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REVIEW REPORT
on
Ph.D. Thesis of Przemysław Wojewódka
entitled:
"Hydrodynamics and mass transfer in spinning fluids reactor"

The review was prepared based on the letter of Professor Tomasz Sosnowski, the chairman of the scientific board for the chemical engineering discipline of the Warsaw University of Technology from 28th of October 2020. After the initial analysis of the dissertation content, I conclude that its subject matter falls within the scope of my scientific interests, which allows me to undertake the preparation of this review. At the same time, I declare that I do not have joint publications with the Ph.D. candidate.

Introduction

The submitted thesis is dedicated to the experimental research concerning hydrodynamics and mass transfer phenomenon in a prototype of a multiphase reactor which may be classified as a bubbling-type gas-liquid contactor. The novelty of the design idea is based on the application of centrifugal forces within spinning fluids for development of extremely high mass transfer surfaces. The dissertation is written on altogether 155 pages and it is divided into 17 chapters. Firstly, a short introduction to the research is presented in which the main features of the Spinning Fluid Reactor (SFR) are discussed. Moreover, Author presents the most important research challenges and tasks related to the SFR design. They may be divided into two groups which are connected with the residence time distribution and mass transfer parameters. In both cases Author's goals are to find correlations for geometrical and process parameters and to determine a mathematical models describing the residence time distribution and mass transfer parameters for a wide range of process conditions. Next, three papers of the Ph.D. candidate are presented:

- R. Aranowski, **P. Wojewódka**, A. Zielińska-Jurek, R. Bokotko, C. Jungnickel, Spinning Fluids Reactor: A new design of a gas – liquid contactor, *Chem. Eng. Proc.* 116 (2017) 40-47.
- **P. Wojewódka**, R. Aranowski, C. Jungnickel, Residence time distribution in rapid multiphase reactors, *J. Ind. Eng. Chem.* 69 (2019) 370-380.
- **P. Wojewódka**, R. Aranowski, C. Jungnickel, Mass transfer in Spinning Fluids Reactor – Measurement and prediction, *J. Ind. Eng. Chem.* 80 (2019) 712-721.

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Each publication is preceded by an introduction, which provides additional information about the conducted research, and conclusions in which the author discusses the obtained results in the context of future investigations. The goal of the first paper is the introduction of the idea of the Spinning Fluid Reactor. General design of the apparatus is discussed. Authors used high-speed camera in order to determine the bubble size distribution. On that basis the mass transfer area was calculated. Moreover, the overall volumetric mass transfer coefficient was measured using the water-oxygen system absorption method.

In the second paper the residence time distribution was measured using impulse-response method. Since the tracer pulse signal was wider than expected residence time distribution the deconvolution method was used in order to obtain valid results. As tracer saturated solution of sodium chloride was used. It was detected by the electrical resistance measurements. Moreover, Authors have successfully developed equations to predict the mean residence time and variance of distribution as the functions of gas and liquid volumetric flow rates, inner porous partition open area, mesh thickness, porosity and pore diameter. For that purpose an evolutionary algorithm was used.

The third publication is strictly devoted to the investigation of mass transfer parameters. In experiments cobalt ions were used with sodium sulfite/oxygen system assuring differing reaction kinetics for determination of specific mass transfer area and overall volumetric liquid side mass transfer coefficient. Additionally, the investigation concerning the correlation between froth electrical conductivity and mass transfer parameters was done.

Next, Authors shortly present their considerations concerning practical application of the spinning fluid reactors. Three cases are discussed, namely absorption of carbon dioxide in diglycolamine as an upgrade of biogas plant, degassing plant as a part of the "Blue Gas" project supported by the National Center for Research and Development, and SFR as a micro-reactor. In the two first cases some exemplary results are presented, whereas in the third case only the key parameters of the reactor are given.

At this point the dissertation is concluded with the overall summary and the literature list which consist of 87 references to books, scientific papers (including three papers of Ph.D. candidate), Ph.D. thesis, patents and commercial product information webpage. Only 24 of them was published during last ten years. The oldest publication is from 1904. Next, the academic achievements of the Ph.D. candidate are presented. As the last chapter the technical drawings of the reactors and laboratory stands are attached. At the end of the dissertation the author's statements are included.

At the moment of this review report creation Mr. Przemysław Wojewódka has the following scientific achievements:

- 3 publications in journals from the JCR list (total impact factor IF = 13.082):
    - Chemical Engineering and Processing: Process Intensification (2017) – IF = 2.826
    - Journal of Industrial and Engineering Chemistry (2 publications, 2018 and 2019) – IF = 4.978 and IF=5.278
- chapters in 2 monographies published by:
    - CRC Press (1 chapter)
    - Wydawnictwo Politechniki Gdańskiej (1 chapter)
- Four of abovementioned works are indexed in Web of Science database. The total number of citations is equal to 13 from which only 2 are autocitations. The Hirsh index is equal to 2.
- 5 presentations during Polish conferences
- 1 patent claim
- Cooperation in three research projects:
    - Blue gas, grant No. BG1/EKOŁUPKI/13 supported by The National Centre for Research and Development
    - Advanced Technology of Energy production, strategic program of research and development supported by ENERGA S.A.
    - High Copper, grant No. CuBR/II/7/NCBR/2015 supported by The National Centre for Research and Development and KGHM Polska Miedź S.A.

It is worth to mention that publication entitled "Spinning Fluids Reactor: A new design of a gas - liquid contactor" is already cited 7 times and publication entitled "Residence time distribution in rapid multiphase reactors" is already cited 5 times.

General and specific comments

From the beginning of its existence one of the aims of chemical engineering is to achieve highest possible efficiency of the processes which will in turn positively influence the economical as well as ecological aspects of the production systems. The topic of the presented dissertation fits well in this trend. According to Web of Science database from over 10 years more than 100 scientific papers dedicated to the multiphase reactors are published each year. In 2019 this number was equal to 202 publications. This fact proves that the discussed filed of science is extremely complicated but gains more and more interest each year. New solutions are proposed like application of microreactors with microporous structure, membranes or nanotechnology. As Ph.D. candidate stated in one of his publications the employment of the spinning movement and resulting centrifugal forces is not a new idea. Such designs were proposed already in the first half of the XXth century. The solution presented in the dissertation is a substantial enhancement of previous ideas allowing one to achieve significantly better results compared to the currently used methods. I would like to strongly emphasize the utilitarian aspect of this work. The spinning fluid reactor applicability for industrial processes was investigated in number of semi-technical scale plants. The experimental research results were published in three scientific papers in prestigious and high impact factor journals.

Nevertheless, I would like to discuss some issues concerning the dissertation with the Ph.D. candidate. At this point I would like to emphasize that my questions and remarks should be treated as an opening for a scientific debate or guidance for future work and not as a thoughtless criticism. They do not affect my high opinion of the presented work. My questions and remarks are presented below:

1. One can find some stylistic and grammar errors in the text of dissertation, e.g.:

   Page 5, it is: "(…)a bubbling-type gas-liquid contactor using centrifugal force develop the mass transfer surface."
   It should be: "(…)a bubbling-type gas-liquid contactor using centrifugal force **to** develop the mass transfer surface."

   Page 15, it is: "(…) method for Sauter diameter determination"
   It should be: : "(…) method for Sauter **mean** diameter determination"

   Page 24, it is: "(…) oxygen concentration on water (…)"
   It should be: "(…) oxygen concentration **in** water (…)"

   Page 25, it is: "(…) media resident time (…)"
   It should be: "(…) media **residence** time (…)"

   Page 86, it is: "(…) the liquid levels were 65 dm^3 (…)"
   It should be: "(…) the liquid **volumes** were 65 dm^3 (…)"

   Page 87, it is: "The sum of mass change rates in the system (…)"
   It should be: "The sum of **volume** change rates in the system (…)"

   Academic achievements:
   It is: "Journal of Industrial an Engineering Chemistry"
   It should be: "Journal of Industrial **and** Engineering Chemistry"

Authors statements:
There is no such word as "nakonceptualizacja"

2. Minor mistakes and errors in the text:

Nomenclature:
SI units should be used.
Henry's constant is not dimensionless. Later in the text the unit atm·dm^3/mol is used.
Flow rates should be specified, i.e. mass flow rate, volumetric flow rate, molar flow rate etc.
If "*R*" has unit J/(mol·K) then it represents **Universal** Gas Constant.
Author should decide which form he wants to use: "flowrate" or "flow rate"

Page 88, Table 3: usually the pressure unit is given as barg (or bar(g)) which stands for gauge pressure and bara (or bar(a)) which stands for absolute pressure; the last row of the table represents degassing efficiency, not rate. Term "rate" suggest that the discussed phenomenon is referred to time unit.

Page 93, Table 4: the first row is titled as "Nominal liquid flow rate" however the unit is given in dm^3

Literature:
[10] – Journal abbreviation is: *Chem. Eng. Proc.*
[20] – city should be given
[21] – the information that this is a Ph.D. thesis should be given
[48] – this is a paper published in Advances in Chemical Engineering vol. 11, pp. 1-133.
[56] – is a chapter in book; editors and publisher should be given

Technical drawings:
Since the dissertation is written in English dots should be used instead of comas.
All figures are not to scale. Although each drawing has the same size different scales and formats are given in description. This issue should be solved by rescaling the drawings to appropriate format or by attaching additional documentation in proper size. Author could also place a comment that the drawings are for reference only and are not to scale.
In some cases the crossing of the auxiliary sizing lines could be avoided.
In some cases the closed chain of sizes is given which should be avoided.

3. Questions concerning paper titled "Spinning Fluids Reactor: A new design of a gas – liquid contactor":

3.1 Since in literature two different frictions factors are used, namely Fanning friction factor and Darcy-Weisbach friction factor, which of them is used in this work?
3.2 In the text of publication "flow ratio" term is used. What does it represents since its unit is dm^3/min?
3.3 In the summary of the article Author stated that one of the conclusions from this work is that the gas hold-up volumetric measurements are not proper for such a dynamic system that the SFR is and require change of methodology or elimination. However, I cannot find such information or analysis in the text of publication. Could author be so kind and explain this issue?

3.4 On the 98th page of dissertation Author mentions that in this paper the impact of the internal porous partition length on the mass transfer coefficient was confirmed. However, as in the case of previous questions I could not find such analysis – just one statement about optimal size of the reactor. Could author be so kind and explain this issue?

4. Questions concerning paper titled "Residence time distribution in rapid multiphase reactors":

4.1 On what basis the value of filtering parameter was set to 10 in presented research? How important was this parameter for the residence time distribution determination (sensitivity analysis)?

4.2 In the introduction Author states that: "Application of CFD and numerical methods of flow modeling is also limited because of significant turbulency of the flow." Here, Author refers to 3 publications of Sheikholeslami and co-workers dedicated to the simulations of nanofluids magneto-hydrodynamics. In each work different numerical method was successfully used, i.e. Homotopy analysis method, Control Volume Based Finite Element Method and Lattice Boltzmann method. Therefore, I cannot agree with that statement. In my opinion, the Computational Fluid Dynamics would be a great tool for the analysis of the researched reactor. Nowadays, even not experienced users have access to a vast variety of mathematical models of turbulence and multiphase flows both in commercial type of software like ANSYS Fluent or open source software like OpenFOAM. Moreover, more advanced users may tweak the existing models or even write these models on their own. The CFD would allow Ph.D. candidate to analyze the behavior of liquid and gas phase within the reactor and thus gain deeper knowledge about the process. Concluding, I strongly recommend to consider the application of CFD in the analysis of the SFR.

5. Questions concerning paper titled "Mass transfer in Spinning Fluids Reactor – Measurement and prediction":

5.1 This question considers the second and third publication of the Ph.D. candidate. In both papers the evolutionary algorithm was used for the development of mathematical dependencies for mean residence time, variance of distribution and overall volumetric liquid side mass transfer coefficient. It was motivated by the engineering approach to this issue. In the result very simple equations were obtained. I want to ask, wouldn't there be a better approach to create equations using dimensionless modules and criterial numbers? Such approach was successfully used for many years for the description of numerous phenomena like heat transfer, mass transfer, pressure drop etc.

5.2 In this publications Ph.D. candidate tried to use Higbie's correlation and Frossling's correlation for liquid-side mass transfer coefficient finding that the obtained values did not match the experimental results. However, it was already proved in multiple publications that these two correlations are valid only for a specific range of gas bubbles sizes. For example according to Motarjemi and Jameson (Chem. Eng. Sci. 33 (1978) 1415-1423) Frossling's correlation is valid for diameters up to 150 μm whereas the Higbie's correlation is valid for diameters bigger than 2 mm. If one take a look at the figure 7 in the first publication it is clear than in the SFR the bubble size range varies from 50 μm to over 5 mm, however the highest volume fractions are encountered in the range 0.5 mm to 2 mm. This simply means that the two abovementioned correlations could not give the valid results. In the same paper authors propose to use correlations of Calderbank and Moo-Young (Chem. Eng. Sci. 16 (1962) 39-54) but one can easily find other models in the literature for such calculations (e.g. Dudley, Wat. Res 29 (1995) 1129-1138).

6. General questions:

6.1 It seems that the key sizes and their ratios for the SFR are fixed values. For example Ph.D. candidate did not investigate the impact of reactor's diameter on the mass transfer phenomenon. One can find only one information concerning the optimal length of the IPP. Could author elaborate on this issue?

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Final evaluation and conclusion

Reading the Ph.D. thesis, it is clear that Mr. Przemysław Wojewódka has all necessary practical skills and high motivation for research work. Candidate proves his understanding of the state-of-the-art in the undertaken research area. Once again I would like to strongly emphasize the utilitarian aspect of this work. The spinning fluid reactor applicability for industrial processes was investigated in number of semi-technical scale plants. The experimental research results were published in three scientific papers in prestigious and high impact factor journals. All performed experiments were well arranged, techniques and methods were correctly applied. Experimental results are presented clearly and with the appropriate statistical analysis. The methodology is sound and fulfils the standards of good scientific practice. To conclude, this thesis presents a very good scientific level and it is interesting to read. Its quality is fully in agreement with requirements for Ph.D. dissertation according to Polish law act of 14th March 2003 on Academic Degrees and an Academic Title as well as on Degrees and a Title in Art (Journal of Laws of 2017, item 1789).

Taking all abovementioned into account I strongly recommend that Mr. Przemysław Wojewódka should be allowed to the public defense and subsequent steps in the procedure leading to Ph.D. degree.