



## Preface: The 1st International Symposium of Indonesian Chemical Engineering (ISICChem) 2018 Hakun Wirawasista Aparamarta

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<sup>5</sup> Mechanical Engineering Department, University of Sumatera Utara (USU), Indonesia

This proceedings volume contains selected articles from the “1<sup>st</sup> International Symposium of Indonesian Chemical Engineering (ISICChem) 2018” held in Kryad Bumi Minang Hotel, West Sumatra, Indonesia from October 4-6, 2018, that has been peer reviewed.

Despite the fact that this is only our first international symposium of Indonesian Chemical Engineers, and the sixth for the national one, we are so honored to announce that this year we have abundance of enthusiastic participants from five different countries participating in the conference. In the October 4-6, 2018, 250 research papers have been presented in the ISICChem, covering eleven research areas, such as: Biochemical Engineering, Catalyst and Reaction Engineering, Waste Water Treatment Technology, Renewable Energy Technology, Nano Materials and Nanotechnologies, System Engineering Process, Thermodynamics and Supercritical Technology, Separation and Purification Technology, Food and Pharmaceutical Technology, Chemical Engineering Management, and Chemical Engineering Education. We are also delighted to announce that the selected papers will be published in IOP Conference Series: Materials Science and Engineering.

We would like to take this opportunity to acknowledge the incredible support from our main sponsors: STARBORN (PT. Luas Birus Utama), PT. Semen Indonesia, BPJS Ketenagakerjaan, PT. Pupuk Indonesia, and PTPN X. I would also like to express my sincere gratitude to all the steering and organizing committees from Institut Teknologi Sepuluh Nopember (ITS) and Universitas Bung Hatta (UBH) and all the parties for the hard work and tremendous effort they have dedicated to make this event successful.

Alongside our seminars, another important agenda of the national chemical engineers is also taking place at the same time, namely the Annual General Meeting (Musyawarah Nasional) of the Indonesian Chemical Engineering Education Association (APTEKIM).

We would like to thank again the speakers, presenters, participants, and sponsors, who were responsible for the success of the seminar, and look forward to the second edition of the ISICChem seminar.

**Hakun Wirawasista Aparamarta, PhD**

Chairman of ISICChem 2018



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# Table of contents

Volume 543

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◀ Previous issue      Next issue ▶

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# Hydrodynamic Analysis of Hydrolysis of the Rice Husk Cellulose by Using CFD Modeling

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**Abstract.** The importance of the rule and the demand of glucose trigger the seeking of the alternative materials to produce glucose. One of the alternatives used is the utilization of biomass such as the rice husk to produce glucose. Rice husk is the agricultural residue whose availability is very abundant. In this research, rice husk contained cellulose (42.2 %), hemicellulose (17.04 %), and lignin (20.46 %). First step to produce the glucose from rice husk was pretreatment stages (Soaking in Aqueous Ammonia pretreatment and Acid pretreatment). Furthermore it was followed by the acid hydrolysis step by using dilute H<sub>2</sub>SO<sub>4</sub> solution. The concentration of H<sub>2</sub>SO<sub>4</sub> used in this work was 1, 2, and 3 N with the various hydrolysis times of 30, 60, 90, 120, and 150 min. The result showed that increasing of hydrolysis time slightly increased glucose concentration. The highest glucose concentration was about 21.51% at H<sub>2</sub>SO<sub>4</sub> concentration of 2 N and the hydrolysis time of 150 min. CFD modeling (ANSYS Fluent 16) was performed to investigate the hydrodynamic phenomena during hydrolysis. The hydrodynamic analysis showed that the best mixing occurred at H<sub>2</sub>SO<sub>4</sub> concentration of 2 N for 150 min. It is a good agreement with the experimental finding.

## 1. Introduction

Rice husk is agricultural residue whose availability is very abundant. It can produce glucose because it contains lignocellulose structure. The steps to produce glucose from rice husk are pretreatment and hydrolysis. Pretreatment aims to damage the lignin bond and break down a structure of cellulose crystal easily into glucose. Immersion in an ammonia solution is performed to reduce the amount of lignin and increase cellulose porosity, while the treatment using dilute sulphuric acid is done to increase cellulose porosity and help partially hydrolyse cellulose. Cellulose can be hydrolysed into glucose using aqueous media and assisted with acid or enzyme catalysts. The imperfection of the hydrolysis process can produce cellulose disaccharides. While the process of hydrolysis that runs perfectly will produce cellulose monomer in the form of glucose.

The advantage of chemical hydrolysis process is low cost. The price of chemicals is cheaper than enzymes. In addition, the enzymatically hydrolysis process takes several days while chemical hydrolysis requires relatively faster time [1]. Alternatively, dilute acids can be used to break the polymer chains of cellulose and hemicellulose. In the acid hydrolysis method, lignocellulose biomass is exposed to the acid at a given temperature and pressure for a certain time and produces a sugar monomer of cellulose and hemicellulose polymers. Sulphuric acid is the most widely studied acid and is used for acid hydrolysis [1]. Cellulose can be chemically hydrolysed using H<sub>2</sub>SO<sub>4</sub> solution.



One indicator of the pretreatment effectiveness is the reduction of lignin during process. Pretreatment of the biomass results in the degradation of cellulose and hemicellulose. Effect of hydrothermal pretreatment of date palm leaflets and vine shoots on lignin was investigated by other researchers [2,3]. However they used a high temperature condition. Chemical pre-treatment using liquid ammonia and aqueous sodium hydroxide (NaOH) are recognized to change the cellulose crystalline structure and effect on enzymatic digestibility [4]. Previous authors [5], performed NaOH-dilute acid pretreatment to remove lignin in rice husk. The use of sulphuric acid is also required in the neutralization process which produces very large amounts of gypsum or lime waste and can pollute the environment [1]. Ammonia pretreatment eliminates lignin by cutting C–O–C bonds in lignin [6]. The effect of aqueous ammonia and dilute acid, on the chemical compositions, cellulose crystallinity, morphologic change, and enzymatic hydrolysis of bamboo was studied by previous researcher [7]. Therefore, this research focused on the soaking in aqueous ammonia (SAA) pretreatment and dilutes sulphuric acid of rice husk. Furthermore, hydrolysis was carried out using sulphuric acid ( $H_2SO_4$ ) with various concentrations. The hydrolysis reaction between water and cellulose was catalysed by acid so that the reaction proceeded quickly and produced faster glucose.

Some studies have tried to describe the hydrodynamic phenomena in pretreatment [8] and enzymatically hydrolysis process [9–11] by using a design and CFD modelling. However this work used a different kind of reactor which is a laboratory spherical reactor. A perfect mixing process was required to obtain optimal glucose levels. Therefore, this study applied CFD modelling (ANSYS Fluent 16) to produce hydrodynamic mixing process in acid hydrolysis.

## 2. Materials and methods

### 2.1. Raw Material Preparation

The rice husk was dried under the sun for  $\pm 1$  day. Its size was reduced to 35 meshes. Then, the composition of cellulose, hemicellulose and lignin was analysed by using a Chesson method [12]. Scanning electron microscope (SEM) micrographs of cross section rice husk of were carried out by a SEM-EDX microscope Phenom Pro X.

### 2.2. Pretreatment Raw materials

Dried rice husk of about 40 g was put into an erlenmeyer of 300 ml and soaked in 15 % (w/v) aqueous ammonia of 160 ml at 600 °C for 5 h. The filtrate was separated from the residue by a vacuum pump. Then the residue was washed with distilled water. Pretreated sample was fed to a glass bottle, and then added 135 ml of  $H_2SO_4$  (0.18 N). It was stirred evenly for 1 min. Then it was heated in the oven at 160 °C for 35 min. It was cooled in room temperature and filtered by vacuum pump. Pretreated sample was analysed by using a Chesson method [12]. Scanning electron microscope (SEM) micrographs of cross section pretreated rice husk were carried out by a SEM-EDX microscope Phenom Pro X.

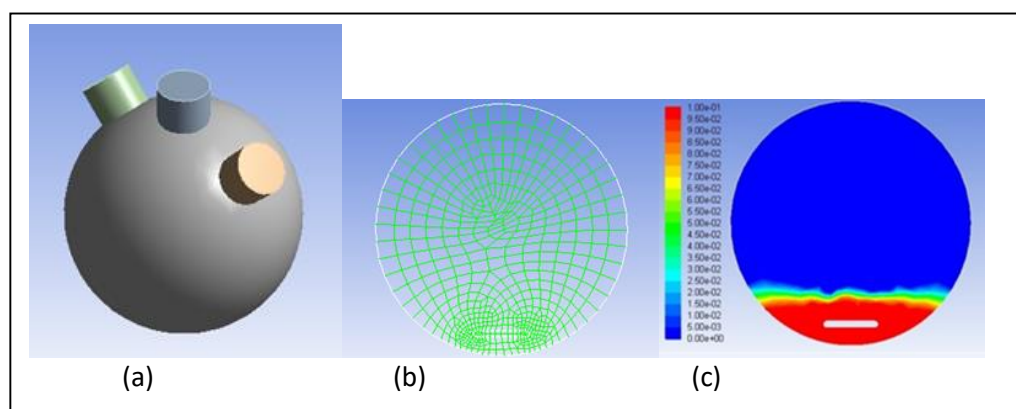
### 2.3. Hydrolysis process

20 g of pretreated sample and 200 ml of  $H_2SO_4$  (1N) were inserted into three neck flask. They were hydrolysed at a temperature of 100 °C, a speed of 200 rpm for 30 min. Repetition was performed at variable  $H_2SO_4$  of 2 N, and 3 N for 60 to 150 min. The contents of glucose were determined using the standard procedures according to Luff-Schoorl [13].

### 2.4. Numerical CFD Simulation

CFD modelling of Hydrodynamic of Hydrolysis of pretreated rice husk was referred by previous authors [14,15]. ANSYS Fluent 16 with a finite volume method was used to solve the general partial differential equations that illustrate fluid flow, momentum and mass transport. The geometry of hydrolysis reactor was shown in figure 1a and 1b. The reactor had a diameter of 0.2 m. Initially, a pretreated sample was put at the reactor bottom to a level above the impeller, and then sulphuric acid was injected to the rest of the reactor. The volume fraction of 10 % of pretreated rice husk in the slurry entered to the reactor

of, with the average diameter of 50  $\mu\text{m}$ . The reactor had the temperature of 100  $^{\circ}\text{C}$  for 30 min and speed of 200 rpm. A reactor schematic and the initial position of slurry solution were described in figure 1c. There were three fluid zones within the domain. The first zone represented the impeller zone; the region where the cellulose slurry was initially posited; the rest of reactor where  $\text{H}_2\text{SO}_4$  solution was fed. A calculation was started by using the initial assumption similar to the specified volume fractions. The pretreatment process was simulated for various hydrolysis time for 60 to 150 min. A pretreated rice husk entered the reactor in the form of slurry. The simulation was operated in unsteady state conditions with time step sizes equal to 0.1 s. The Eulerian-Eulerian Multiphase flow and standard  $k-\epsilon$  model was applied in all cases for accounting the turbulence effects. Hydrodynamic analysis of the hydrolysis reactor was obtained from 5 unsteady state simulations (30, 60, 90, 120, and 150 min) at different sulphuric acid concentrations of 1N, 2N and 3 N.



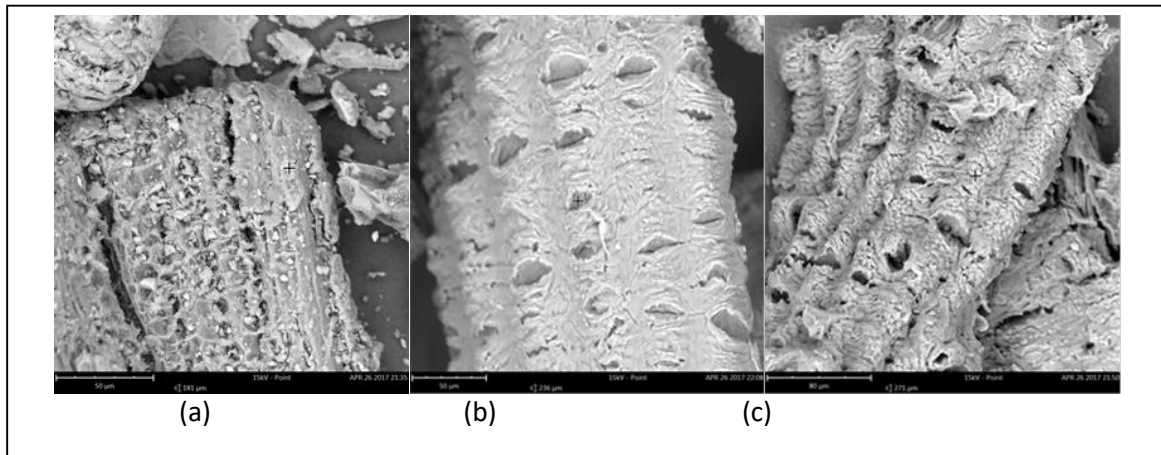
**Figure 1.** (a) 3-Dimensional geometry of reactor; (b) 2-Dimensional geometry of reactor; (c) initial condition of slurry.

### 3. Results and discussion

#### 3.1. Morphological of the Rice Husk Structure

To ensure the successful of SAA-dilute acid pretreatment process, it is crucial to see a morphological of the rice husk structure. Chemically pretreated rice husk was analysed by SEM to investigate the change in rice husk before and after the pretreatment. Figure 2 illustrates the morphology of the rice husk structure before pretreatment (a), after SAA (b), after SAA-acid pretreatment (c). It is observed that a morphological of rice husk (figure 2a) changed to fibrous clusters (figure 2b). It is described by SEM that there was a changing in a microstructure of rice husk. This is due to more lignin was removed by SAA pretreatment. Significant lignin was also reduced after SAA-acid pretreatment (figure 2c). It can be concluded that the pretreatment of rice husk led it much more porous to be hydrolysed. The elimination of lignin and changing of its structure enhanced the degradation of cellulose and hemicellulose from biomass. In the SAA-acid pretreatment, a major part of the amorphous material (xylan and lignin) was reduced. Meanwhile, either a small part of the crystalline material (glucan) was eliminated or the crystalline structure was destroyed by swelling.

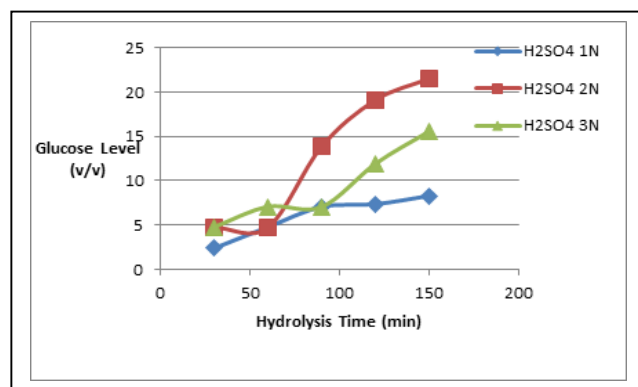




**Figure 2.** The morphology of the rice husk structure: (a) before pretreatment; (b) after SAA pretreatment; (c) after SAA-acid pretreatment. Magnification of images given in figure 2a, 2b and 2c are 1000X, 2000X and 3000X.

### 3.2. Effect of Hydrolysis Time on Glucose Levels at Various Concentrations of $H_2SO_4$

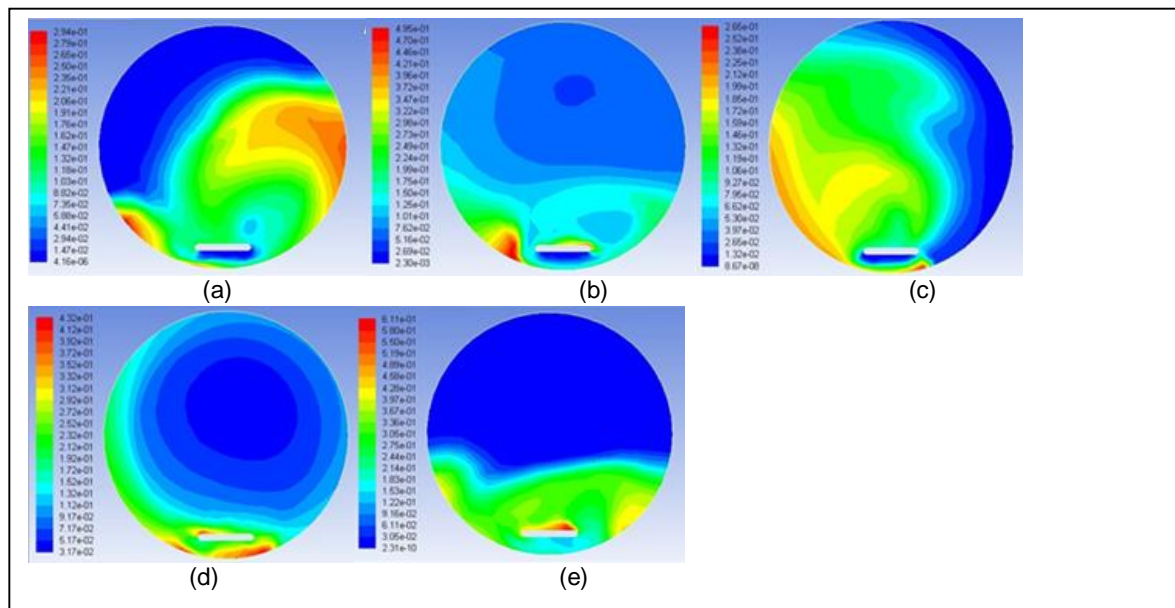
Figure 3 describes the effect of hydrolysis time on glucose levels at Various Concentrations of  $H_2SO_4$ . It is shown that the longer hydrolysis times produced the higher of glucose level. It illustrates that the length of reaction time between cellulose and water affected the amount of glucose. This is due to the longer of the reaction time caused more collisions between cellulose and water. In this study the optimum hydrolysis reaction time is 150 min. The higher concentration of acids enhanced the activation energy. The highest glucose level was about 21.51% at the  $H_2SO_4$  concentration of 3 N for 150 min.



**Figure 3.** Effect of hydrolysis time on glucose levels at various concentrations of  $H_2SO_4$ .

### 3.3. Hydrodynamic Analysis of Cellulose Volume Fraction during Hydrolysis at $H_2SO_4$ Concentration of 1N

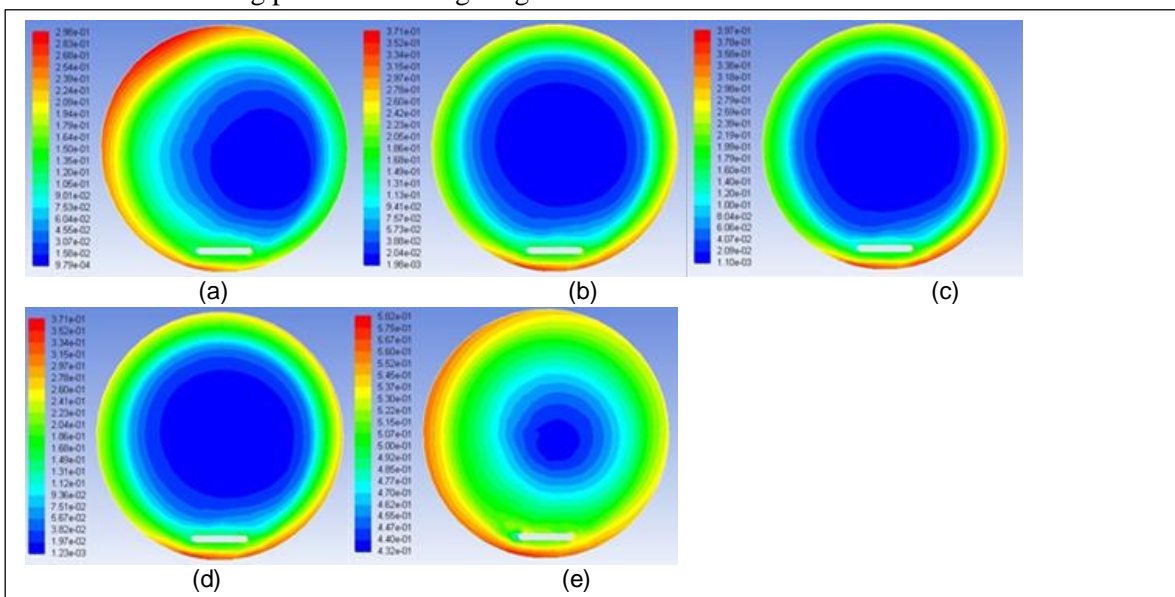
Figure 4 shows cellulose volume fraction during hydrolysis at  $H_2SO_4$  concentration of 1N. It presents the volume fraction of cellulose, predicted by the CFD simulation of this research. The red colour indicates the high volume fraction of cellulose. The blue colour shows the low volume fraction of cellulose. The distribution of cellulose volume fraction was heterogeneous and mainly distributed about the middle. This non homogeneity was caused by the circular liquid flow under the solid-liquid interaction.



**Figure 4.** Hydrodynamic analysis of cellulose volume fraction during hydrolysis process at  $H_2SO_4$  1N: (a) 30 min; (b) 60 min; (c) 90 min; (d) 120 min; (e) 150 min.

### 3.4. Hydrodynamic Analysis of Cellulose Volume Fraction during Hydrolysis at $H_2SO_4$ Concentration of 2N

Figure 5 describes the pattern of mixing motion in cellulose hydrolysis process at the  $H_2SO_4$  concentration of 2 N. The predicted cellulose volume fraction distribution illustrates a higher cellulose concentration near the wall and absence of cellulose at the central of reactor. The benefit of the static mixers is that they avoid sedimentation of solids through modifications in reactor geometry. It is shown that the perfect mixing patterns tended to be constant. This is due to the mixing affected the hydrolysis process. Perfect mixing produced the higher glucose.

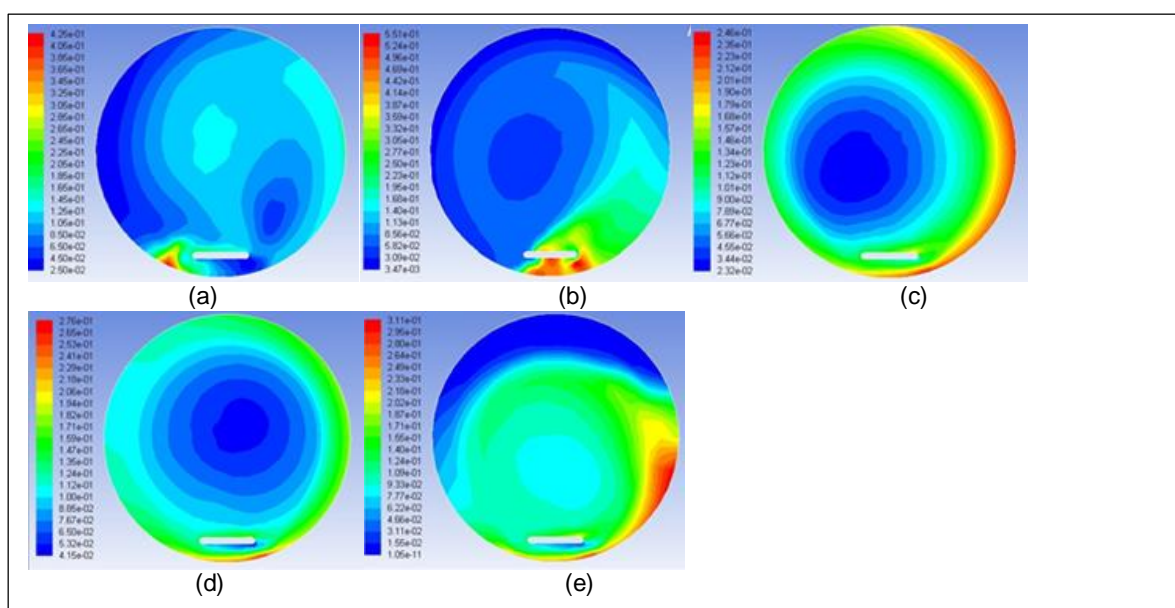


**Figure 5.** Hydrodynamic analysis of cellulose volume fraction during hydrolysis process at  $H_2SO_4$  2N: (a) 30 min; (b) 60 min; (c) 90 min; (d) 120 min; (e) 150 min.

It can be seen from figure 5 that the longer time of hydrolysis time led more cellulose degrades to glucose. It is reasonable with the experimental test. It can be concluded that the optimum condition of hydrolysis process was obtained at  $\text{H}_2\text{SO}_4$  concentration of 2N for 150 min.

### 3.5. Hydrodynamic Analysis of Cellulose Volume Fraction during Hydrolysis at $\text{H}_2\text{SO}_4$ Concentration of 3N

Figure 6 presents the flow field of cellulose volume fraction at the  $\text{H}_2\text{SO}_4$  concentration of 3N. The distribution of cellulose volume fraction was heterogeneous and mainly distributed about the middle. This non homogeneity was caused by the circular liquid flow under the solid-liquid interaction.



**Figure 6.** Hydrodynamic Analysis of Cellulose Volume Fraction during Hydrolysis Process at  $\text{H}_2\text{SO}_4$  3N: (a) 30 min; (b) 60 min; (c) 90 min; (d) 120 min; (e) 150 min.

## 4. Conclusion

It can be concluded that a morphological of rice husk changed to fibrous clusters after SAA pretreatment. Significant lignin was also removed after SAA-dilute acid pretreatment. The pretreatment of rice husk led it much more porous to be hydrolyzed. The longer hydrolysis time of rice husk cellulose increased glucose level significantly. The optimum concentration of  $\text{H}_2\text{SO}_4$  produced the highest glucose level of 21.51% at  $\text{H}_2\text{SO}_4$  concentration of 2N for 150 min. Through hydrodynamic analysis with CFD modeling, cellulose hydrolysis with  $\text{H}_2\text{SO}_4$  concentration of 2N for 150 min had the best mixing distribution pattern for almost every time variable. It is reasonable with the experimental test. The model and results presented in this study are valuable for the application of CFD model to calculate the flow of biomass suspensions in spherical reactors.

## 5. References

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# CERTIFICATE OF APPRECIATION

THIS CERTIFICATE IS PROUDLY PRESENTED TO

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