ASP using tracer technique, hence the conclusion has been divided into two subsections.

#### **6.1.1 RTD study on Industrial scale ASP**

A radiotracer investigation was successfully conducted to study the hydraulic behavior of an ASP system in an integrated pulp and paper industry in India. The flow behavior of the individual sub-systems i.e. the aeration tank-1, secondary clarifier-1, aeration tank-2 and the secondary clarifier-2 as well as the entire ASP was investigated. The following conclusion can be drawn from the study:

- The investigation revealed that there were no major flow abnormalities such as bypassing and parallel flow paths inside the aeration tank-1 and the secondary clarifier-1.
- The dead volume inside the aeration tank-1 and aeration tank-2 was negligibly small. Therefore, both the aeration tanks were operating at its designed hydraulic efficiency.
- The modeling of the measured RTD of the wastewater in the aeration tank-1 revealed that the hydraulic behavior of the aeration tank could be represented by two CSTRs with a low degree of back-mixing between them. Therefore, it can be concluded that there is a good degree of axial mixing of the wastewater in the aeration tank and its behavior tends towards an ideal continuously stirred tank reactor.
- The secondary clarifier-1 was also found to be operating normally without any significant malfunctioning.

- A simple axial dispersion model with recycle was found suitable to describe the flow behavior of the secondary clarifier-1. The significantly high degree of axial mixing predicted in the clarifier (Peclet number,  $Pe = 2$ ) could be due to the recycle of the radiotracer.
- The secondary clarifier-2 was best fitted with simple axial dispersion model and the Peclet number,  $Pe = 10$ , indicates that the clarifier operating approximately as an ideal plug flow reactor.
- A phenomenological model was proposed for the entire activated sludge process system which was a combination of tank-in-series with back-mixing component connected in series with an axial dispersion model component along with a recycle line. The model was found to be fitting very well to the measured RTD of the system.
- The compartment modeling approach applied in our study proved to be very effective tool for providing a suitable representation of the hydraulic behavior of the activated sludge process system in a pulp and paper industry.
- The results of the investigation also helped the industry to take a decision to selectively operate the aerators within the aeration tank with an optimum speed. This led to significant economic benefit due to reduced power consumption.

### **6.1.2 RTD study on Pilot scale ASP**

Residence time distribution study was also performed on a pilot scale activated sludge process to study the effect of mean hydraulic residence time and recycle rate on the hydrodynamic as well as the biological performance of the aeration tank and entire ASP. The following conclusion can be drawn from the study:

- The RTD data obtained at various MHRT of the aeration tank indicated the presence of bypassing stream at lower MHRT (14 h). The bypassing stream disappeared for aeration tank operating at MHRT higher than 16 h.
- For the aeration tank, at the lower MHRTs (14 h and 16 h) the fraction of dead zone were less than 2%. However, dead zone fraction increased at higher MHRT and raised to 4% for 24 h MHRT.

- At lower MHRT (14 h) the aeration tank was well fitted with single mixing tank model which predicts that the reactor was behaving nearly as a perfect mixing tank. The aeration tank for higher MHRT (16 h, 20 h and 24 h) were best fitted with tank-in-series with back-mixing model connected with a bypassing line. The back-mixing ratio was higher at higher MHRT (24 h) which indicated a good degree of axial mixing in the aeration tank.
- The model predicted for the complete ASP comprised of tank-in-series with back-mixing component and axial dispersion model to account for aeration tank as well as secondary clarifier. It was also observed that at higher MHRT (ASP MHRT 39 h) the  $Pe = 20$ , which suggest an ideal plug flow behavior in the clarifier, hence enhanced settling ability of the biomass in the clarifier at higher MHRT.
- The compartment modeling approach adopted to represent the hydrodynamics of the aeration tank and complete ASP was more appropriate compared to the often used conventional tank-in-series model.
- The biological performance of the aeration tank and complete ASP were also simultaneously evaluated in terms of percentage BOD and COD removal efficiency.
- The treatment efficiency was found to increase substantially with the increase of MHRT and recycle rate.
- The effect of MHRT was more evident on the substrate removal efficiency. The BOD and COD removal efficiencies were maximum for ASP operating at MHRT of 39 h with 20% sludge recycle.
- The results obtained from the RTD experiment on a pilot scale ASP shows that varying the operating parameters affects the hydrodynamics as well as biological treatment efficiency of the reactor.
- The present study has immense implication in industrial wastewater treatment reactors as it provides the performance of aeration tank and ASP over a range of operating parameters that can be helpful in deliver information for choosing an optimum working condition in ASP.

## **6.2 Recommendation for future work**

The following recommendations are proposed for future work in the related field.

- The application of convention tracer has immensely boomed as new and improved analytical methods are being developed for their online tracing. Hence, they can be used for industrial scale RTD study.
- The application of convectional tracers in tracking the solid phase has been recently explored and can be used to study the behavior of biomass and sludge in the complete ASP.
- The process in wastewater treatment reactors can be further studied in more details using computational fluid dynamics (CFD) and can be carried out as an extension of this work. CFD is an advanced mathematical modeling techniques that provides validation to the RTD study and helps to further identify shortcoming in the system like short-circuiting streams and dead volumes in the system more specifically.
- Radiotracer particle tracing (RPT) is one of the newer version of tracing technique that has not been exploited for the wastewater treatment system. RPT technique can be explored in ASP to find intricate details of the multiphase flow, interaction of liquid and solid phases in the reactor, local solid and liquid phase velocity fields and a void fractions.

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### 1. *Peer Reviewed (SCI) Journals*

1. **Metali Sarkar**, Vikas Kumar Sangal, Vijay Kumar Sharma, Jitendra Samantray, Haripada Bhunia, Pramod Kumar Bajpai, Anil Kumar, Anil K. Naithani and Harish Jagat Pant, “Radiotracer investigation and modeling of an activated sludge system in a pulp and paper industry”. *Applied Radiation and Isotopes* 2017, 130, 270-275.

2. **Metali Sarkar**, Vikas K. Sangal, Haripada Bhunia, Pramod K. Bajpai, Harish J. Pant, Vijay K. Sharma, Anil Kumar, A. K. Naithani, “Radiotracer investigation of a pulp and paper mill effluent treatment plant”. *Nukleonika* 2017, 62, (4), 289-294.

3. **Metali Sarkar**, Vikas K. Sangal, Haripada Bhunia, “Hydrodynamics and parametric study of an activated sludge process using residence time distribution technique”. *Environmental Engineering Research*, 2019, EER-D-19-00114. (*Accepted*)

### 2. *Under Review/ Preparation*

1. **Metali Sarkar**, Vikas Kumar Sangal, Harish Jagat Pant, Vijay Kumar Sharma, Haripada Bhunia and Pramod Kumar Bajpai, “Application of Tracer Technology in Wastewater Treatment Processes: A Review”. *Water Science and Technology*, 2019. (*Manuscript No. WST-EM19824*).

2. **Metali Sarkar**, Vikas Kumar Sangal, Harish Jagat Pant, Vijay Kumar Sharma, Sunil Goswami Haripada Bhunia and Pramod Kumar Bajpai, “Radiotracer investigation and modeling of aeration tank systems in a pulp and paper mill”. (*Under preparation*)

### **3. Conferences/ Symposia etc.**

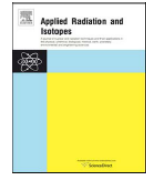
1. **Metali Sarkar**, Vikas K. Sangal, Haripada Bhunia, Pramod K. Bajpai, Harish J. Pant, Vijay K. Sharma, Anil Kumar, A. K. Naithani “Radiotracer investigation and modelling of secondary clarifier in pulp and paper mill effluent treatment plant.”, National symposium on application of radioisotopes and radiation technology in industry healthcare and agriculture (ARRTIHA-2016), November 28-29, 2016.

2. **Metali Sarkar**, Vikas K. Sangal, Haripada Bhunia, Pramod K. Bajpai, Harish J. Pant, Vijay K. Sharma, Anil Kumar, A. K. Naithani “Radiotracer Investigation of a Pulp and Paper Mill Effluent Treatment Plant”, International Conference on Applications of Radiation Science and Technology, Vienna, Austria, 24th -28th April 2017.

3. **Metali Sarkar**, Vikas K. Sangal, Haripada Bhunia, Pramod K. Bajpai, Vijay K. Sharma, Harish J. Pant and Anil K. Naithani “Comparison of aeration tanks in activated sludge process with varying aeration systems” NAARRI International Conference Advanced applications of Radiation Technology (NICSTAR), Mumbai, India, 3-5 March, 2018.

# REPRINT OF PUBLISHED ARTICLES

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## Radiotracer investigation and modeling of an activated sludge system in a pulp and paper industry



Metali Sarkar<sup>a</sup>, Vikas Kumar Sangal<sup>a</sup>, Vijay Kumar Sharma<sup>b</sup>, Jitendra Samantray<sup>b</sup>,  
Haripada Bhunia<sup>a</sup>, Pramod Kumar Bajpai<sup>a</sup>, Anil Kumar<sup>c</sup>, Anil K. Naithani<sup>c</sup>, Harish Jagat Pant<sup>b,\*</sup>

<sup>a</sup> Department of Chemical Engineering, Thapar University, Patiala 147004, Punjab, India

<sup>b</sup> Isotope and Radiation Application Division, Bhabha Atomic Research Centre, Mumbai 400085, Maharashtra, India

<sup>c</sup> Shreyans Paper Ltd., Ahmedgarh, Sangrur, Punjab 148021, India

### HIGHLIGHTS

- A radiotracer investigation was carried out in an activated sludge process system.
- RTDs of an aeration tank and secondary clarifier were measured.
- Mean hydraulic residence times and dead volumes were determined.
- The measured RTDs were simulated using suitable models.

### ARTICLE INFO

#### Keywords:

Activated sludge process, Aeration tank  
Secondary Clarifier  
Radiotracer  
Iodine-131  
Residence time distribution  
Mean hydraulic residence time  
Bypassing  
Dead volume  
Tanks-in-series with backmixing model  
Axial dispersion model

### ABSTRACT

A radiotracer investigation was carried out in an activated sludge process (ASP) system of an effluent treatment plant in a pulp and paper industry. The system consists of an aeration tank and a secondary clarifier connected in series. The primary objective of the investigation was to measure mean hydraulic retention times (MHRTs) of wastewater and investigate the hydraulic performance of the ASP. Residence time distributions (RTD) of the wastewater were measured in an aeration tank and a secondary clarifier of the system using Iodine-131 as a radiotracer. The measured RTD data was treated and MHRTs were estimated. No bypassing was found to exist in the aeration tank and the secondary clarifier. However, the dead volume in the aeration tank and the secondary clarifier was found and estimated to be 2.34% and 4.6%, respectively. The treated curves were further simulated using suitable hydraulically representative mathematical models and detailed flow patterns in the aeration tank and the secondary clarifier were deciphered.

### 1. Introduction

Pulp and paper industry is a water intensive industry that uses water for most of its processes. Water is used in all stages of the paper production, including washing of raw materials, pulping, bleaching and operation of various machines. Water is also used for cooling of equipment, cleaning of systems and generation of steam for process use and on-site electricity production. Although most of the process water is recycled, still a significant amount of wastewater is generated during the pulp washing. The quality and quantity of wastewater generated vary according to the process adopted by specific industry (Kamali and Khodaparast, 2015; Thompson et al., 2001). The untreated effluent or wastewater contains a considerable amount of organics and toxic pollutants, which when discharged in the open atmosphere or water

bodies, causes an adverse effect on human health and the environment. The adverse impact such as excessive solid accumulation, eutrophication, oxygen depletion in the receiving bodies and chemical accumulation in aquatic food web caused by discharge of wastewater from the pulp and paper industries on aquatic system has been studied and reported by many researchers (Afroz and Singh, 2014; Ali and Sreekrishnan, 2001; Andersson et al., 1988; Owens, 1991). Over the years the growing concern about the environment has led water intensive industries such as pulp and paper industry to implement necessary treatment processes to treat wastewater to predefined standards before final disposal in the open atmosphere.

Various wastewater treatment technologies comprising of a combination of primary, secondary and tertiary treatment processes are applied depending on the degree of treatment required (Bajpai, 2001). As

\* Corresponding author.

E-mail address: [hjpant@barc.gov.in](mailto:hjpant@barc.gov.in) (H.J. Pant).

<http://dx.doi.org/10.1016/j.apradiso.2017.10.016>

Received 12 April 2017; Received in revised form 20 July 2017; Accepted 7 October 2017

Available online 10 October 2017

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## Radiotracer investigation of a pulp and paper mill effluent treatment plant

Metali Sarkar,  
Vikas K. Sangal,  
Haripada Bhunia,  
Pramod K. Bajpai,  
Harish J. Pant,  
Vijay K. Sharma,  
Anil Kumar,  
A. K. Naithani

**Abstract.** The pulp and paper industry is highly dependent on water for most of its processes, producing a significant amount of wastewater that should be treated to comply with environmental standards before its discharge into surface-water reservoirs. The wastewater generated primarily consists of substantial amounts of organic, inorganic, toxic and pathogenic compounds in addition to nutrients, which are treated in an effluent treatment plant that often combines primary, secondary, tertiary and advanced treatments. However, the treatment methods vary from industry to industry according to the process utilized. The effective performance of effluent treatment plants is crucial from both environmental and economic points of view. Radiotracer techniques can be effectively used to optimize performance and detect anomalies like dead zones, bypassing, channelling, etc. in wastewater treatment plants. Experiments on the distribution of residence time were performed on the aeration tank and secondary clarifier of a full-scale pulp and paper mill to study the flow behaviour as well as locate system anomalies and hence evaluate the performance of the treatment plants using the radiotracer I-131. The convolution method was applied to model the system with an imperfect impulse radiotracer input. The aeration tank was working efficiently in the absence of any dead zones or bypassing. Various hydrodynamic models available in the literature were applied on the aeration tank and secondary clarifier to obtain the hydraulic representation of the systems.

**Keywords:** aeration tank • secondary clarifier • residence time distributions RTD • radiotracer • convolution

M. Sarkar, V. K. Sangal<sup>✉</sup>, H. Bhunia, P. K. Bajpai  
Department of Chemical Engineering,  
Thapar Institute of Engineering and Technology,  
Bhadson Road, Patiala, Punjab 147004, India,  
Tel.: +91 981 501 5705,  
E-mail: vksangal@gmail.com

H. J. Pant, V. K. Sharma  
Isotope Production and Application Division,  
Bhabha Atomic Research Centre,  
Mumbai, India

A. Kumar, A. K. Naithani  
Shreyans Industries Ltd.,  
Village Malikpur, Sangrur District, Ahmedgarh,  
Punjab 148021, India

Received: 21 April 2017  
Accepted: 7 November 2017

### Introduction

The Indian pulp and paper industry is a major consumer of water, utilizing around 60–120 m<sup>3</sup> of water per ton of paper produced, depending on the type of process used for paper production [1]. The process of manufacturing paper and pulping generate tons of wastewater that contains large quantities of pollutants which are unfit to be directly disposed of in surface-water reservoirs. Wastewater from the pulp and paper industries contains a significant amount of organic, suspended solids, nutrients and occasionally toxic compounds [2, 3]. However, the organic load of this industry requires special attention as it causes the maximum amount of pollution and has widespread implications for what is exposed to it [4, 5].

The activated sludge process (ASP) is one of the most commonly used processes for the biological treatment of dissolved organics in the pulp and paper industries. The aeration tank, used widely for the removal of dissolved organic compounds in the wastewater, is the most crucial part of the ASP. It is essentially a bioreactor that employs active microorganisms suspended throughout the tank. The



DOI: <https://doi.org/10.4491/eer.2019.114>

## Hydrodynamics and parametric study of an activated sludge process using residence time distribution technique

Metali Sarkar<sup>1</sup>, Vikas K. Sangal<sup>2†</sup>, and Haripada Bhunia<sup>1</sup>

<sup>1</sup>Department of Chemical Engineering, Thapar Institute of Engineering and Technology, Patiala-147004, Punjab, India

<sup>2</sup>Department of Chemical Engineering, Malaviya National Institute of Technology, Jaipur-302017, India

Corresponding Author: Vikas K. Sangal, Tel: +91-981-501-5705, Email: [vksangal.chem@mnit.ac.in](mailto:vksangal.chem@mnit.ac.in)

Received: March 20, 2019; Accepted: May 28, 2019.

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### ABSTRACT

Hydrodynamic study of Activated Sludge Process (ASP) is important to optimize the reactor performance and detect anomalies in the system. Residence time distribution (RTD) study has been performed using LiCl as tracer on a pilot scale aeration tank (AT) and ASP, treating the pulp and paper mill effluent. The hydraulic performance and treatment efficiency of the AT and ASP at different operating parameters like residence time, recycle rate was investigated. Flow anomalies were identified and based on the experimental data empirical models was suggested to interpret the hydrodynamics of the reactors using compartment modelling technique. The analysis of the RTD curves and the compartment models indicated increase in back-mixing ratio as the mean hydraulic retention time (MHRT) of the tank was increased. Bypassing stream was observed at lower MHRT. The fraction of dead zone in the tank increased by approximate 20-25% with increase in recycle rate. The fraction of the stagnant zone was found well below 5% for all performed experiments, which was under experimental error. The substrate removal of 91% for COD and 96% for BOD were observed for the ASP working at a hydraulic mean residence time 39 h MRT with a 20% recycling of activated sludge.

**Keywords:** [Activated sludge process](#) | [BOD](#) | [Compartment models](#) | [Hydrodynamics](#) | [Residence time distribution](#) | [Tank-in-series model](#)

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