



RADIOTRACER RESIDENCE TIME DISTRIBUTION METHOD IN DIAGNOSING INDUSTRIAL PROCESSING UNITS: CASE STUDIES

J. Thereska, E. Plasari***

** Institute of Applied Nuclear Physics, Tirana, Albania*

*** Ecole Nationale Supérieure des Industries Chimiques -INPL, Nancy, France*

*[*thereska@gmail.com](mailto:thereska@gmail.com)*

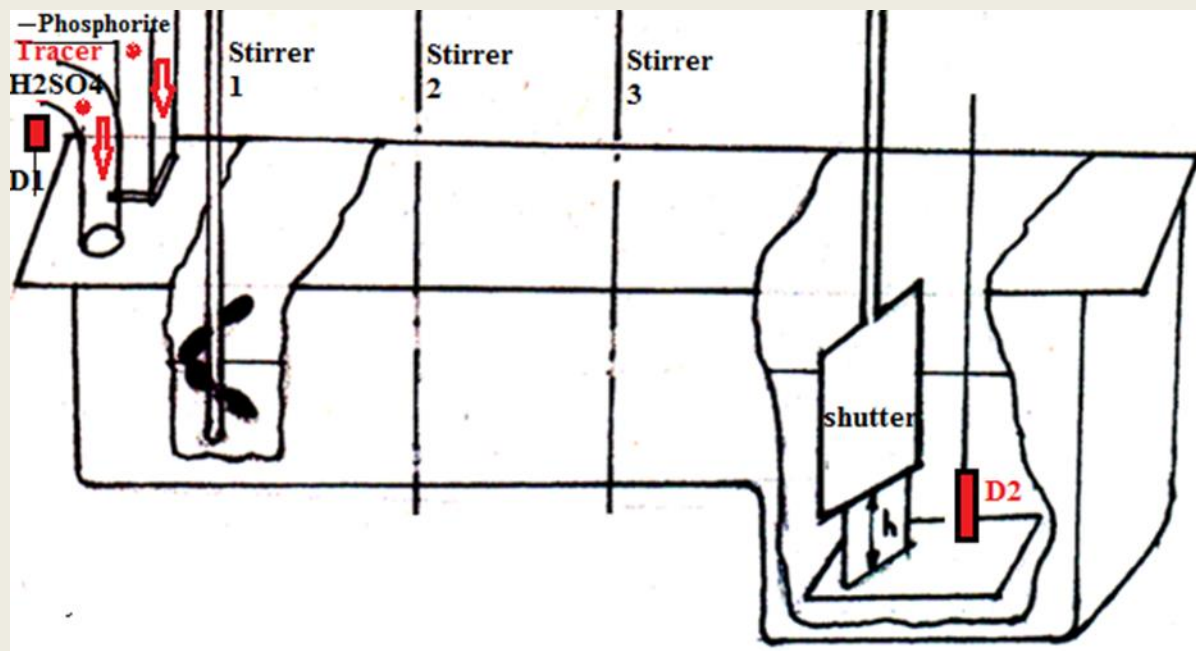
In spite of its „old age“, the residence time distribution (RTD) modelling is still the subject of many publications in international journals concerning general or practical aspects of the RTD for troubleshooting and diagnosing industrial processing units.

Radiotracers are unique in measuring accurate RTD experimental curve in harsh and opaque industrial field conditions; especially online without shutting down or disturbing the process.

The paper gives examples showing how the radiotracer RTD measurements can analyse the operation of some industrial processing units, to eliminate troubles and to optimise the performance of processes.

Case study 1: Flow Characteristics in a Superphosphate Production Chamber

Problem: *Three-stirrer chamber in which the ground phosphorite ore is mixed with sulphuric acid H_2SO_4 plays an important role in the whole process of production of superphosphate. Two chambers were employed, one with three stirrers, and the other with four stirrers. The quality of produced superphosphate (in particular the content of P_2O_5) was rather poor, in both chambers. Figure gives the design of the continuous mixing chamber (with three stirrers).*



Case study 1: Flow Characteristics in a Superphosphate Production Chamber

Mixing chambers are chemical reactors in forms of parallelepiped, equipped with three and four mechanical stirrers placed one after the other in line of flow, with no baffles between them, with following sizes:

- *Volume of three stirrers chamber was 1.9 m³*
- *(2.5 m long, 0.70 m wide and 1.1 m high),*
- *Volume of four stirrers chamber 2.5 m³*
- *(3.2 m long, 0.70 in wide and 1.1 m high).*
- *Filling coefficient with the pulp is about 0.3-0.4.*
- *Speed of stirrer is 3 - 4 rps.*
- *Phosphorite ore flow rate: 16 t/h*
- *Sulphuric acid (68%) flow rate: 14 t/h*

Ground phosphorite and sulphuric acid enter at one end of the chamber through separate pipes, pass through the three-four stirrers and are discharged at the other end of the reactor in the direction of the maturing chamber through a shutter.

EXPERIMENTS

Case study 1: Flow Characteristics in a Superphosphate Production Chamber

^{113m}In (from Sn-In- ^{113m}In generator) radiotracer was used for tracing phosphorite ore and sulphuric acid, in order to investigate the hydrodynamics of both of them separately within the mixing chambers.

For sulphuric acid, ^{113m}In in 0.01M aqueous solutions of EDTA has been employed, as good tracer of liquid phase.

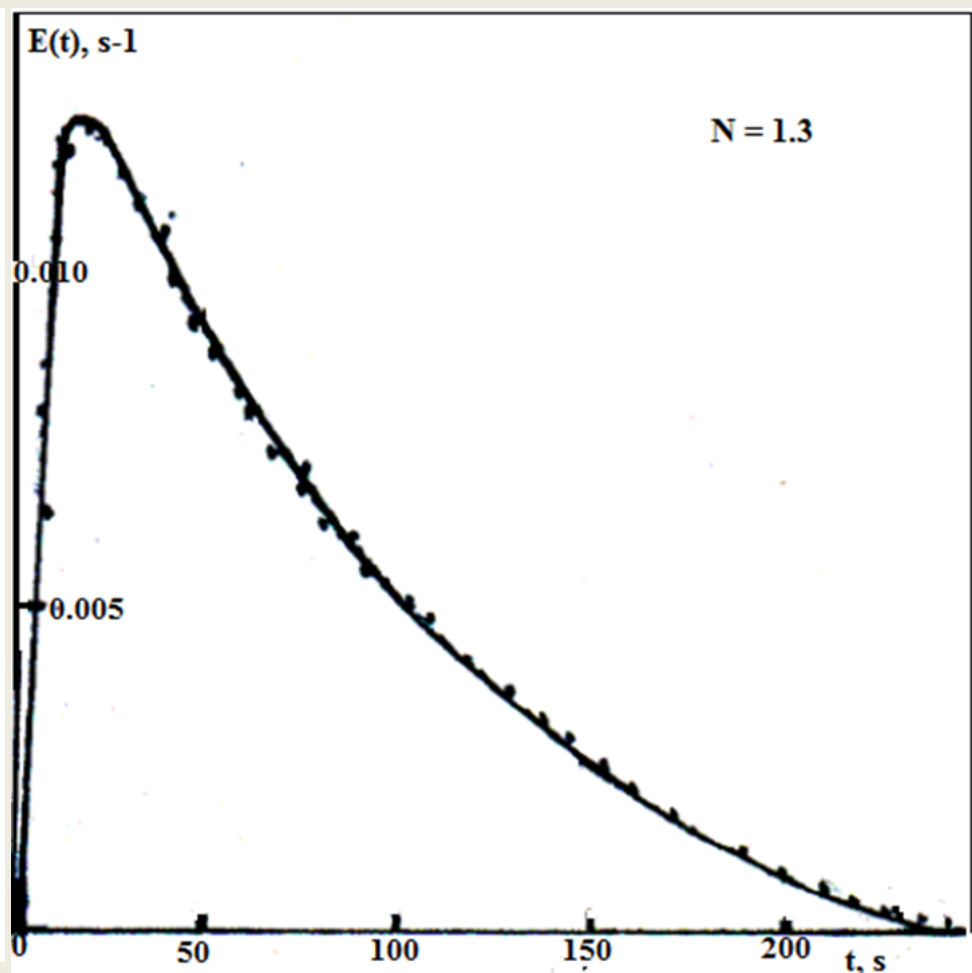
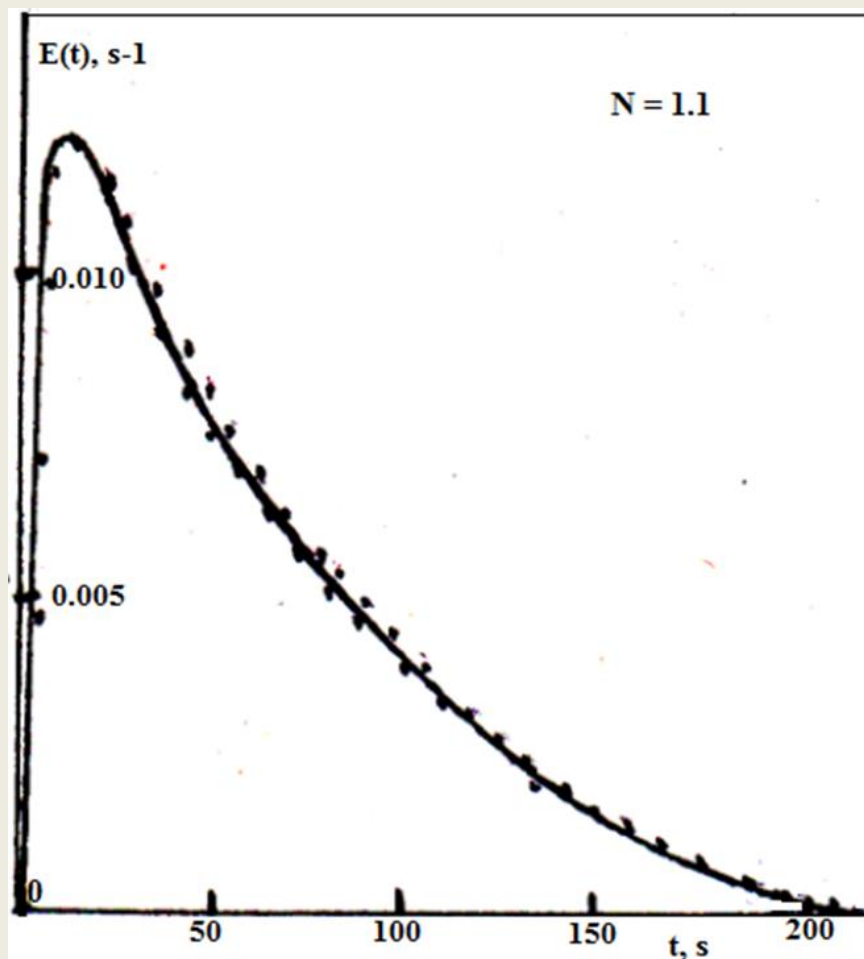
For phosphorite ore, ^{113m}In as $^{113m}\text{InCl}_3$ solution was mixed up with ore, as good tracer of solid phase.

The activity of ^{113m}In was 40 MBq in each test, there were several tests performed for shutter opening : $h = 35$ cm.

Results

Case study 1: Flow Characteristics in a Superphosphate Production Chamber

Experimental RTD curves fit well with the model of tanks in series. Figures show experimental and model RTD curves for chambers with three (left) and four stirrers (right), for acid phase.



Discussion of results

The results of the tracer experiments were:

Mixing chambers with three stirrers (N=3):

- Mean Residence Time (MRT) for sulphuric acid = 41 s
- MRT for phosphorite ore = 49 s
- Number of tanks in series (for both phases) : $N_{\text{model}} = 1.1$

Mixing chambers with four stirrers (N = 4):

- MRT for sulphuric acid = 46 s
- MRT for phosphorite ore = 56 s
- Number of tanks in series (for both phases) : $N_{\text{model}} = 1.3$

Three stirrers mixing chamber, behaves like 1.1 ideal mixer, that means the real mixing rate is: $1.1/3 = 0.23 = 37\%$ of three ideal mixers.

Four stirrers mixing chamber, behaves like 1.3 ideal mixer, that means the real mixing rate is: $1.3/4 = 0.33 = 33\%$ of four ideal mixers.

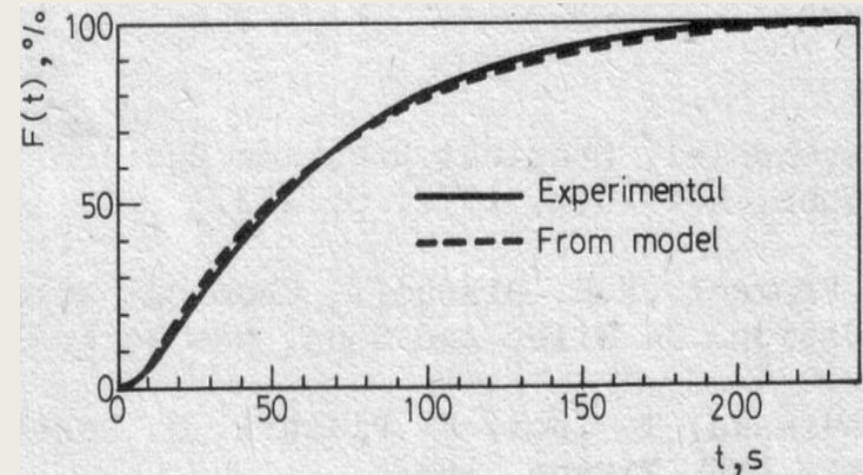
Conclusion

Three and four stirrers mixing chambers, are not performing well, the real mixing is one third of the stirrers they have.

The almost ideal mixer model of both chambers is not appropriate, there is not seen the role of individual stirrers compartments. This means that the residence time of various fractions of the pulp (phosphorite – acid mixture) within the whole chamber is different, from few seconds till several minutes, creating problems for homogenisation and reacting.

The integral residence time function, showed:
 About 30% of the pulp remains less than 30 sec in the mixing chamber.

This time is insufficient for satisfactory homogenization and reaction of the phosphorite with sulphuric acid.



Recommendations

Baffles were recommended to be installed between mechanical stirrers to individualize the effect of each stirrer.

It increases the MRT and numbers N of tanks in series, thus improving the micro and macro homogenization of the pulp and as consequence increase the reaction time that means the quality of the product.



Case study 2: Flow dynamics in laboratory and pilot-plant molecular sieves columns for dehydrating organic liquids

Problem:

Organic liquid drying in adsorption molecular sieve column is practiced extensively in industry.

The designing of small mobile units used for dehydrating of a large variety of organic liquids was planned. For that it was necessary to know the dynamic characteristics of the fluid flow through adsorption columns of various diameters filled with spherical particles of molecular sieves.

Goal: to investigate the dependence of the Peclet number upon the ratio of column diameter-to-particle diameter.

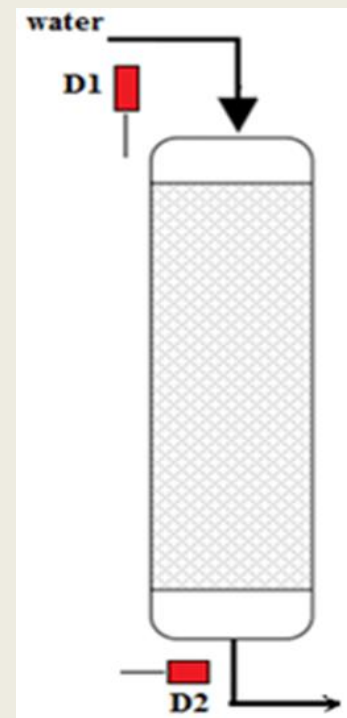
Literature sources give contradictory data about the dependence of the Peclet number upon the particle Reynolds number in packed beds.

EXPERIMENTS

Two laboratory columns having inner diameters of 12.5 and 25.5 mm, respectively, and a pilot-plant column of an inner diameter of 55.5 mm filled with commercial Merck molecular sieve spheres of 2 mm diameter were studied using radiotracers. In all cases the height of the packed bed was 300 mm.

Figure shows a typical column.

Experiments with ^{113m}In - EDTA radiotracer, was performed to investigate fluid flow dynamics in columns of three different diameters. **0.5 mL with an activity of 20 MBq** were injected as the tracer at the entrance of the columns. The duration of injection was shorter than 1 s and the volume of liquid injected was smaller than 1% of the volume of the liquid phase in the columns. Thus entry impulse was considered as a Dirac pulse. The experimental RTD curve was obtained with detector D2 at the outlet of columns.



Case study 2: Flow dynamics in laboratory and pilot-plant molecular sieves columns for dehydrating organic liquids

Aqueous solutions were used as liquid phase in experiments instead of organic liquids, because, according to Dimensional Analysis, any liquid can be used to investigate flow dynamics.

In all experiments flow velocities were typically in the range recommended by the molecular sieves manufacturers, from 2 up to 10 cm/min.

Results and discussion

Piston (or axial) dispersion model is the simplest mathematical description of the flow system in which both convection and diffusion are important.

Piston-dispersion model depends on a single dimensionless parameter known as the Peclet number,

$$\mathbf{Pe} = \mathbf{v} \mathbf{L}/\mathbf{D},$$

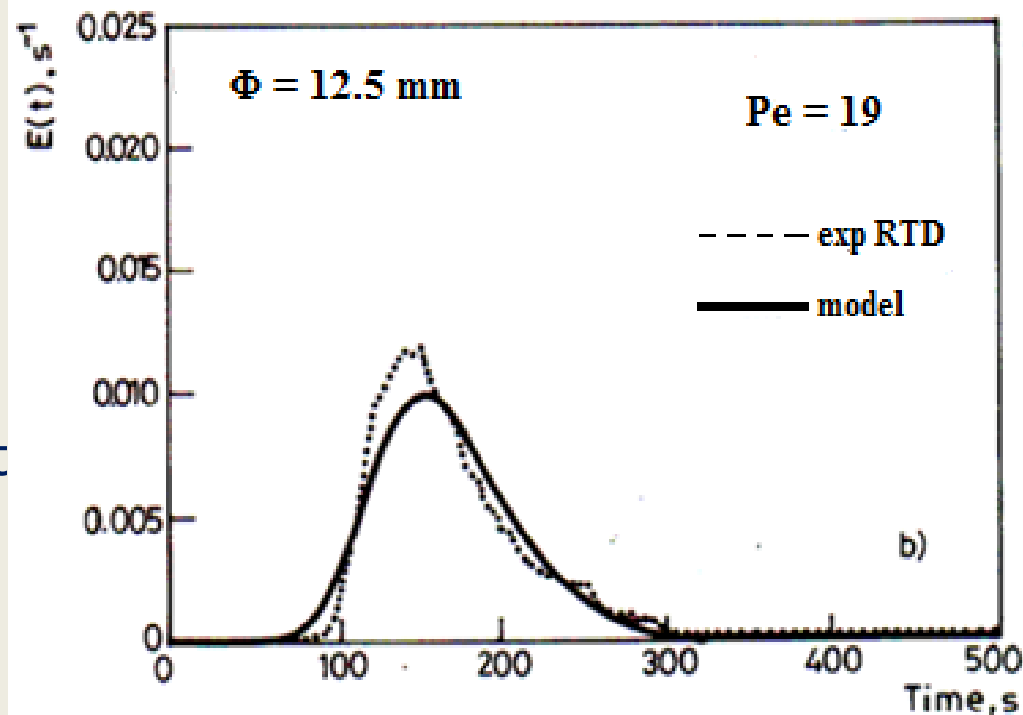
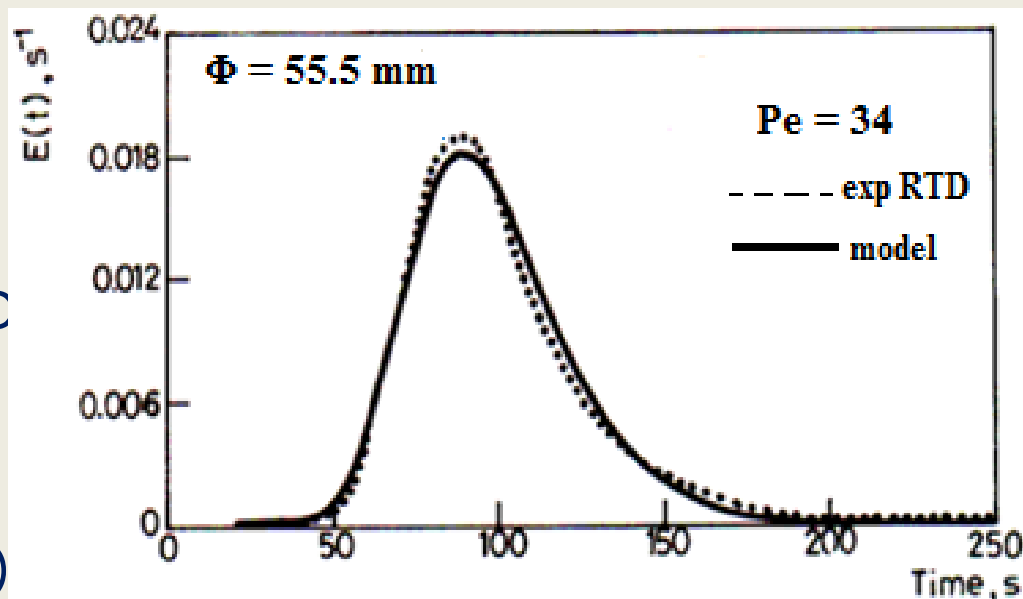
where v is the flow velocity, L the total length of the column and D the axial dispersion coefficient. For **$Pe > 30$** the flow is considered piston, which is the best model for higher efficiency of molecular sieves.

Case study 2: Flow dynamics in laboratory and pilot-plant molecular sieves columns for dehydrating organic liquids

High degrees of fitting of experimental RTD curves with RTD model were obtained for two larger columns with inner diameters of 55.5 and 25.5 mm (*almost piston model: $Pe = 34$*)

For smallest column with an inner diameter of 12.5 mm, fitting was lower and experimental RTD curves showed irregularities (**$Pe = 19$**)

For illustration, the best and worst fitting cases of experimental and theoretical RTD curves are represented in Figures.



CONCLUSION

The dependence of Peclet number Pe upon column diameter-to-particle diameter ratio was found and rather bad fitting for ratios smaller than 10 were observed.

Radiotracer experiment pointed out that the column of 12.5 mm diameter has more irregularities and experimental RTD curve does not fit well with Piston model, which is the required model for molecular sieves columns.

Consequently, it was recommend to avoid the use of columns of small tube diameter-to particle diameter ratios (smaller than 10) for molecular sieves for drying of organic liquids.