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Modelling of chromatographic reactors

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Highlights

- A new chromatographic reactor model was developed.
- Methyl acetate hydrolysis was simulated.
- The performances were compared with most advanced existing model.
- Model implemented in gPROMS ModelBuilder.

1. Introduction

The esterification of carboxylic acid with alcohols is a hot topic of the modern bio-refinery, to produce, for example, solvents, plasticizers [1]. Nowadays, a lot of effort is in progress to optimize the synthesis of several esters. The right reactor should optimize the physical characteristics of the system that is, as before mentioned, a liquid-solid reaction. Among the different choices, the use of a chromatographic reactor [2]. For example, Mazzotti et al., instead, used the simulated moving bed reactor (SMBR) packed with Amberlyst-15 catalyst to perform acetic acid esterification to ethylacetate, demonstrating that the resin can be used both to catalyze the reaction and separate the stream. It is important to underline that to design such apparatus, accurate and reliable models are needed, involving profiles along different coordinates (time, reactor radius, reactor axis and catalyst radius) [3-5], the knowledge of physical and chemical properties, and powerful tools to reach a reliable solution. In the present work, the development of a reactor model, that can be used to interpret experimental data collected in chromatographic reactors will be presented. Methyl acetate hydrolysis catalyzed by DOWEX 50W-X8 catalyst was chosen as case study [6].

2. Methods

The developed model can be considered an advancement respect to the recent efforts published in the literature [6]. The breakthrough idea is to develop a model where no rate-determining-step is considered, writing the opportune mass balance equations on both the liquid bulk and intraparticle phases (see Table 1 for details). All the physical parameters were calculated from existing correlations.

Table 1. Model hypothesis and upgrades with existing strategy.

Vu and Seidel-Morgenstern [6]	*Present research*
Pseudo-homogeneous model	Fluid-solid model
Lumped axial dispersion	Axial dispersion
Kinetic regime	Fluid-solid mass transfer resistance
Kinetic regime	Intraparticle diffusion

The liquid bulk and intraparticle mass balance equations are respectively reported in Eq.s 1-2.

$$\frac{\partial C_{i,B}}{\partial t} = -u\frac{\partial C_{i,B}}{\partial z} + D_z\frac{\partial^2 C_{i,B}}{\partial z^2} - k_m a_{sp}\left(C_{B,i} - C_{i,L}\big|_{r_P=R_P}\right) \tag{1}$$

$$\varepsilon\frac{\partial C_{i,L}}{\partial t} + (1-\varepsilon)K_i\frac{\partial C_{i,L}}{\partial t} = \varepsilon\frac{D_P}{r_P^s}\frac{\partial}{\partial r_P}\left(r_P^s\frac{\partial C_{i,L}}{\partial r_P}\right) + (1-\varepsilon)\frac{D_S}{r_P^s}\frac{\partial}{\partial r_P}\left(r_P^s\frac{\partial K_i C_{i,L}}{\partial r_P}\right) + (1-\varepsilon)\nu_{ij}r_j \tag{2}$$

The model was implemented in gPROMS ModelBuilder v.4 software, by solving the axial and particle radial partial derivatives with a centered finite difference method with respectively 150 and 25 discretization points.

3. Results and discussion

Methyl acetate (MA) hydrolysis kinetic rate laws with related parameters are fixed and directly taken from literature [6]. The following simulation conditions were set: flowrate=0.75 mL/min, temperature=298K-328K, stream=pure water. The results of the developed model were compared with the output obtained by Vu and Seidel Morgenstern. Firstly, a simulation of an unreactive system was made with the two models (present and literature model), by injecting a three-component pulse (MA, acetic acid, methanol, 0.5 mol/L each) at the inlet of the column. The results reported in Figure 1-left clearly demonstrates that two models show the same output. Even if the output is very similar, the novelty of our model is that all the necessary physical parameters are estimated with existing correlations, i.e. the diffusivities. This is clearly a novelty in the field, because the existing models lump the intraparticle diffusion in adjustable semi-empiric parameters fitted on the peak width, having no physical meaning. Thus, our model is characterized by a more predictive power.

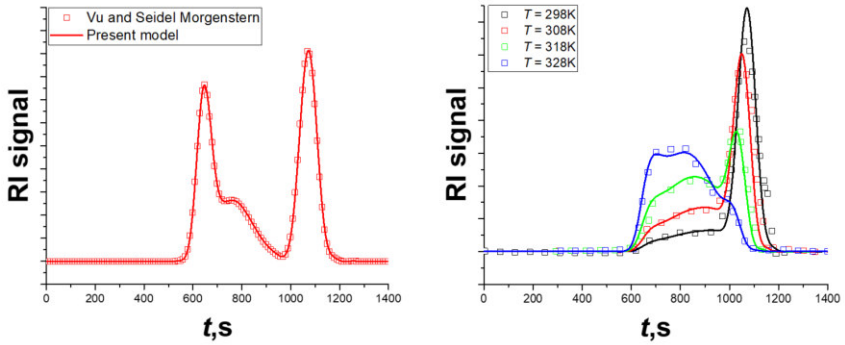

Figure 1. Comparison with literature model (left): symbols represent the output obtained with Vu and Morgenstern model [6], lines the output of our model; description of experimental data [6] (right): symbols are experimental data, lines the model description.

A second simulation was performed by considering the reaction, by simulating the injection of 100µL 0.5mol/L MA in water at different temperatures (Figure 1-right). As it can be seen, our model correctly describes the experimental data reported in the literature, with no additional fitting parameters.

4. Conclusions

A novel chromatographic reactor model was developed to interpret experimental data dealing both with the esterification of carboxylic acids and with the hydrolysis of organic esters. The model is advanced respect to the state of the art because no rate-determining steps are considered. All the physical and chemical phenomena are taken into account. In perspective, the model can be applied in optimizing the esterification/hydrolysis processes.

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Keywords
Reactive chromatography; modelling; esterification; heterogeneous catalysis.

Vincenzo Russo, PhD

- 43 papers, 2 book chapters
- *h*-index = 10
- 338 total citations by 252 documents
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Short history

Vincenzo Russo was born in Naples on 02/05/1985. On 09/05/2014 he got his PhD title in Chemical Sciences in the Chemical Sciences Department of the University of Naples "Federico II", with a thesis entitled “Kinetic and Catalytic Aspects in Propene Oxide Production”, on the synthesis of propene oxide subject, from propene and hydrogen peroxide in the presence of TS-1 catalyst, investigating both the kinetic and catalytic aspects. From May 2014 to July 2016 he was a Post-Doctoral Researcher at the Department of Chemical Sciences of the University of Naples "Federico II" (Napoli, Italy), working on reaction modelling, catalysis and kinetic investigation. From July 2016 till September 2017 he worked at Åbo Akademi University (Turku, Finland) on multiphase reactors modelling as senior researcher. From September 2017 till now he is working as Assistant Professor (RTD-A) at the at the Department of Chemical Sciences of the University of Naples "Federico II" (Napoli, Italy).

Scientific contributions and research field of interest

- Author of 43 papers appeared on international peer-reviewed Journals and 2 book chapters.
- His main scientific activities deal with:
  - Reaction and reactor modelling of multiphase systems
  - Kinetic investigation of catalytic reactions
  - Safety criteria for exothermic reactions
  - Design and realization of lab-scale pilot plants
  - Plant automation and control

- His scientific activities are carried out in collaboration with public and private partners:
  - Åbo Akademi University, Turku (FI)
  - Eindhoven University of Technology, Eindhoven (NL)
  - Fraunhofer ICT-IMM, Mainz (D)
  - Dipartimento di Ingegneria Civile, Chimica, Ambientale e dei Materiali, Università di Bologna, Bologna (IT)
  - Dipartimento di Ingegneria Civile Edile e Ambientale, Università degli Studi di Napoli “Federico II”, Napoli (IT)
  - Dipartimento di Ingegneria chimica, dei Materiali e della Produzione industriale, Università degli Studi di Napoli “Federico II”, Napoli (IT)
  - Istituto per i Polimeri, Compositi e Biomateriali (IPCB) – CNR, Portici/Pozzuoli (IT)
  - CONSER S.p.A., Roma (IT)

Job experiences and formation

• Date	04/09/2017 – today
• Institute Name	University of Naples “Federico II” – Chemical Sciences Department
• Subject	Industrial Chemistry (CHIM/04)
• Qualification	Assistant Professor (RTD-A).

• Date	19/07/2016 – 01/09/2017
• Institute Name	Åbo Akademi– Laboratory of Industrial Chemistry and Reaction Engineering
• Subject	Industrial Chemistry
• Qualification	Fellowship holder: "Multidimensional reactor models for fluid-solid systems", Johan Gadolin Scholarship – Process Chemistry center.

• Date	01/01/2016 – 19/07/2016
• Institute Name	University of Naples “Federico II” – Chemical Sciences Department
• Subject	Industrial Chemistry
• Qualification	Research grant: “Studio della sintesi di poliesteri e poliammidi da acido succinico ottenuto da brodi di fermentazione e scale-up del processo”, ambito disciplinare Scienze Chimiche, BioPoliS (PON03PE_00107_1/1 CUP E48C14000030005).

• Date	01/07/2015-30/11/2015
• Institute Name	University of Naples “Federico II” – Chemical Sciences Department
• Subject	Industrial Chemistry
• Qualification	Fellowship holder: "Sviluppo di modelli matematici di simulazione ed ottimizzazione di fermentatori industriali", POR Campania FERS 2007/2013 - Bio Industrial Processes (BIP) - project for a regional biotechnologies network in Campania CUP B25C13000290007.

• Date	12/05/2014-15/05/2015
• Institute Name	University of Naples “Federico II” - Civil, Architectural and Environmental Engineering Department
• Subject	Industrial Chemistry
• Qualification	Fellowship holder: PON04_a2F B&SAVE-AQUASYSTEM-SIGOLD (CUP E61H2000170005), BORS 14 DICEA/2014.

• Date	09/05/2014
• Institute Name	University of Naples “Federico II” - Chemical Sciences Department
• Subject	Industrial Chemistry
• Qualification	PhD in Chemical Sciences

• Date	01/11/2013 - 28/02/2014
• Institute Name	Åbo Akademi– Laboratory of Industrial Chemistry and Reaction Engineering
• Subject	Industrial Chemistry
• Qualification	Visiting student

• Date	15/09/2010-01/03/2011
• Institute Name	University of Naples “Federico II” - Chemical Sciences Department
• Subject	Industrial Chemistry
• Qualification	Fellowship holder: "Metodi innovativi per la produzione di oli Epossidati", cooperation with Mythen SpA.

• Date	23/06/2010
• Institute Name	University of Naples “Federico II” - Chemical Sciences Department
• Subject	Industrial Chemistry
• Qualification	Master degree in Sciences and Technologies of Industrial Chemistry - 110/110 cum laude.

• Date	28/03/2008
• Institute Name	University of Naples “Federico II” - Chemical Sciences Department
• Subject	Industrial Chemistry
• Qualification	Bachelor degree in Industrial Chemistry - 110/110 cum laude.

Scientific contributes

Papers on magazines

1. E. Santacesaria, R. Tesser, M. Di Serio, V. Russo, R. Turco. A New Simple Microchannel Device To Test Process Intensification. *Industrial & Engineering Chemistry Research* **2011**, 50, 2569-2575. (*Citations*: 9, *5-Years I.F.*: 2.567)
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35. V. Russo, O. Ortona, R. Tesser, L. Paduano, M. Di Serio. On the importance to choose the Best Minimization algorithm for the determination of ternary diffusion coefficients by Taylor dispersion method. *ACS Omega* **2017**, 2, 2945-2952.

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37. D.Yu Murzin, S. Garcia, V. Russo, T.T. Kilpiö, L.I. Godina, A. Tokarev, A. Kirilin, I.L. Simakova, S. Poulston, D.A. Sladkovskiy, J. Warna. Kinetics, modelling and process design of hydrogen production by aqueous phase reforming of xylitol. Accepted on *Industrial & Engineering Chemistry Research* **2017**. DOI: 10.1021/acs.iecr.7b01636. (*Citations*: 0, *5-Years I.F.*: 2.567)

38. V. Russo, T. Salmi, F. Mammitzsch, O. Jogunola, R. Lange, J. Wärnåa, J.-P. Mikkola. First, second and nth order autocatalytic kinetics in continuous and discontinuous reactors. *Chem. Eng. Sci.* **2017**, 172, 453-462. (*Citations*: 0, *5-Years I.F.*: 2.567)

Conference papers (Scopus source)

1. E. Santacesaria, M. Di Serio, R. Tesser, R. Turco, M. Tortorelli, V. Russo. Biodiesel process intensification in a very simple microchannel device. *Institution of Chemical Engineers Symposium Series* **2011**, 157, 122-130. EPIC2011.
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3. E. Salzano, V. Russo, R. Tesser, M. Di Serio. Runaway reaction in the epoxydation of vegetable oil. *21st International Congress of Chemical and Process Engineering, CHISA 2014 and 17th Conference on Process Integration, Modelling and Optimisation for Energy Saving and Pollution Reduction, PRES* **2014**, 4, 1946.
4. V. Russo, T. Kilpiö, M. Di Serio, R. Tesser, E. Santacesaria, T. Salmi. Dynamic non isothermal trickle bed reactor with both internal diffusion and heat conduction: Arabinose hydrogenation as a case study. *21st International Congress of Chemical and Process Engineering, CHISA 2014 and 17th Conference on Process Integration, Modelling and Optimisation for Energy Saving and Pollution Reduction, PRES* **2014**, 1, 6.
5. M. Stanzione, V. Russo, A. Sorrentino, R. Tesser, M. Lavorgna, M. Oliviero, M. Di Serio, S. Iannace, L. Verdolotti. Bio-based polyurethane foams from renewable resources. *AIP Conference Proceedings* **2016**, 1736. TOP2016.

Book chapters

1. T. Kilpiö, V. Russo, K. Eränen, T. Salmi. Design and modelling of laboratory scale three-phase fixed bed reactors. B. Saha, Catalytic Reactors. De Gruyter Textbook **2015**.
2. V. Russo, R. Tesser, M. Di Serio. Advances in Catalysts for Biofuels Production Processes. M.R. Riazi, D. Chiaramonti, Biofuels Production and Processing Technology. CRC Press **2017**.

PRIZES

1. PSE Model-Based Innovation Prize runner-up, 2015. Winning paper: V. Russo, T. Kilpiö, J. Hernandez Carucci, M. Di Serio, T. Salmi. Modeling of microreactors for ethylene epoxidation and total oxidation. Chemical Engineering Science **2015**, 134, 563–571.

Conferences contributes

Chairman activity

1. Chair activity at CHISA 2016 - 27-31 August **2016** Prague, Czech Republic.

Oral presentations

1. E. Santacesaria, M. Di Serio, R. Tesser, R. Turco, M. Tortorelli, V. Russo. Biodiesel process intensification in a very simple microchannel device. Abstracts book of European process intensification conference (EPIC). Manchester (UK), 20-23 June **2011**.
2. V. Russo, R. Turco, M. Di Serio, R. Tesser, E. Santacesaria. A Biphasic Kinetic Approach to Biodiesel Production. Abstracts book of XXIV SCI Congress, Lecce (IT), 11-16 September **2011**.
3. V. Russo, R. Tesser, E. Santacesaria, M. Di Serio. Kinetics of Propene Oxide Production via Hydrogen Peroxide with TS-1. Abstracts book of XVII National Congress of Catalysis GIC 2013 and XI National Congress of Zeolites Science and Technology, Riccione (IT), 15-18 September **2013**.
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6. V. Russo, R. Tesser, M. Trifuoggi, M. Di Serio. Fluid-solid adsorption from batch to continuous. CHISA 2016 - 27-31 August **2016** Prague, Czech Republic.
7. C. de Araujo Filho, D. Mondal, T. Kilpiö, V. Russo, K. Eränen, T. Salmi. Continuous technology in the valorization of glycerol: the flow pattern changes radically inside the bubble column. 14th Multiphase Flow Conference & Short Course, Dresden (D) in 8-10 November **2016**.

Poster presentations

1. V. Russo, R. Tesser, M. Di Serio, E. Santacesaria. A New Kinetic Approach Applied to Biodiesel Process Intensification. Abstracts book of DGMK, Berlin (D), 8-10 October **2012**.
2. V. Russo, T. Kilpiö, A.P. Nebreda, M. Di Serio, T. Salmi. Laminar flow reactor for acid hydrolysis of hemicelluloses. IMRET 13 "International Conferences on Microreaction Technology" - Budapest, Hungary in June 23-25, **2014**.
3. V. Russo, L. Minieri, R. Tesser, A. Aronne, M. Di Serio. Ru-based catalysts for γ-valerolactone synthesis. XIX Congresso Divisione Chimica Industriale - Salerno, Italy in 14-16 September, **2015**.
4. V. Russo, R. Tesser, D. Masiello, M. Trifuoggi, M. Di Serio. Fluid-solid adsorption from batch to continuous: methylene blue on silica as a case study. ECCE 10, "10th European Congress of Chemical Engineering" - Nice, France in 27 September - 1 October **2015**.
5. R. Tesser, M. Di Serio, V. Russo, S. Iannace, E. Di Maio, M. Oliviero. Bio-based Aliphatic Polyesters for Foams Formulations. Biofoams 2015, "5th International Conference on Biofoams" - Sorrento, Italy in 13-16 October **2015**.

Reviewer activity

- Chemical Engineering Journal
- Bioresource Technology
- Canadian Journal of Chemical Engineering
- Industrial & Engineering Chemistry Research
- Chemical Engineering Communications
- Adsorption
- International Journal of Oil, Gas and Coal Technology
- International Journal of Thermal Sciences
- Fuel Processing Technology

Teaching activity

Since 2014 for the Chemical Science Department of the Università di Napoli "Federico II" (Consiglio del Dipartimento di Scienze Chimiche n. 2 del 20/02/20), for CHIM04 sector (Industrial Chemistry), he is part of the exam commissions of the following teachings:

1. Introduzione alla Chimica industriale e al calcolo di processo
2. Principi di Chimica Industriale con esercitazioni
3. Chimica Industriale I
4. Tecnologie Avanzate nei processi chimici industriali
5. Qualità, sicurezza e tutela brevettuale nell'industria chimica

Invited lectures

1. Lecture entitled "Reactor simulations" for the course "Principles of Chemical Reaction Engineering", held for students of the English Master's program in Chemical Engineering, Åbo Akademi University, Turku, Finland on October 17th, **2016**.

2. Lecture entitled "Reactor simulations" for the course "Kemisk Reaktionsteknik" ("Chemical Reaction Engineering"), held for students of the Master's program in Chemical Engineering, Åbo Akademi University, Turku, Finland on December 9th, **2016**.

Co-advisor activity

Master degree thesis

1. B. Esposito. HPPO: validazione dei modelli cinetici e ottimizzazione dei parametri operativi. A.Y. 2013-2014.
2. A. Natale. Studio della reazione di sintesi di poliesteri alifatici da acido succinico e un diolo. A.Y. 2013-2014.
3. D. Masiello. Dynamic Model for Adsorption Kinetics in Multi-Scale Systems. A.Y. 2014-2015.

Bachelor degree thesis

1. A. Opera. Studio della decomposizione dell'acqua ossigenata per il processo di formazione degli oli epossidati. A.Y. 2012-2013.
2. R. Morlando. Cinetica del processo HPPO ad alta pressione. A.Y. 2013-2014.
3. C. Salvi. Studio dell'adsorbimento del blu di metilene su silice in condizioni batch. A.Y. 2014-2015.
4. L. Carnevale. Studio cinetico della reazione di decomposizione dell'acido performico in condizioni di utilizzo industrial. A.Y. 2014-2015.
5. A. Oglio. Studio dei catalizzatori eterogenei per la decomposizione dell'acqua ossigenata. A.Y. 2014-2015.
6. D. Zannini. Metodi analitici per la sintesi del polietilensuccinato. A.Y. 2014-2015.
7. G. Coppola. Studio dell'adsorbimento del blu di metilene su silice in continuo. A.Y. 2014-2015.
8. C. Capolongo. Studio cinetico del processo HPPO ad alta pressione. A.Y. 2014-2015.

9. A. Broccoli. Nuovi catalizzatori eterogenei per la sintesi del γ-valerolattone. A.Y. 2014-2015
10. L. Scotto d'Antuono. Sintesi di poliammidi a partire da derivati dell'acido succinico. A.Y. 2015-2016.
11. G. Di Rienzo. Materiali derivati da biomasse per l'adsorbimento fluido-solido. A.Y. 2015-2016.
12. R. Guidone. Modelli matematici per il processo di sintesi delle cloridrine con acido cloridrico gassoso. A.Y. 2015-2016.

Activity in national and EU projects

01/01/2016 – 19/07/2016	BIOPOLIS PON 2007-2013. **Activity**: "Studio della sintesi di poliesteri e poliammidi da acido succinico ottenuto da brodi di fermentazione e scale-up del processo"
01/07/2015 – 30/11/2015	P.O.R. Campania FERS 2007/2013 - Bio Industrial Processes (BIP) - project for a Regional Biotehcnologies Network in Campania, CUP B25C13000290007. **Activity**: "Sviluppo di modelli matematici di simulazione ed ottimizzazione di fermentatori industriali"
12/05/2014 – 15/05/2015	PON04_a2F B&SAVE-AQUASYSTEM-SIGOLD (CUP E61H2000170005), BORS 14 DICEA/2014. **Activity**: "Modellazione di colonne di adsorbimento fluido-solido"
2007 – 2013	COPIRIDE project (Combining Process Intensification-driven Manufacture of Microstructured Reactors and Process Design regarding to Industrial Dimensions and Environment), directed by prof. V. Hessel (http://www.copiride.eu/). **Activity**: "Modeling of epoxidation reactors".

Activity in cooperation with industrial partners

2011 – 2015	Propene oxide synthesis in cooperation with CONSER S.p.A.
15/09/2010 – 01/03/2011	Epoxidized soybeanoil production in cooperation with Mythen S.p.A.

Attended schools

1. "LabVIEW Core 1 and Core 2" – Università di Napoli "Federico II" 1-5 July 2013.
2. "Scuola di Calcolo Scientifico con MATLAB" – Palermo 29th August 2011 – 2nd September 2011.
3. "Deposition of catalyst into microchannels" 10th-12th February 2010.
4. Tenth Post-Graduate Summer School on Green Chemistry – Interuniversity Consortium "Chemistry For The Environment" 12th-18th October 2008.
5. Certified English course: Entry Level Certificate in English (ESOL) – Entry 3 – Preliminary English Test (PET) – Level B1 November 2002.

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