RAPIC PROJECT: TOWARD COMPETITIVE HEAT-EXCHANGER/REACTORS

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INTRODUCTION

Process intensification (PI) methodology, introduced in the 1980s, was recently defined\(^1\) as “any chemical engineering development that leads to a substantially smaller, safer, cleaner and more energy-efficient technology”. Among the numerous options to intensify a process, the transposition from a batch reactor to a continuous plug flow reactor is a good alternative when the selectivity and the thermal exchange are an issue: this case is illustrated in this study through the concept of multifunctional heat exchanger/reactor\(^2,3\). Many benefits are expected such as waste reduction, energy and raw materials savings, yield and selectivity increase, and cost reduction. Despite these benefits, to date only a few intensified processes are in industrial operation. Nevertheless, typical academic studies\(^3,9\) of multifunctional heat exchanger–reactors in the literature report the large potential of this technology. The relatively slow transfer to industry is presumably linked to many factors, among them the lack of successful industrial demonstrations.

The RAPIC project focuses on the implementation in industry of intensified continuous unit and aims to be polyvalent and flexible (batch processes as well), robust (not too sensitive to varying conditions) and low-cost (crucial for process industrialization). The present paper details this research project. It presents the main results on specification and definition of the reaction channel, and on design, manufacture and tests of the two final pilot-scale prototypes.

RAPIC PROJECT DESCRIPTION

Main objectives

The RAPIC R&D project started in December 2007 and is funded for 42 months by the French National Research Agency (ANR) with a financial support from Axelera (chemistry environment competitive cluster). It aims at developing an innovative but low-cost component in the 10 kg/hour range. It is original in that it must not only comply with the implementation constraints of exothermal reactions, but also be as close as possible to mature technologies in the heat-exchanger sector by adapting them to reactive media and by respecting the cost imperatives imposed by the market. In principle, the plate reactor is composed of reaction plates sandwiched between cooling plates. Reaction plates are brazed in order to ensure good thermal contact. Thus, using enhanced plate-fin geometry available on the market, the RAPIC plate reactor has very high heat-transfer ability to ensure excellent thermal control of the reaction.

We plan to use Hot Isostatic Pressing (HIP) fabrication technology for the reaction channels so that we can produce modules with different materials (copper, stainless steel) around the reaction channels for efficient management of exothermal effects\(^10\). The complex geometries needed for process intensification are obtained by assembling machined metallic plates or bent tubes embedded in sintered metal powders. Large industrial HIP facilities available today can produce low-cost components with enhanced thermal performance.

Project organization

Over its three years, the RAPIC project dealt with design from the local to the global scale and experimentation from elementary mock-ups to pilot-scale systems. Five main tasks are involved (\(T_0=\)December 2007)

1. Specification of the plate reactor by the end user in terms of performance level and acceptable price (\(T_0 \rightarrow T_0+3\) months)
2. Definition of the reaction channel, based on numerical simulation and analytical tests (\(T_0+3 \rightarrow T_0+18\) months)
3. Manufacture (by traditional or innovative techniques) and testing (thermo-hydraulic and micromixing) of plate modules designed for reaction ($T_o + 3$ months $\Rightarrow T_o + 18$ months)

4. Manufacture and test of two prototypes (1-10 l h$^{-1}$); several representative exothermal reactions will be tested ($T_o + 18$ months $\Rightarrow T_o + 42$ months)

5. Market assessment and dissemination ($T_o + 33$ months $\Rightarrow T_o + 42$ months)

**Partnership**

The RAPIC project consortium is highly complementary (figure 1). Indeed, one end-user (Rhodia Chemicals) and one heat-exchanger manufacturer (Fives Cryo) are involved to ensure the industrial view of the work. In addition, French laboratories active in basic research on process engineering (LGC) and thermal-hydraulic engineering (LTN) are involved. The Atomic Energy Commission (CEA/LITEN) is handling project coordination, component design, and manufacture of the reaction plates using HIP technology.

![Figure 1: Role of partners in the RAPIC project.](image)

The involvement in RAPIC of a leading chemical group (Rhodia), as well as a parts manufacturer (Fives Cryo) wishing to diversify its industrial activities, constitutes a driving force and ensures the future industrial application of the work.

**SPECIFICATIONS**

The specification, in terms of the chemical reaction target, has been defined by Rhodia. The reaction is a homogeneous exothermic reaction in liquid phase, with an adiabatic temperature rise of 200°C. It is carried out at 100°C and under atmospheric pressure due to the design features of the batch reactor. However, the pressure is not a sensitive parameter to run the reaction. This is a fast reaction, with a residence time of 2-3 minutes. The reactor must be as isothermal as possible, with a maximum temperature rise of 10°C. *Figure 2* gives the required reactor volume as a function of volumetric heat-transfer performance and coolant temperature (the chosen coolant throughput allows overcoming any heat exchange limitation in the "coolant side").

![Figure 2: Specified operating conditions.](image)
Three main sizes have been defined as production targets for the project:

- Elementary scale: 1 L·h⁻¹
- Laboratory scale: 10 L·h⁻¹
- Pilot scale: from hundreds of L·h⁻¹ up to few m³·h⁻¹

Only laboratory-scale units will be constructed and tested within the RAPIC project. However, extrapolation to the pilot scale is included in the choice of concept and geometry.

A detailed economic analysis made by Rhodia for an industrial chemical production of 6 kTs·year⁻¹ is based on a methodology for transferring batch to continuous reaction. For this estimate, the reactor investment assumes a price equal to that of a 27 m³ semi-batch technology. In addition, the analysis also includes modification of the process environment, such as upstream and downstream storage tanks (larger for batch process), injection simplification, and data measurement and acquisition systems. Using these assumptions, the overall investment cost of a batch unit for 6 kT·y⁻¹ has been estimated. For a continuous process, in order to achieve the same investment cost, an acceptable price level for the RAPIC plate reactor has been set.

In term of manufacturing costs, we assume in a first time that the transposition from a batch process to a continuous process does not allow saving money: to meet the economical stakes, the investment cost is the key economical parameter to manage. However, this assumption on manufacturing costs is very pessimistic. Indeed, the following potential gains could be expected in a continuous configuration:

- Energy savings: less thermal losses due to the smaller volume.
- Raw materials savings: if selectivity issues are encountered in the batch process, higher yield for the continuous reaction step are expected. Moreover, the investment costs decrease (fewer throughputs to treat) for the following separation and for the effluent treatment steps.
- Decrease of fixed costs in terms of labour costs (less shifts to manage the process) and depreciation (proportional to the investment).

As a consequence, the acceptable price level set for the RAPIC plate reactor is the very lowest threshold and gives us a reasonable financial margin.

**FIRST PLATE REACTOR CONCEPT**

**Simplified Modeling**

Using simplified thermal-hydraulic modeling, a pre-sizing of a first plate reactor based on the specification has been made. This sizing is based on:

- The transfer of CEA’s know-how in nuclear fusion activity₁⁰ to the reaction part: integrating straight stainless steel tubes (8/10 mm) into a thermal conducting copper matrix (figure 3). The elementary plate size was determined by the availability of HIP vessels: 1.25 m long, 0.8 m wide.
- The use of standard straight and perforated fins (4 mm high, 0.15 mm thick) on the utility side in order to improve reactor coolant capacity.

![Figure 3: Schematic of the first design of RAPIC reaction plate (gray – stainless steel; orange – copper).](image)

Thus, in order to reach the predefined specifications, the full-size plate reactor has the following characteristics:

- 25 reaction plates (2 passes) in parallel, hence 50 plates surrounded by 51 utility plates with straight and perforated strip fins;
- Each plate includes 60 tubes (8/10 mm), with twisted tape inserts inside and external turning boxes;
• Plates are mainly copper, with stainless steel as the external skin (container). The final thickness of each reaction plate is around 20 mm.
• The final component has external dimensions 800 mm×1440 mm×1250 mm and volume 1.44 m³.

With this design, we reach a heat transfer capability of 580 kW·m⁻³·K⁻¹ and a pressure drop of 7 bars; the design thus meets both the required residence time and heat transfer goals. It is also a feasible technological solution.

Full prototype cost estimation
A precise cost estimate has been made for the above design:
• Utility plate using offset strip fins: 22% of the total cost
• Reaction plates: 78% of the total cost with 50% for materials supplying and machining

Therefore, this first design has an overall cost equal to the one of an equivalent batch technology for a 6 kT·year⁻¹ production. This cost could be significantly reduced by enhanced design, which could lead to more compact geometries, and by manufacturing technology improvements. In any case, however, the RAPIC project has a realistic basis.

Manufacture of the elementary mock-up
The objective of the first mock-up is to demonstrate that the elementary design criteria selected for RAPIC leads to a low cost reaction plate by the choice of a mature manufacturing process and a simple geometry. The mock-up size is around 320x130x25 mm, with two levels of tubes to increase compactness.

As shown in figure 4, the mock-up is made of straight stainless steel tubes inserted in a copper matrix. Internal stainless steel swirls are placed into the tubes to enhance heat transfer and mixing. Tubes are placed between copper plates grooved by milling. The distribution and turning over of the fluid is ensured by two machined stainless steel turning boxes.

Once the tubes are welded to turning boxes and inserted between copper, all parts are placed into a metallic container that is sealed and degassed under vacuum. Hot Isostatic Pressing (typically under 1000°C/1000 bar/1hour) is then applied to weld all materials (tubes to distribution boxes, tubes to copper, copper to the container) by diffusion bonding. A final machining is performed on top and lower surfaces to ensure the proper flatness.

Figure 4: Manufacturing steps for the first reaction plate mock up : a) tubes with internal swirls are placed between copper plates, b) turning and distribution boxes are welded to tube ends, c) mock up after HIP and final machining.

STRATEGY FOR REACTION CHANNEL OPTIMIZATION
So far, a feasible concept has been designed and manufactured that meets all the predetermined specifications, including the price. However, this design is based on the use of straight tubes with inserts, which is not globally optimal. Thus design work has been lead in order to develop a more efficient and up-scalable geometry¹. Some orientations, based on enhanced design, have been considered:
• modification of channel cross-section shape (circular, square, rectangular)
• channels structural geometries (two-dimensional waves, three-dimensional geometries, …)

Hence, the definition of an improved reaction plate pattern was based on a systematic multiscale analysis, using analytical, experimental or simulation tools (figure 5).
Five mock-up designs, involving different channels’ shapes and fabrication technologies have been considered. Their thermal performances and hydrodynamic behaviours have been characterized. It consists in experimental measurements of thermal transfer coefficients, mixing times, residence time distribution and pressure drops. They have been compared with the reference mock-up and ranked according to these technical characteristics and the fabrication feasibility. Three mock-ups have been eliminated because of the fabrication processes. They effectively led to either too expensive manufacturing costs or non-optimized design (low compactness and too thick plate). The thermo-hydraulic performances of the two last mock-ups have been experimentally characterized. The optimum mock-up (figure 6) has been chosen because of:

- its good performances in terms of heat transfer (ratio between the heat transfer coefficients of 1.5 at least and no limiting thermal conduction resistance);
- its high compactness;
- and its low cost and polyvalent (various channel shape) fabrication process.

![Figure 5](image.png)

**Figure 5:** Strategy for developing improved plate reactor geometry.

The wavy channel is of rectangular cross section (2 mm x 4 mm) and is machined with through-laser. Each plate is made of stainless steel of 2 mm thick (closing plates) or 4 mm thick (machined plate) and the overall size of the mock-up is 0.198x0.095x0.008 m. The volume of fluid in the wavy channel is of 17.6 mL.

**CHARACTERIZATION RESULTS**

The experimental characterizations concern the heat and mass transfer performances, the Residence Time Distribution (RTD) and the pressure drops.

**Pressure drops**

The general expression of pressure drop is written:

\[ \Delta P = 4 \cdot f \cdot \frac{L}{d_h} \cdot \rho \cdot \frac{u^2}{2} \]  \[1\]
with \( \rho \) the density of the fluid, \( u \) its velocity; \( L \) and \( d_h \) respectively the length and the hydraulic diameter of the channel and \( f \), the friction factor which is characteristic of the size and of the geometry of the channel. The evolution of the friction factor with the Reynolds number is shown in figure 7.

![Figure 7: Friction factor versus Reynolds number, experiments with water at 25°C.](image)

The evolution of the friction factor versus the Reynolds number is similar in the two mock-ups. However, it should be noticed that the characteristic size of each channel is different (8 and 2.7 mm) and so is the utilization field in terms of flow rate (respectively 100 and 10 L·h\(^{-1}\)). As a consequence, pressure drops will be different in each mock-up as shown in table 1.

<table>
<thead>
<tr>
<th>Flow rate (kg·h(^{-1}))</th>
<th>Fluid velocity (m·s(^{-1}))</th>
<th>Reynolds number</th>
<th>( \Delta P ) (mbar)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Straight tube with twisted insert</td>
<td>7 to 177</td>
<td>0.02 to 0.5</td>
<td>260 to 8510</td>
</tr>
<tr>
<td>Wavy channel</td>
<td>3.6 to 49</td>
<td>0.13 to 1.7</td>
<td>550 to 10460</td>
</tr>
</tbody>
</table>

*Table 1: Pressure drops measured in each mock-up.*

**Thermal characterization**

Thermal study is based on experiments which aim at cooling the fluid flowing in the reaction plate mock-up. The fluid is distilled water heated at about 85°C whereas the cooling fluid is raw water at about 10°C. For each experiment, the cooling flow rate is set to 6 m\(^3\)·h\(^{-1}\) whereas the process flow rate varies from 4 to 180 L·h\(^{-1}\). Four temperature sensors are set between the inlet and the outlet of the reaction plate and of the cooling system. To compare each geometry, the Nusselt number versus Reynolds number is plotted in figure 8.

![Figure 8: Nusselt number versus Reynolds number for each mock-up.](image)

The experimental results show that the heat transfer in the second mock-up (wavy channel) is until 3 times more efficient than in the straight tubes filled with a twisted insert. Moreover, the characteristic curve of this mock-up seems to stabilize for high Reynolds number. This may be due to the conductive
effects which start to limit the heat transfer between the cooling fluid and the other one around \( Re=5000 \).

**Residence Time Distribution (RTD)**

RTD experiments have been carried out in order to characterize the hydrodynamic behaviour in the two mock-ups. A conductimetric technique has been used. Two measuring probes are set up at the inlet and at the outlet of the mock-up. Each experiment has been performed at room temperature with a flow rate varying from 10 to 200 L·h\(^{-1}\) (Reynolds number ranges from 1000 to 11000). The experimental RTD has been modelled by an axial dispersion model characterized by the Peclet number:

\[
P_e = \frac{u \cdot L}{\varphi}\quad [2]
\]

\( u \), \( L \) and \( \varphi \) are respectively the flow velocity, the channel length and the dispersion coefficient. The higher the Peclet number is, the more the flow behaves like a plug flow, which is necessary to perform safely and efficiently chemical syntheses. Numerical results are given in table 2.

<table>
<thead>
<tr>
<th>Flow rate (L·h(^{-1}))</th>
<th>Reynolds number</th>
<th>Peclet number</th>
<th>Difference between theoretical and measured residence time (%)</th>
<th>Correlation coefficient</th>
</tr>
</thead>
<tbody>
<tr>
<td>Straight tube with twisted insert</td>
<td>20.8</td>
<td>1040</td>
<td>108</td>
<td>14</td>
</tr>
<tr>
<td>Wavy channel</td>
<td>9.4</td>
<td>880</td>
<td>45</td>
<td>19</td>
</tr>
<tr>
<td></td>
<td>44.0</td>
<td>4000</td>
<td>102</td>
<td>5</td>
</tr>
</tbody>
</table>

*Table 2: Peclet number and residence time in each mock-up.*

For each geometry the Peclet number is high enough, i.e. the residence time distributions are narrow enough to consider that the flow behaves like a quasi plug flow (\( Pe>50 \)). This characteristic is necessary to make sure that there are no dead zones or by-passes that could lead to decreases of the reactor performances (yields, selectivity,...).

**Mixing performances**

The iodide-iodate chemical probe method by using the adaptive procedure\(^8\) is used to investigate the mixing abilities of the two compact reactors: straight tubes with helical inserts, and zigzag channels which are presented respectively in figures 5 and 7. The purpose of the adaptive procedure consists to adapt the chemical method for characterizing the global mixing, by enlarging the measurement volume so as to capture and take into account all mixing scales. In the adaptive procedure, the kinetics of the reactions are adjusted in such a way as to impose the same reactive volume for different Reynolds numbers, leading to more relevant results for the segregation index \( X_S \).

Figure 11 represents the segregation index \( X_S \) obtained from the chemical method as a function of the Reynolds number in the three geometries: \( X_S \) gives qualitative information on the mixing, the lower \( X_S \), the better the mixing. It is clearly observed that \( X_S \) decreases exponentially with Reynolds number, exhibiting the mixing enhancement with Reynolds number. The selectivity obtained in the wavy channel is significantly better than in the straight tubes with helical inserts, implying better mixing quality. This result is in agreement with those obtained for the thermal characterisation presented in Figure 9.
Conclusion of preliminary studies

The first two mock-ups have been successfully designed, manufactured and characterized. The promising performances in the reaction channels led to experimental correlations for pressure drops and heat transfer coefficients versus Reynolds number.

<table>
<thead>
<tr>
<th>Straight tube with twisted insert</th>
<th>Pressure drop correlation</th>
<th>Heat transfer correlation</th>
</tr>
</thead>
<tbody>
<tr>
<td>f = 6.33 · Re(^{-0.52})</td>
<td>[3]</td>
<td>Nu = 0.11 · Re(^{0.57}) · Pr(^{0.33})</td>
</tr>
<tr>
<td>Wavy channel</td>
<td>f = 1.97 · Re(^{-0.38})</td>
<td>Nu = 0.16 · Re(^{0.66}) · Pr(^{0.33})</td>
</tr>
</tbody>
</table>

Table 3: Experimental correlations assessed in the elementary mock-ups.

The two laboratory-scale pilots were designed from these correlations to carry out two exothermic reactions. The first one is the oxidation of sodium thiosulfate with hydrogen peroxide. The results described in this article concern the implementation of this reaction in the wavy channel prototype. The other results are in process; these are the tasks of the RAPIC project last four months.

LABORATORY-SCALE PILOTS

In the 1\(^{st}\) step of the project, the reaction plates have been designed, manufactured and characterized. The results led to the selection of two reaction channel geometries and two fabrication processes. The 2\(^{nd}\) step objective was to integrate these results in a whole pilot including the coolant plates. It led to two prototypes:

- 1\(^{st}\) prototype ("straight tubes"): the process plate is made of straight tubes filled with internal swirls, inserted in a copper matrix and closed by HIP process. The cooling plates are made by brazing herringbones plates on both sides of the process plate. The prototype is made of 3 series process plates with a total reactant volume of 1.17 L and 4 alternately cooling plates fed in parallel. The characteristic sizes of the 1\(^{st}\) prototype are: 800x400x70 mm\(^3\).

- 2\(^{nd}\) prototype ("wavy channel"): both process and cooling plates are machined with through-laser. The characteristic size of the square cross-section wavy channels is of 2 mm. The prototype is entirely closed by HIP process. The prototype is made of 3 series process plates with a total reactant volume of 30 mL and 4 alternately cooling plates fed in parallel. The characteristic sizes of the 2\(^{nd}\) prototype are: 320x140x33 mm\(^3\).

Implementation of an exothermic reaction in the 2\(^{nd}\) prototype

A strongly exothermic reaction has been selected in order to demonstrate the benefits of coupling heat exchange and reaction in the same apparatus. This reaction is the oxidation of sodium thiosulfate \(\text{Na}_2\text{S}_2\text{O}_3\) by hydrogen peroxide \(\text{H}_2\text{O}_2\):
\[
2 \text{Na}_2\text{S}_2\text{O}_3 + 4 \text{H}_2\text{O}_2 \rightarrow \text{Na}_2\text{S}_3\text{O}_6 + \text{Na}_2\text{SO}_4 + 4 \text{H}_2\text{O}
\]

This liquid homogeneous reaction is irreversible, fast and highly exothermic. The heat released by this reaction is \(\Delta H = -586.2 \text{ kJ.mol}^{-1}\) of \(\text{Na}_2\text{S}_2\text{O}_3\). Moreover, as this reaction is temperature sensitive the conversion rate will depend on operating temperature of reactants and cooling power of the utility stream. This reaction is thus well adapted to test thermal performances of continuous intensified reactors \(^6\) \(^12\) \(^13\).

The experimental procedure is well documented in the literature \(^6\) \(^13\). Experimental conditions as well as conversion rates at reactor outlet are recapitulated in Table 4. \(Q_p\) is the total process fluid flowrate and \(t_r\) is the residence time of the process fluid in the reactor. \(T_{p,in}\), \(T_{p,out}\), \(T_{u,in}\), and \(T_{u,out}\) are respectively the temperatures at process and utility lines, inlet and outlet. During each experiment the utility line was fed with water at a 113 L.h\(^{-1}\) flowrate. The concentrations of sodium thiosulfate and hydrogen in their respective solutions were both fixed to 9% in weight. The ratio of hydrogen peroxide to sodium thiosulfate flowrates was 2.4, so the hydrogen peroxide was in excess.

Figure 10 shows the temperatures recording during the experiment carried out with \(Q_p = 14.0 \text{ L.h}^{-1}\). At 3340 seconds, the reactor is fed with reactants instead of water. Then the steady-state is reached at about 3480 seconds. It is thus possible to calculate the conversion rate at reactor outlet. For this purpose, two methods are used. They are both based on thermal balances: between inlet and outlet of process and utility streams in the reactor, and between sampling and thermal equilibrium in a Dewar vessel.

Conversion rates reported in Table 4 are rather high, and even of 100% for some experiments, depending on working temperature and flowrate. The conversion rate can be increased by a temperature increase as well as by a residence time increase.

The oxidation reaction has been run under strict temperature \((T_{u,in} \approx 40 – 60^\circ\text{C})\) conditions that would be impossible in a classical stirred tank reactor. In fact, working with such utility fluid temperatures would be impossible because of safety constraints, as it would certainly lead to reaction run-away. That is why this reaction could not have been implemented in a strict batch process since heat removal capacity would not have been sufficient, and a semi-batch mode would have been preferred consisting in feeding the reactants in the reactor during the reaction \(^6\).

**Figure 10:** Temperature profiles for experiment at \(Q_p = 14.0 \text{ L.h}^{-1}\)

<table>
<thead>
<tr>
<th>Process Fluid</th>
<th>Utility Fluid</th>
<th>Conversion rate (\chi)</th>
</tr>
</thead>
<tbody>
<tr>
<td>(Q_p) (L.h(^{-1}))</td>
<td>(t_r) (s)</td>
<td>(T_{p,in}) (°C)</td>
</tr>
<tr>
<td>14.0</td>
<td>6.9</td>
<td>17.6</td>
</tr>
<tr>
<td>5.0</td>
<td>19.3</td>
<td>19.3</td>
</tr>
<tr>
<td>7.0</td>
<td>13.8</td>
<td>20.0</td>
</tr>
<tr>
<td>7.0</td>
<td>13.8</td>
<td>20.7</td>
</tr>
<tr>
<td>7.0</td>
<td>13.8</td>
<td>21.1</td>
</tr>
</tbody>
</table>

**Table 4:** Experimental conditions and conversion rates in the 2\(^{nd}\) prototype.

**CONCLUSIONS AND FUTURE WORK**

The first two reactor concepts have been successfully designed, manufactured and experimentally characterized. The first design (straight tubes filled with twisted inserts) is very simple and fits all the predetermined specifications for the Rhodia application case, particularly the high flow rate request.
The investment costs are equal to the ones of an equivalent batch reactor and potential gains are expected in terms of product manufacturing costs (energy and raw materials savings, decrease of fixed costs,…). The main barrier of this design remains the quite high pressure drops in the case of pressure dependent reactions.

In order to intensify heat and mass transfer and improve the compactness of the apparatus, a second design has been proposed. The channel geometry has been 2D structured (wavy channel) while technological options have been validated to allow a low-cost manufacture. The investment cost corresponds to 50% of a batch reactor process and additional gains are expected thanks to the transposition from batch to continuous process. An exothermic reaction has been successfully implemented and highlighted the benefits of the transposition from a batch process to an intensified heat exchanger/reactor. Indeed, the reaction has been implemented in operating conditions which can not be reached in a batch reactor because of the poor heat removal performances. Thus, this opportunity to work with such temperatures and concentrations allows an increase of the kinetics and thus compensates for the small residence time of the process plate.

At the end of the RAPIC project, the two prototypes will be tested on the Rhodia test loop which implements a real process reaction, limited by the heat transfer performances of the technology. It will allow the comparison between classical semi-batch mode and the continuous one in intensified heat exchanger/reactor.

REFERENCES