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Residence Time Distribution: Literature Survey, Functions, Mathematical Modeling, and Case Study—Diagnosis for a Photochemical Reactor

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Abstract: This paper aims to provide an overview of the fundamentals, development, and evolution of residence time distribution (RTD) methodology and its applications to the flow and mixing of fluids (and solid particles) modeling in different systems. A concise literature analysis is followed by a succinct presentation of RTD methodology's experimental and theoretical foundations and RTD-based mathematical modeling, highlighting its importance. An experimental demonstration of RTD diagnostics on a photochemical reactor is performed to identify the most practical locations for the inlet/outlet pipes (axial or radial) and the photochemical reactor's ideal working posture (horizontal, vertical, or inclined) and to understand the level of mixing and to determine the fluid flow defects. Using the relevant RTD functions and the corresponding central moments, it was possible to show that short circuits and dead zones occurred in each of the six considered reactor configurations. Following these investigations, design solutions were proposed to achieve a convenient exposure time, proper mixing, and uniform irradiation inside the reactor.

Keywords: tracer methodology; non-ideal flow; mixing degree; photochemical reactor

1. Introduction

The flow behavior of materials inside reactors or other flow systems can be studied using residence time distribution (RTD). Processes and systems can experience delays, mixing, and other effects that can be analyzed using RTD, which measures the time it takes for a fluid or solid particle to travel through the system. The method's fundamental premise is that the individual fluid elements or particles spend different amounts of time inside a reactor or system. The residence times can be statistically examined using probability distribution functions. For the RTD methodology, three main stages may be identified: (i) experimental, (ii) statistical analysis, and (iii) mathematical modeling. In the experimental stage, a tracer material is typically injected into the system, and its concentration is measured as a function of time from its insertion to its complete exit from the system. The resulting concentration-time plot serves as the starting point for the following phase—statistical analysis. Statistical functions (e.g., probability density function, cumulative distribution function) are employed at this stage to assess the experimental data and derive valuable parameters (e.g., mean residence time, variance) to explain the flow and/or mixing behavior. Ideal and non-ideal mathematical models are further developed to simulate, compare, and/or predict fluid flow and mixing and reaction yields (for known kinetics) [1].

RTD technology was initially developed for chemical reactors, but its scope was quickly broadened to include virtually any industry where material flow and/or mixing are involved. Given its foundation and historical evolution, RTD can be called a "mature"



Citation: Nechita, M.T.; Suditu, G.D.; Puiţel, A.C.; Drăgoi, E.N. Residence Time Distribution: Literature Survey, Functions, Mathematical Modeling, and Case Study—Diagnosis for a Photochemical Reactor. *Processes* 2023, *11*, 3420. https://doi.org/10.3390/ pr11123420

Academic Editor: Roberto Pisano

Received: 13 November 2023 Revised: 6 December 2023 Accepted: 11 December 2023 Published: 13 December 2023



Copyright: © 2023 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). method. Nevertheless, it is far from being complete or outdated, with new models and applications reported on a regular basis in the literature.

The current work is structured as follows: (i) a quick overview of the history and uses of RTD methods; (ii) a succinct explanation of the fundamentals of the tracer methodology; (iii) a brief presentation of the main RTD functions and central moments; (iv) a short introduction in RTD based mathematical modeling; and (v) a case study: RTD diagnosis for a photochemical reactor, followed by some practical solutions addressing the position of inlet/outlet reactor pipes.

2. RTD Development: Defining Moments in Time, Representative Works, Current Trends

There is a debate about the implementation of residence time distribution. According to some authors [2–5], the genuine pioneers of this method were MacMullin and Weber in 1935 [6]. A few authors [7,8] go back in science history to attribute the first attempts to study fluid flow to Irving Langmuir in 1908 [9]. Many others [5,10–12] credit Dankwerts, in 1953, with developing the method, as he was the one who devised the mathematical foundation [13]. The RTD approach was improved [14–18], critically analyzed [19,20], revisited [10,21], and reviewed [4,7,22,23] in the 70 years that followed Dankwerts' study, and its applications were expanded to a wide range of areas. Table S1 presents the historical progress and an analysis of the RTD method.

It is considered that the RTD procedure was fully developed at the end of the 1990s. Moreover, as Dudukovic remarked in his study "Tracer methods in chemical reactors; Techniques and applications" [11], for more than 50 years, the RTD methodology has been covered in standard chemical reaction engineering textbooks (Table S2).

The methodology developed to describe the flow of fluids in chemical reactors has since spread and been used in other fields. It is used nowadays not only to describe fluid flow but also to describe mixing and solid flow. Several reviews written after the 1990s enumerate and highlight the tremendous development of this approach (examples in Table S3).

Over 1600 articles dealing with "residence time distribution" were indexed by the Web of Science Core Collection between 2016 and 2017, according to a review published by Berard et al. [3]. Using the SCOPUS platform and the VOSviewer software version 1.6.19 we conducted a similar bibliometric search (Figure 1) that yielded 464 results for "residence time distribution" search term in title/abstract/keywords for the year 2022 alone. The five significant clusters identified by VOSviewer were residence time distribution, residence time, particle size, retention time, and hydrodynamics.

Nowadays, the applications of RTD go far beyond the mathematical modeling of chemical reactors. The information revealed by statistical residence time analysis has found various applications in different fields. Few examples include: (i) fluids and particulate solids [24] flow and mix through various systems, from small laboratory scale [25] to large industrial applications [26,27], from human body [28] to geological studies [29]; (ii) equipment design—reactors (vessels) sizing [30], reaction yield predictions [31]; (iii) diagnosis and troubleshooting for existing systems [27]; and (iv) experimental validation of software modeling [32,33].

The RTD methodology and the RTD-based mathematical modeling are still developing [34–36]. Various authors frequently propose new models in different domains [37–39]. Even though powerful computational modeling methods (e.g., computational fluid dynamics—CFD) have been developed since the appearance of RTD, these still require experimental validation, usually performed using tracer experiments [40–42].



Figure 1. The bibliometric map for "residence time distribution" for 2022 using SCOPUS.

3. Tracer Methodology Fundamentals

Tracer methodology (Figure 2) is a powerful tool for studying the behavior of fluids and/or particles in complex systems. Although numerous approaches have been suggested throughout the years, there is still a debate on the accuracy of this methodology [10,11,19,43]. The procedure was extensively described by Octave Levenspiel [44]. However, there are still issues related to the type of the tracer, how it is inserted into the flow, the vessel boundaries, and how its signal is detected and eventually converted to finally plot the concentration vs. time curve (the C-curve) [7,45–48]. In brief, the tracer methodology is an experimental procedure that introduces a known amount of tracer into a system and measures its concentration over time at different points (most commonly at the exit).



Figure 2. The principle of tracer methodology (pulse input).

3.1. Tracer Selection

Ideally, the tracer should possess similar flow properties to the material of interest and the capacity to be easily measured or detected. Moreover, the tracer must be detectable at very low concentrations, and the measurement technique must be sensitive and accurate. Since the tracer method is applied in various domains [11], including chemical engineering [3], environmental engineering [46], metallurgy [49], hydrology [48], pharmaceutics [5,50], medicine [51], and many others, there is a large variety of tracers such as electrolytes, dyes, stable or radioactive isotopes, [52] and others [3]. Conductivity, fluorescence, color, X-ray emission, or even temperature are among the properties that allow tracer detection, and most of the time, calibration curves are used to convert a specific property into tracer concentration.

The proper selection of the tracer is critical for the accuracy of the data acquisition [53]. For instance, a relatively minor difference between tracer's and the targeted material density, viscosity, temperature etc., can generate negative or positive buoyancies [54]; a slight difference between tracer particles' size and density can lead to powder segregation [55]; all these differences can affect the experimental results, and consequently the flow diagnosis. Although inert (non-reactive) tracers and steady flow are generally advised, attempts to study reactive tracers and unsteady flow were also reported [56,57].

3.2. Tracer Insertion

There are four ways to introduce the tracer into a system: (i) as a pulse, when a known amount of tracer is injected as fast as possible (assumed instantaneously); (ii) as a step, when a steady flow with a constant tracer concentration is introduced for a certain amount of time (iii) periodic, following a specific function (e.g., sinusoidal input) and (iv) random, arbitrary [44]. The first two methods are the most popular due to their practical and mathematical simplicity (Figure 3) [3,10,53]. The tracer amount is critical: it should be large enough to reach detection limits while remaining small enough to be injected at once or as quickly as possible.



Figure 3. System response (dotted line) to pulse and step inputs.

3.3. System Boundaries

The principle of the tracer methodology (pulse input) is presented in Figure 2. To compute the response curve accurately, the injection/detection position should be adequately chosen (Figure 2), and the boundary conditions at both ends of the system must be known (Figure 4) [58].



Figure 4. System boundaries (L denotes the length of the system under consideration) (**A**): open–open; (**B**): closed–closed; (**C**): closed–closed; (**D**): open–closed.

The fluid flow in an open system is virtually unaffected as it passes through. In contrast, in a closed system, there are some fluctuations in axial dispersion at the system's inlet and outlet (Figure 4) [3,58].

3.4. Practical Limitations

Even after selecting the proper tracer and detection method, several practical limitations are usually overcome by experiment repetition. The term "instantaneously" is relative and, from a practical point of view, impossible. During step injection, the tracer should be instantaneously and continuously introduced starting at t = 0, while for pulse input, the entire amount of tracer should be instantaneously introduced at t = 0 [3]. The injection time depends on the tracer amount, which directly affects the detection limit, a severe limitation of tracer methodology [59]. The detector response and sensitivity are crucial when conversion to concentration is achieved. The sudden injection causes jet formation that may affect the tracer dispersion and possibly the flow in the analyzed system, inducing perturbations. The relative position of the injection vs. detection points towards the system boundaries may also affect the experimental accuracy [59].

4. RTD Functions and Central Moments

Four functions and four central moments can be used for calculating residence-time distributions (Figure 5). However, only two functions and two central moments (red in Figure 5) are commonly used for simple flow diagnosis.



Figure 5. RTD statistical analysis.

4.1. Residence Time, Space-Time, Mean Residence Time, and Dimensionless Time

The foundation of RTD statistical analysis is the straightforward observation that the fluid elements simultaneously entering a system may spend different periods before leaving the system. From this perspective, the concepts of age, life expectancy, and residence time of a fluid element are easily comprehensible. The existence of fluid populations of a specific age or a certain life expectancy gives meaning to the mean residence time concept. The statistical study of all residence periods at or after a given time provides essential details regarding the fluid flow and mixing intensity within a system.

The nominal holding time or the space-time t_s (Equation (1)) is defined as the ratio between the total reactor volume (V, m³) and the volumetric flow rate of the fluid $(M_v^0, m^3/s)$:

 t_s

$$=\frac{V}{M_{v}^{0}}$$
(1)

It is a common practice to use the dimensionless time (Equation (2)), given by the ratio between the actual time t and the space time [44]:

$$\theta = \frac{t}{t_s} \tag{2}$$

4.2. *The C*-*Curve*: C = f(t)

The C-curve displays the time evolution of the tracer concentration and provides the raw data for RTD investigation. Although often overlooked in RTD presentations, it is the first and most crucial function established exclusively experimentally using the tracer methodology. The precision with which the concentration vs. time dependence is plotted gives the accuracy of the subsequent RTD statistical analysis. For an experienced researcher, the shape of the C-curve can provide acceptable information about some typical flow defects (e.g., short circuits or dead zones). The time the tracer spends inside the vessel can be statistically examined using the RTD functions and their central moments. Based on the results (particular shape of a specific function and/or central moment values), fluid flow and mixing degree knowledge can be derived.

4.3. The E-Curve: E(t)

This function is known as "the residence time distribution" or "exit age distribution". The exit age distribution considers that different fluid particles may experience different residence times within the system before exiting. It is a function of time, where E(t) dtrepresents the fraction of fluid elements leaving the system of age between t and t + dt(Figure S1), Equation (3). The units of E-curve are $[time^{-1}]$.

$$\int_{0}^{\infty} \mathbf{E}(\mathbf{t}) \cdot d\mathbf{t} = 1 \tag{3}$$

According to its definition: $\int_{0}^{t_1} E(t) \cdot dt + \int_{t_1}^{\infty} E(t) \cdot dt = 1$. The fraction of fluid elements with residence times less than t_1 is given by: $\int_{0}^{t_1} E(t) \cdot dt$.

The E-curve is useful when dealing with flow diagnosis. The shape of the curve reveals essential information about potential flow defects in the analyzed system.

4.4. The F-Curve: F(t)

The fraction of fluid elements with a residence time less than t is defined as the cumulative residence time distribution function F(t). It is also the probability that a fluid element that entered at t = 0 has left the system at or by time t [60]. According to its definition, the equalities presented in Equation (4) are valid. The typical shape of the F-curve is presented in Figure S1.

$$F(0) = 0 \text{ and } \lim_{t \to \infty} F(t) = 1$$
 (4)

$$\mathbf{F}(\mathbf{t}) = \int_{0}^{\mathbf{t}} \mathbf{E}(\mathbf{t}) \cdot d\mathbf{t}$$
(5)

$$E(t) = \frac{dF(t)}{dt}$$
(6)

Since it is defined as a fraction, it is dimensionless. According to its definition: $0 \le F(t) \le 1$. The connections between E-curve and F-curve are given by Equations (5) and (6).

The F-curve is less sensitive to flow defects than the E-curve [1] but provides valuable information about the mixing degree [61].

4.5. *The I-Curve: I(t)*

The internal age distribution measures the distribution of fluid elements ages inside the system, not on the system's exit as the exit age distribution. I(t) dt represents the fraction of fluid elements within the system, with the age between t and t + dt (Figure S1). I(t) has the same [time⁻¹] units as the E(t) function. There are some connections between the E(t), F(t), and I(t) functions, as shown in Equation (7).

$$\mathbf{I}(\mathbf{t}) = \frac{1}{\mathbf{t}_{s}} \cdot \left[1 - \mathbf{F}(\mathbf{t})\right] = \frac{1}{\mathbf{t}_{s}} \cdot \left[1 - \int_{0}^{\mathbf{t}} \mathbf{E}(\mathbf{t}) \cdot d\mathbf{t}\right]$$
(7)

where t_s is the space-time (Equation (1)):

The geometry and flow pattern of the system determines the shape of the I-curve, which is an essential tool for comprehending the behavior of fluids inside a system.

4.6. The λ -Curve: $\lambda(t)$

The intensity function $\lambda(t)$ is defined such that $\lambda(t) \cdot dt$ indicates the fraction of fluid that will leave the system in the time interval between t and t + *dt* or, in terms of internal ages, the fraction of fluid elements with zero life expectancy (that will immediately leave the system) [10]. The typical shape of the intensity function is presented in Figure S1. The connections between the $\lambda(t)$, E(t), and F(t) functions are given by Equation (7). The intensity function allows for minimum and maximum points, frequently related to flow imperfections such as short-circuits and stagnancies. The shape of the λ -curve also provides information about the mixing degree.

$$\lambda(t) = \frac{\mathrm{E}(t)}{1 - \mathrm{F}(t)} = \frac{\mathrm{E}(t)}{t_{\mathrm{s}} \cdot \mathrm{I}(t)}$$
(8)

4.7. Dimensionless Representation of RTD

Dimensionless time is commonly used in RTD analysis. The representation of RTD functions in terms of θ is known as "normalization" (Table 1). The normalization of the RTD functions allows the direct comparison of flow characteristics in different size systems [62].

Table 1. The "normalized" expressions of the RTD functions.

	Properties			
$E_{\theta}(\theta) = t_s \cdot E(t)$	$F_{\theta}(\theta) = F(t)$	$I_{\theta}(\theta) = t_s \cdot I(t)$	$\lambda_{\theta}(\theta) = t_s \cdot \lambda(t)$	$\int_{0}^{\infty} \mathbf{E}(\theta) \cdot d\theta = 1$

4.8. The RTD Central Moments

The central moments of the RTD (Table 2) provide supplementary information about the properties of the functions. Rigorously, all moments must be determined for a complete distribution description. Commonly, the first three moments are calculated to reasonably characterize an RTD [62].

Description	Symbol	Label	Mathematical Expression	Significance
First moment	τ	Mean residence time	$ au = \int\limits_{-\infty}^{\infty} \mathbf{t} \cdot \mathbf{E}(\mathbf{t}) \cdot d\mathbf{t}$	Central tendency
Second moment	σ^2	Variance	$\sigma^2 = \int_{0}^{\infty} (t - \bar{t})^2 \cdot E(t) \cdot dt$	Degree of dispersion around the mean
Third moment	s ³	Skewness	$\mathrm{s}^3 = \int\limits_0^{\mathrm{g}} \left(\mathrm{t} - \bar{\mathrm{t}} ight)^3 \cdot \mathrm{E}(\mathrm{t}) \cdot d\mathrm{t}$	Symmetry (positive or negative)
Fourth moment	k^4	Kurtosis	$k^4 = \int_0^\infty (t - \bar{t})^4 \cdot E(t) \cdot dt$	Shape (tall or flat)

Table 2. Central moments of residence time distribution.

The first moment gives the mean residence time of the fluid elements inside the studied vessel or system. The second moment commonly used, δ^2 , is taken for the mean and is called variance, or square of the standard deviation [62]. The variance indicates the mixing degree: a higher value indicates intense mixing, while a lower value indicates a lower mixing degree. Skewness, s³, is the third moment of a probability distribution that measures the distribution's asymmetry. The fourth moment, kurtosis—k⁴, measures the "peakedness" or "tailedness" of the RTD. A higher kurtosis indicates a sharper peak and a narrower distribution, while a lower kurtosis indicates a flatter peak and a wider distribution.

It is critical to understand that using all functions and central moments is not always necessary. Specific functions and central moments are required, while others are optional, depending on the study's purpose (e.g., flow diagnosis, reactor design, mixing analysis). For instance, it is not always necessary to calculate the intensity function or the skewness; a simple flow diagnosis does not necessitate such complex modeling as, for example, a second-order reaction in a mechanically stirred (autoclave) reactor.

5. RTD Based Modelling

Over time, a multitude of models were proposed by various authors in an attempt to describe the real fluid flow and/or mixing behavior. Some of these models received general recognition and nowadays represent a standard in the area [1,62,63]. Mathematical modeling uses RTD functions and central moments in conjunction with idealized models to represent fluid flow and mixing in real systems. Mathematical modeling can be extended further by integrating chemical reaction kinetics to forecast conversion and reaction yield.

5.1. The Ideal Flow Models

There are two distinct ideal flow models (Figure 6) that serve as benchmarks for fluid flow modeling: the plug flow (piston flow) and perfectly mixed flow (complete mixing) [10,44]. Each model is founded on a unique set of assumptions.



Figure 6. Typical representations of ideal flow models: (**A**): completely mixed, (**B**): plug flow, (**C**): laminar flow.

5.1.1. The Plug Flow Model

This model assumes that the fluid's velocity is constant along any pipe cross-section perpendicular to the pipe's axis. In addition, it is assumed that the system is completely filled with fluid, the diffusion terms are negligible, the volumetric flow rate is constant, and the radial gradients are ignored. There is no mixing of earlier with later entering fluid, no overtaking, and no backmixing, eddies, or vortexes [44]. All fluid elements flowing through the system have the same residence time.

A particular derivative of the ideal plug flow model is the laminar flow (LF) model (Figure 6) [7]. The fundamental difference between them is the shape of their velocity profiles. The velocity profile for the plug flow model is flat across the cross-section, whereas for LF, the fluid velocity increases steadily towards the center, forming a parabolic velocity profile. As a result, the fluid constituents in an LF have varying residency times: those in the center leave the system first, while those along the wall leave last.

5.1.2. The Complete Mixed Flow

The model assumes that the fluid elements are always perfectly mixed inside the system. Therefore, all properties (e.g., temperature, concentration, density) are uniform inside the reactor at any given time. In addition, it is assumed that the fluid instantly and uniformly mixes with the system's contents upon entry [44,64] and that the properties inside the system are identical to those in the outlet flow.

Although fluid flow and fluid mixing cannot be considered separately in real-world systems, two ideal mixing models were proposed for modeling purposes: the segregation model and the maximum mixedness model (Figure 7).



Figure 7. Typical representation of the segregation and maximum mixedness models: (**A**): segregation model; (**B**): maximum mixedness model.

5.2.1. The Segregation Model

According to this model, the fluid elements move and travel independently through the system. As a result of the hypothetical boundaries that separate fluid elements, a chemical reaction can only occur inside the molecules of a fluid element and not between molecules of two distinct elements. A fluid exhibiting complete or total segregation is called a macrofluid [62].

5.2.2. The Maximum Mixedness Model

There are no boundaries among the fluid elements. In fact, there are no fluid elements. The fluid molecules, regardless of their age inside the system, are perfectly mixed, and there is no segregation at all. Such a fluid is also known as a microfluid [62].

5.2.3. The Earliness and Lateness of Mixing

Another aspect that relates to the fluid flow and mixing models is the timing of mixing (the earliness or lateness of the mixing). A single stream's fluid elements may mix as they enter the system or later as they pass through it. The timing of mixing is crucial for systems with multiple streams (such as two reactant streams entering a chemical reactor). Gorzalski et al. [65] proposed a suggestive visual representation of earliness and lateness of mixing for a two-stream system (Figure 8).



Figure 8. Typical representations for ideal earliness or lateness of mixing: (**A**): perfect segregation; (**B**): perfect (instantaneous) mixing; (**C**): early segregation, late mixing; (**D**): early mixing, late segregation.

5.3. Flow and Mixing in Real Systems—Non-Ideal Flow

In real systems (non-ideal flow), the fluid may have complex flow patterns or exhibit mixing that is not uniform or predictable [66]. Compared to ideal models, the fluids behave differently for many reasons (such as the vessel's geometry, the type, size, location of the inlet and outlet pipes, the mixer type, the fluids' properties, and the fluid flow rate). The mixing degree is also affected by non-ideal flow. Some of these deviations from ideality are particularly recognized and acknowledged, such as dead zones, stagnant regions, back-mixing, short-circuits (bypasses), and preferential flows (channeling) [66].

Although the terms "dead zone" and "stagnant region" are sometimes used interchangeably in fluid flow systems, they typically refer to slightly different concepts (Figure 9). A dead zone is a portion of the system that is not filled, also known as a free volume, while a stagnant region is a part of the system where the fluid is rather stationary. Because the fluid is only slightly moving or not moving in this region, it is also known as the "dead zone".



Figure 9. Typical representations of flow defects.

Short-circuiting or bypassing occurs when flow elements cross the system quickly, resulting in uneven mixing and short residence times. The most common cause of this undesired flow defect is incorrect inlet–outlet pipe placement.

Preferential flow or channeling typically occurs when fluids flow through solid beds (e.g., catalysts, packed columns). The fluid follows the minimal resistance pathway. Systems of fluids with significant variations in the fluid's properties (e.g., viscosity, density) may also exhibit preferential flow patterns.

5.4. Dimensionless Numbers: Péclet, Bodenstein, Damköhler

The Péclet (Pe) number is the ratio between convective and diffusive transport rates (Table S4). It determines whether a system's mass transport is dominated by convection or diffusion. If Pe << 1, the mass transfer occurs mainly by the diffusion mechanism; if Pe >> 1, the mass transfer occurs mainly by the convection mechanism; if Pe = 1, both mechanisms contribute equally to mass transfer. Fogler [62] defines two Pe numbers, one to characterize the reactor—the reactor Pe (Pe_r) and one to characterize the fluid—the fluid Pe (Pe_f). The Pe_f is commonly used in literature as the Péclet number Pe. When the characteristic length is the reactor length, and the diffusion is mainly axial, the Pe_r is referred to as the Bodenstein number (Table S4)—typical for reactive systems [62].

Bodenstein number (Bo) is defined similarly to Pe_r, yet the values of the Bo number usually refer only to the axial mixing intensity (backmixing). Higher Bo numbers indicate reduced axial mixing, while lower Bo numbers indicate higher mixing degrees.

Damköhler number (Da) is the ratio between reaction and convection rates [62]. If Da >> 1, the reaction rate is higher than the transport rate (the reaction is instantaneous), while if Da << 1, the time required to travel through the system exceeds the time required to complete the reaction. The expression of the Damköhler number depends on the reaction order, just as the expression of the reaction rate depends upon the reaction order (Equation (9)).

$$Da_{n} = k \cdot \left(C_{A}^{0}\right)^{n-1} \cdot t \tag{9}$$

where k represents the specific reaction rate, n represents the overall reaction order, and C_A^0 represents the initial concentration of the reactant A.

5.5. Non-Ideal Flow and Mixing Models

According to Fogler [62] the non-ideal flow models can be grouped into two categories: (i) one parameter models, which include the tank-in-series model (Figure S2 and Table S5) and axial dispersion model including its boundaries (Figure 4) combinations (the parameter is D_{ax}) and (ii) two parameters models, where the reactor is modeled using combinations of ideal reactors (e.g., the Cholette–Cloutier model associate a perfectly mixed tank with a dead zone and a by-pass region). Ravi [67] proposed a third group: zero model parameters (including complete segregation and maximum mixedness models).

Rodrigues [10] proposed two classes of models. First, the compartment models, where the actual models are various combinations of seven basic concepts called "nuclei", which includes a perfectly mixed system, a plug flow system, a laminar flow system (Figure 6), and a recycle region, a by-pass region, a dead zone, and a stagnant region (Figure 9). The second category includes the dispersion models, considering axial and/or radial dispersion, backmixing, and open/closed boundaries.

The complete segregation model considers a plug-flow reactor equipped with an external evacuation system (Figure S2). The evacuation arrangement is designed so that fluid elements of different ages are not mixed until they leave the system.

The maximum mixedness model considers a plug-flow reactor equipped with an external feed system (Figure S2). The fluid particles of zero age are instantaneously mixed (as they enter the system) with fluid particles of different ages introduced upstream.

A brief description of some of the most widely held flow models is presented in Table S5.

5.6. Guidelines for Building Models for Non-Ideal Flow and Mixing Systems

Choosing a suitable model is not always an easy task. The fluids' (and sometimes particulate solids) flow and mixing behavior depend on many factors, such as the physicochemical properties (e.g., density, viscosity), the vessel geometry, the flow rate, and the size of the analyzed system. The model selection process becomes even more complex when one or more chemical reactions occur [68]. Bodner et al. proposed 10 simple rules for tackling mathematical modeling in general [69]. A model can never be completely precise or completely imprecise. When proposing a model, it is critical to stay goal-oriented, gather all pertinent data, identify crucial and insignificant details, and avoid oversimplifying or overcomplicating the model. Fogler [62] suggests some specific guidelines to follow when developing models for non-ideal flowing systems: (i) the model goal should be "calculated" as RTD data + model + kinetics = prediction; (ii) the processes that occur in the non-ideal system must be described physically, chemically, and mathematically with reasonable accuracy; (iii) the model must be mathematically accessible; and (iv) the model should not have more than two variable parameters, except the complex systems, were more independent parameters can be identified. The last recommendation is made because an equation with more than two variables can easily fit experimental data, reducing the modeling step to curve fitting.

6. Practical Application: RTD Diagnosis of Photochemical Reactor

6.1. Problem Statement

The design of photoreactors, either homogeneous (photochemical) or heterogeneous (photocatalytic), is slightly more complicated than the design of conventional reactors [70,71]. In addition to the factors analyzed for the design of conventional chemical reactors, the design of photochemical reactors must take into account several specific factors: the radiation emission, absorption, scattering, and transmission; the radiation energy contribution to thermal balance; the photon balance and the kinetic effects of the absorbed radiation [72–74]. Great efforts have been made to develop a standard methodology for photoreactors model-

ing, design, and scale-up [71,75], and a few distinct classes of photoreactors were acknowl-edged [75–79].

Among all types of photoreactors, annular photochemical reactors are the most frequently used to perform photochemically enhanced advanced oxidation processes [80,81]. The reactors in this category consist of a vessel in which one or more UV lamps are located in various positions, usually parallel with the vessel axis of symmetry, to irradiate the reaction mass as uniformly as possible. Adequate materials (e.g., quartz), having good UV transparency and mechanical resistance, are used to protect the lamps.

There are two crucial aspects to consider while designing an annular photochemical reactor: the homogeneity of the radiation field and the reactor hydrodynamics [82–84]. The number and arrangement of the lamps inside the reactor equally affect the radiation intensity [80] and the mass reaction hydrodynamics [85–87]. In a real-life reactor, from practical considerations, the number of lamps is quite limited, from 1 up to 7, and rarely 10 [88]. The arrangement follows the corresponding geometrical shape (e.g., equilateral triangle, square, pentagon, hexagon, and so on) or circularly, with or without a central lamp, to produce a uniform irradiation field [86]. In computer-simulated analysis, the number of lamps can grow significantly from 1 to 63 [84].

Additional issues that significantly affect the flow pattern inside the reactor, besides the lamp's number and arrangement, are the inlet/outlet geometry and relative position towards the reactor's axe of symmetry [87,89–92]. Several technical solutions exist regarding the inlet/outlet position and geometry [83]. The most common positions are: (i) parallel with the central axis of symmetry, through the reactor lid; (ii) sideways feeding, perpendicular to the axe of symmetry, through the reactor wall); and (iii) tangential on the reactor wall, through the lid or reactor wall. Recent studies [83,89] show that the tangential positioning of inlet/outlet pipes promotes a specific helical movement of the fluid around the irradiation source, increasing reactor performance due to a more effective UV exposure.

The photochemical reactor (PHR) under study is equipped with four UV-C lamps protected with quartz sleeves and designed to function horizontally, vertically, or at various inclination angles. Axial or radial (wall tangent) inlets and outlets may be used for filling and/or emptying the reactor.

This experimental study aimed to determine the optimal working position and most practical location for inlet/outlet pipes using the RTD approach. Obtaining data on the degree of mixing and fluid flow was another objective.

6.2. Materials and Methods

6.2.1. Tracer Method

The insertion of the tracer was done using the pulse procedure, as described in Section 3.2. A sodium chloride solution of 20% mass concentration was used as a tracer, and the response was collected with a WTW conductivity meter Cond 315i.

6.2.2. Reactor Geometry and Working Position

The PHR under study belongs to the tubular reactors category, with 1.6. length/diameter ratio (L/D). The quartz sleeves' outer diameter is 45 mm.

The PHR was designed to be operated horizontally, vertically, or at various inclination angles. For this particular study, three operating positions were considered: horizontal, vertical, and inclined at a 45° angle (Figure 10).

6.2.3. UV-Lamp Arrangement

The PHR is equipped with four UV-C lamps protected with quartz sleeves with an inner diameter of 39 mm. Three lamps are placed as an equilateral triangle, while the fourth is positioned centrally, having the same axe of symmetry as the reactor. With this arrangement, the lamps can be used in three configurations, of one, three, or four lamps simultaneously, as presented in Figure 11A. In this manner, the intensity of the irradiation



can be modulated when necessary. Moreover, the lamps can be exchanged and/or replaced without stopping the process (Figure 11B) [93].

Figure 10. Working reactor positions: (A): horizontal, (B): inclined (45°), and (C): vertical.



Figure 11. The UV-C lamps configuration schemes: (**A**): one lamp on, three lamps on, four lamps on; (**B**): four lamps on at the actual reactor.

6.2.4. Inlet/Outlet Type

This study used two different arrangements of inlet and outlet pipes: (i) axial, parallel with the central axis of symmetry of the PHR, and (ii) radial, tangent to the reactor wall (Figure 12). The inner diameter of inlet and outlet pipes, either axial or radial, is 12 mm.



Figure 12. (A): Axial (through the lids); (B): radial (through the walls) inlet/outlet.

6.3. Results and Discussions

6.3.1. Characteristics and Profiles of RTD Functions

The C-Curve

The C-curve displays the time evolution of the tracer concentration and provides the raw data for RTD investigation. The contour of the C-curve is always identical to the contour of the E-curve. Therefore, the transformations generated by changing the inlet and outlet positions are evident by looking at the C-curves alone, without any supplementary data analysis (Figure 13A,B). The occurrence of flow defects, short-circuits or channeling and dead or stagnant zones, are indicated by the sharp peaks at the very beginning of the curves (short-circuits) and of the long tails at the end of the curves (dead zones). Although the shapes of the curves are different (Figure 13A,B), the presence of both short-circuits and dead zones is unquestionable, regardless of the reactor operating position and/or the inlet/outlet type.



Figure 13. The C-curves: (**A**): axial inlet/outlet, (**B**): radial inlet/outlet, with reactor operated horizontally, inclined, and vertically.

The RTD Functions

The plug flow reactor is the ideal reactor commonly used for modelling an annular photochemical reactor [94–96]. The representative RTD functions from Figure 14 for PHR operated horizontally clearly indicate a noticeable deviation from the ideal plug flow model for both inlet/outlet types.



Figure 14. Profiles of RTD functions, axial vs. radial inlet/outlet, reactor operated horizontally. (A): E-curve; (B): F-curve; (C): I-curve; (D): λ -curve.

In order to highlight the influence of reactor working position and inlet/outlet type on the fluid flow through the PHR, two of the main RTD functions, residence time distribution (E) and cumulative residence time distribution (F) were plotted as functions of time (t) and/or as a function of dimensionless time (θ) and further analyzed. In addition, the

first and second moments of residence time distribution, usually known as the mean residence time, respectively, the variance, were determined for each working position and inlet/outlet type.

Space Time, First and Second Central Moments

The space-time value, as determined by Equation (1), was 270.3 s for all experiments conducted. The values of the first two central moments and the corresponding standard deviation for all six reactor configurations considered in the current study are presented in Table 3.

Reactor Position	Inlet/Outlet Type	ī	σ^2	σ
Horizontal	axial	226.94	36,606.30	191.33
	radial	226.56	37,705.47	194.18
Inclined (45°)	axial	274.06	57,750.94	240.31
	radial	296.45	42,300.83	205.67
Vertical	axial	290.66	60,084.68	245.12
	radial	311.65	43,635.04	208.89

Table 3. The first and second central moments of residence time distribution.

By comparing the values of space-time and mean residence time, which are equal for ideal flowing systems [62,97], some information related to the presence of short circuits or dead zones can be attained. If $\bar{t} < t_s$, a part of the fluid rapidly leaves the vessel (short-circuit or channeling). If $\bar{t} > t_s$, a part of the fluid stays longer inside the reactor (dead zones or stagnancies). However, the use of only the first moment can be misleading since its value can be significantly affected by measurement errors: (i) the presence of internal dead zones (the reactor is not entirely filled with fluid); (ii) the density differences between the tracer and the analyzed fluid; (iii) the tracer tendency to adsorb on the surfaces inside the vessel [44]. Therefore, as it indicates the spread of the distribution around the mean, it is recommended that the values of the second moment go along with those of the first moment [62]. For example, the \bar{t}_{ax} value for the inclined-operated reactor (Table 3) is very close to the actual space-time (274.06 vs. 270.3 s). However, the flow pattern is far from the ideal plug flow, as revealed by the RTD functions (Figure 15) and confirmed by the corresponding σ_{ax}^2 value (Table 3).

The E-Curve Analysis

The analysis of E-curve allows a relative quantification of the residence time distribution [62]. The E-curve for the axial inlet (Figure 15A) shows that 17.8% of the material has spent 30.8 or less than 30.8 s in the reactor. Note that no signal was recorded in the case of radial inlet during this time. The area under the second peak corresponds to approximately 10% of the material that has resided in the reactor between 30.8 and 70.79 s. The remaining 72.27% of the materials (the area under the tail) reside somewhere between 70.79 and 772 s. The area under the E-curve for the radial inlet (Figure 15B) can also be divided into three regions: 7.98%, 28.24%, and 63.78%.

Even if the shape of the E-curves is very different at the beginning, the presence of short-circuits manifests for both reactor configurations, and after 120 s (Figure 16), the amount of material that spent 2 or less than 2 min in the reactor is very similar (around 38%).

The F-Curve Analysis

The shape of F-curve provides a series of information related to the fluid flow through the reactor. It depends on the relative time required by various elements (portions) of fluid to flow through the vessel (the distribution of residence times). Therefore, the term "mixing" is not related to chemical homogeneity but to the age distribution of material in the reactor and outgoing stream [13]. The experimental F-curve profile indicates complete mixing for all six reactor configurations. The degree of departure from perfect mixing is given by the area between the experimental and theoretical curves (Figure 17A,B). If the area is above the theoretical curve, it indicates the presence of a stagnant region (dead zone). The area under the F-curve between $\theta = 0$ and $\theta = 1$ is called "hold-back" (H)—the surface A₁ in Figure 18. The sum of A₁ and A₂ areas from Figure 18 represents the degree of departure from the ideal mixing. The critical values of H are 0 for piston flow, 1/e for complete mixing, and approximately 1 if most of the vessel space is dead zone [13]. A high hold-back indicates that a significant part of the vessel volume is occupied by material already reacted. At the same time, the reactant passes rapidly through the reactor by a short-circuit route [13].





Figure 15. E-curve analysis, reactor operated horizontally. (A): axial inlet/outlet, (B): radial inlet/outlet.



Figure 16. E-curve analysis, reactor operated horizontally: axial vs. radial inlet/outlet—short circuits evidence.



Figure 17. F-curve analysis, reactor operated horizontally. (A): axial inlet/outlet, (B): radial inlet/outlet.



Figure 18. F-curve analysis, reactor operated horizontally, axial vs. radial inlet/outlet: hold-back.

6.3.2. Influence of Reactor Position

The influence of the reactor operating position is minor, as revealed by the profiles of the E-curves and confirmed by the increasing values of the mean residence time. The differences produced seem more significant for the axial inlet/outlet (Figure 19A–C) than for the radial inlet/outlet (Figure 19D–F). These alterations are related to the density difference between the sodium chloride tracer and the water inside the reactor. Higher density generates negative buoyancy; changing the reactor position increases the relative height to be climbed by the tracer (Figure 10) and is shown to cause the delay, as indicated by the values of the mean residence time.



Figure 19. E-curve analysis on the short-circuit region for different reactor positions: (**A**): horizontal reactor with axial inlet; (**B**): 45° inclined reactor with axial inlet; (**C**): vertical reactor with axial inlet; (**D**): horizontal reactor with radial inlet; (**E**): 45° inclined reactor with radial inlet; (**F**): vertical reactor with radial inlet.

Further analysis of the E-curves on the short-circuiting corresponding peak shows that the amount of fluid that flows through a short circuit slightly decreases with the increase of the inclination angle (e.g., 17.83% < 16.87% < 16.17% for axial inlet/outlet). The alterations are small enough to confirm the hypothesis that the density differences cause them and can be easily associated with the inequalities among the relative height.

6.3.3. Influence of Inlet/Outlet Type

Regardless of the reactor operating position, changing the inlet/outlet type significantly affects the flow pattern inside the reactor. The relative length to be traveled by the tracer through the reactor is longer when the jet is following a spiral path from the inlet to the outlet compared to when the jet is following a straight route from the inlet to the outlet, and consequently generates the differences between the mean residence times on axial vs. radial inlet/outlet (Table 3). Regardless of the inlet/outlet type, the quartz sleeves act like baffles. However, for the radial inlet, the baffles are oriented almost perpendicularly to the flow, while for the axial inlet, the baffles are nearly parallel with it. Since the flow pattern differs, the position of dead zones and short-circuits differs. The radial inlet/outlet stimulates a specific helical movement of the fluid that may shift the dead zone in the central area of the reactor, surrounded by the four quartz sleeves. As for the axial inlet/outlet, the location of dead zones is typically at the opposite "corners" of the reactor.

6.3.4. Practical Decisions following RTD Diagnosis

Due to the constructive particularities of the annular photochemical reactors, flow defects such as short circuits and dead zones cannot be avoided by changing the reactor operating position or the inlet/outlet type. It was found that the reactor position has less or no significant influence on the flow pattern, while the inlet/outlet position is critical. Changing the reactor position modifies the relative height to be climbed by the tracer while changing the inlet/outlet type modifies the pathway and the relative length to be traveled by the tracer. The radial inlet/outlet stimulates a specific helical movement of the fluid around the quartz sleeves.

Based on the current investigation results and recent literature reports [87,98], the following design and operation recommendations were made:

- (i) The PHR should be equipped with a recirculation pump that can be used to adjust the UV exposure time and to improve the mixing of chemical species;
- (ii) The radial inlet/outlet is recommended for several reasons: (1) it promotes the development of a helical motion of the fluid around the UV lamps, increasing the relative length to be traveled by the fluid inside the reactor and consequently increasing the UV exposure time; (2) the location of dead-zones tends to be shifted from the reactor's "corners" to its central region, among the quartz sleeves, which increase both the exposure time and the intensity of the irradiation field for the fluid trapped in the stagnant region; (3) due to the flow orientation, the quartz sleeves act like baffles, improving the mixing inside the reactor;
- (iii) The reactor operating position should be horizontal since it does not significantly influence the UV-C exposure time or irradiation intensity. However, when specific reagents are used, the reactor position may be adjusted to consider their buoyancy;
- (iv) To diminish the effect of short-circuiting or channeling, it is recommended to use lower flow rates, possibly in the region of semi-turbulent jets.

7. Conclusions

Residence time distribution is a versatile method that can characterize vessel hydrodynamics to reveal design flows (e.g., dead zones, stagnancies, by-passes, preferential flows) and estimate the mixing degree. The statistical processing of fluid element residence times using typical RTD functions, combined with mathematical modeling of ideal and non-ideal flow and reaction kinetics, allows conversion and yield predictions. That makes RTD analysis an indispensable tool for chemical reactor design. Moreover, the method has several applications in other fields (e.g., anatomy, geology, metallurgy). An RTD analysis was performed on a photochemical reactor to prove the method's utility. The analysis revealed the presence of short-circuits and dead zones. A series of design decisions related to the reactor's inlet/outlet orientation and the reactor's working position were made after the RTD diagnosis. These modifications affect the reactor's hydrodynamic, which directly influences the photo-chemical process efficiency due to the: (i) increase of the residence time (which means prolonging the exposure to UV irradiation); (ii) mixing intensification (that improves the catalyst dispersion).

Supplementary Materials: The supporting information can be downloaded at: https://www.mdpi. com/article/10.3390/pr11123420/s1. References [99–152] are cited in the supplementary materials.

Author Contributions: Conceptualization, G.D.S. and M.T.N.; methodology, E.N.D.; validation, G.D.S. and A.C.P.; formal analysis, M.T.N.; writing—original draft preparation, M.T.N., E.N.D. and G.D.S.; writing—review and editing, E.N.D. and A.C.P.; visualization, A.C.P.; supervision, M.T.N.; funding acquisition, E.N.D. All authors have read and agreed to the published version of the manuscript.

Funding: This research was funded by "Program 4: Fundamental and Frontier Research—Exploratory Research Projects" financed by UEFISCDI (project no. PCE 58/2021).

Data Availability Statement: Data is available upon request.

Conflicts of Interest: The authors declare no conflict of interest.

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