

## Effect of Dilute Acid - Alkaline Pretreatment on Rice Husk Composition and Hydrodynamic Modeling with CFD

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### Abstract

The high cellulosic content of rice husk can be utilized as a feedstock for pulp and biofuel. Pretreatment is necessary to break the bonds in the complex lignocellulose matrices addressing the cellulose access. This work aims to utilize the rice husk using dilute acid and alkaline pretreatment experimentally and CFD modeling. The study consists of three series of research. The first stage was the dilute acid pretreatment with sulfuric acid concentration of 1% to 5% (v/v) at 85°C for 60 minutes, and alkaline pretreatment with NaOH concentration of 1% to 5% (w/v) at 85°C for 30 minutes separately. The second stage used the combination of both pretreatment. Moreover the last stage of research was hydrodynamic modeling of pretreatment process by CFD (ANSYS FLUENT 16). The experimental results showed that the lowest lignin content after acid pretreatment was about 10.74%. Alkaline pretreatment produced the lowest lignin content of 4.35%. The highest cellulose content was 66.75 % for acid-alkaline pretreatment. The lowest content of lignin was about 6.09% for acid-alkaline pretreatment. The lowest performance of alkaline pretreatment on HWS (hot water solubility) of about 7.34% can be enhanced to 9.71% by using a combination alkaline-acid. The combined pretreatments result hemicellulose of about 9.59% (alkaline-acid) and 9.27% (acid-alkaline). Modeling results showed that the mixing area had the minimum pressure of about -6250 Pa which is vortex leading minimum efficiency of mixing. The rice husk flowed upward to the upper level and mixed with reagent in the perfect mixing.

### Keywords

Alkaline Pretreatment, CFD Modeling, Dilute Acid pretreatment, Lignin, Rice Husk

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## 1. INTRODUCTION

Programs on energy utilization have been sustained by finding other kinds of renewable energy. Several studies relating to the conversion of lignocellulose biomass as a raw material to produce bioethanol have been implemented (Chen et al., 2011; Hermansyah et al., 2015; Karimi et al., 2006; Novia et al., 2017; Sudiyani et al., 2017; Xavier et al., 2010).

The conversion of bioethanol is still challenging. The lignin component in lignocellulose biomass must be eliminated in order to enable the digestibility of enzymatic hydrolysis. Pretreatment is the most important step to reduce lignin and hemicellulose, to decrease the crystallinity of cellulose, and to improve the materials porosity (Karimi et al., 2006). It is needed to change the structure of cellulosic biomass, making the cellulose more porous to enzymes which can alter the polymers of carbohydrate into fermentable sugars (Mosier, 2005). After pretreatment, cellulose and hemicellulose are hydrolyzed to monomeric sugars. The pretreatment needs higher cost, so

the best pretreatment can reduce the amount of chemicals for hydrolyzed. Pretreatment also enhance the glucose yield. Unpretreated samples gives lower than 20 %, using the pretreatment main process the glucose yield to about 90% (Hamelinck et al., 2005).

Methods for the pretreatment of lignocellulose biomass to increase the hydrolysis have been studied: steam explosion and alkaline peroxide (Cara, 2006; Fang et al., 2010; Ohgren et al., 2007); alkaline pretreatment (Harun et al., 2011; McIntosh and Vancov, 2010); and hydrothermal pretreatment (Kaparaju and Felby, 2010; Kumar et al., 2011; Thomsen et al., 2008). These technologies use extreme operating conditions of reaction with a greatly capital cost, highly processing fees and large investment risks (Li et al., 2009). Several pretreatment methods create strong acidic, poisonous materials in the preserved lignocellulose and inhibit mixtures for hydrolysis and following fermentation of ethanol. In addition, alkaline pretreatment was demonstrated to be more suitable on agricultural residues

than on materials of wood (Tahezadeh et al., 2008). It also selectively reduces the lignin portion. Pretreatment of alkali apply at lower temperatures and pressures compared to other pretreatment methods (Mosier, 2005). The lime pretreatment process uses slurring the lime with water, showering it onto the biomass material and keeping the material for a particular duration, generally from days to weeks (Mosier, 2005). Ammonia has been usually used in alkali pretreatments; its use produces many environmental problems due to the recovering processes that must also be carried out along with this pretreatment. Reducing hemicellulose and lignin content could highly increase enzyme digestibility by enhancing the enzyme accessibility to cellulose. Pretreatment with dilute sulfuric acid followed by aqueous ammonia would then be required for the effective hydrolysis and lignin separation. With the benefits of dilute acid and alkaline method, this work investigated the combination of dilute acid-alkaline pretreatment to eliminate the lignin component in rice husk. Pretreatment is a high cost process to allow lignocellulose biomass to be utilized to become an alternative fuels. However it has the possibility to increase the effectiveness and decreasing of the fee through more study and advance.

Hydrodynamic simulation of the alkaline-dilute acid pretreatment was performed using the computational fluid dynamics (CFD) modeling (ANSYS FLUENT 16). The fundamental processes that occur in the pretreatment process have been investigated. Numerous researches (Novia et al., 2007, 2017; Cundari, 2016) are being applied to advance the computational methods appropriate for pairing the many crucial characteristics of chemistry and physics in a procedure that is effective enough for explaining the pretreatment problems. CFD modeling can offer a huge scope of information for the design of pretreatment reactor. The use of CFD modeling can therefore decrease the cost of time-consuming experimental research.

## 2. EXPERIMENTAL SECTION

### 2.1 Materials

The rice husk samples were obtained from nearby (South Sumatera). They were stored in plastic bags at 50C, after determination of moisture content. The rice husks were collected from the location of Inderalaya, South Sumatera province, Indonesia. Then they were air-dried and kept at room temperature. They were crushed and put through a sieve tray (less than 0.8 mm).

### 2.2 Methods

#### 2.2.1 Experimental Method of Dilute Acid Pretreatment

Lignocellulose rice husks were pretreated by the following procedure. Each dry rice husk, of about 30 g, was fed into a stirred reactor of 500 mL and blended with 150 ml of sulfuric acid (H<sub>2</sub>SO<sub>4</sub>) with various concentration of 1% to 5% (v/v). The mixture was kept warm in a rotary air bath at a speed of 500 rpm and temperature of 85°C for 30 min. The mixture was inserted in an autoclave at a temperature of 121°C for 30 min. The mixture was then put into 1L of pre-cold acetone and

blended carefully. The slurry was separated using centrifuge at a speed of 8000 rpm. After 10 min of time, the filtrate was gathered. The residue remains was rinsed in 1L of water and separated. The component of lignin before and after dilute-acid pretreatment was analyzed by a standard method of Chesson (Datta 1981). Scanning electron microscope (SEM) micrographs of cross section rice husk before and after pretreatment were taken using SEM-EDX microscope Phenom Pro X.

#### 2.2.2 Experimental Method of Dilute Acid-Alkaline Pretreatment

Dilute-acid pretreated samples were followed by alkaline pretreatment to improve the removal percentage of lignin content in the samples

#### 2.2.3 Experimental Method of Alkaline-Dilute Acid Pretreatment

Alkaline pretreated samples were followed by dilute-acid pretreatment to reduce percentage of lignin content in the samples

#### 2.2.4 Modeling of Dilute Acid-Alkaline Pretreatment by Using CFD

The CFD simulations were carried out by the Euler-Granular multiphase model available in the commercial CFD software (ANSYS FLUENT 16.0 Documentation 2015). Simplification assumption is each phase coincides at every grid in domain in the form of interpenetrating continua.

The mass conservation of phase *i* (*i* = fluid, solid):

$$\frac{\partial}{\partial t}(\rho_i \varepsilon_i) + \nabla \cdot (\rho_i \varepsilon_i U_i) = r_i \tag{1}$$

where,  $\varepsilon_f + \varepsilon_s = 1$

The conservation of momentum of phase *i* (*i* = fluid, *k* = solid, *k* ≠ *i*):

$$\frac{\partial}{\partial t}(\rho_i \varepsilon_i U_i) + \nabla \cdot (\rho_i \varepsilon_i U_i U_i) = -\varepsilon_i \nabla P + \nabla \cdot \tau_i + \rho_i \varepsilon_i g - \beta(U_i - U_k) \tag{2}$$

The conservation of energy for phase *i* gives:

$$\frac{\partial}{\partial t}(\varepsilon_i \rho_i H_i) + \nabla \cdot (\varepsilon_i \rho_i U_i H_i) = -\varepsilon_i \frac{\partial P_i}{\partial t} + \tau_i : \nabla U_i - \nabla q_i + S_i \tag{3}$$

with,

$$H_i = \sum_i \varepsilon_i \int_{T_{ref}}^T C_{p_i} dT \tag{4}$$

For the drag force formulation, we use the Syamlal-O'Brian model (ANSYS FLUENT 16.0 Documentation 2015):

$$\beta = \frac{3}{4} C_D \frac{\varepsilon_s \varepsilon_g \rho_g}{v_{r,s}^2 d_s} \left( \frac{Re_s}{v_{r,s}} \right) |U_s - U_g| \tag{5}$$

The drag coefficient,  $C_D$  is given by:

$$C_D = \left( 0.63 + \frac{4.8}{\sqrt{\frac{Re_s}{v_{r,s}}}} \right)^2 \quad (6)$$

$$Re_s = \frac{\rho_g d_s |U_s - U_g|}{\mu g} \quad (7)$$

The terminal velocity correlation for the solid phase:

$$v_{r,s} = 0.5A - 0.03Re_s + 0.5\sqrt{(0.06Re_s)^2 + 0.12Re_s(2B - A) + A^2} \quad (8)$$

where,

$$A = \varepsilon_g^{4.14} \quad (9)$$

$$B = 0.8\varepsilon_g^{1.28} \text{ for } \varepsilon_g \leq 0.85 \quad (10)$$

$$B = 0.8\varepsilon_g^{2.65} \text{ for } \varepsilon_g > 0.85 \quad (11)$$

Solid Pressure. The solids phase pressure ( $P_s$ ) contains a kinetic term and the particle collisions term.

$$P_s = (1 + 2(1 + e_s)e_s g_o)\varepsilon_s \rho_s \Theta_s = \rho_s \varepsilon_s \Theta_s + 2g_o \rho_s \varepsilon_s^2 \Theta_s (1 + e_s) \quad (12)$$

where  $\Theta_s$  is the granular temperature,  $e_s$  is the coefficient of restitution for particle collisions. The radial distribution function,  $g_o$ , is defined by:

$$g_o = \left[ 1 - \left( \frac{\varepsilon_s}{\varepsilon_{s,max}} \right)^{\frac{1}{3}} \right]^{-1} \quad (13)$$

Solids Shear Stress. The solids stress tensor contains bulk and shear viscosities. The solid phase bulk viscosity are expressed as:

$$\mu_b = \frac{4}{3}\varepsilon_s \rho_s d_s g_o (1 + e_s) \left( \frac{\Theta_s}{\pi} \right)^{\frac{1}{2}} \quad (14)$$

The solids phase shear viscosity is given by:

$$\mu_b = \frac{2\mu_{s,dil}}{(1 + e)g_o} \left[ 1 + \frac{4}{5}(1 + e_s)g_o \varepsilon_s \right]^2 + \frac{4}{5}\varepsilon_s \rho_s d_s g_o (1 + e_s) \left( \frac{\Theta_s}{\pi} \right)^{\frac{1}{2}} \quad (15)$$

The solid phase dilute viscosity is:

$$\mu_{s,dil} = \frac{5}{16}\rho_s \varepsilon_s l_s \sqrt{2\pi\Theta_s} \quad (16)$$

where is,

$$l_s = \frac{\sqrt{2} d_s}{12 \varepsilon_s} \quad (17)$$

The granular temperature,  $\Theta_s$ , is calculated by solving the turbulent kinetic energy equation for solid phase:

$$\frac{3}{2} \frac{\partial}{\partial t} (\rho_s \varepsilon_s \Theta_s) + \nabla \cdot (\rho_s \varepsilon_s U_s \Theta_s) = T_s : \nabla U_s + \nabla \cdot (k_{\Theta_s} \nabla \Theta_s) - \gamma_s \quad (18)$$

The diffusion coefficient for granular energy  $k_{\Theta_s}$  is given by:

$$k_{\Theta_s} = \frac{2k_{\Theta_s,dil}}{(1 + e_s)g_o} \left( 1 + \frac{6}{5}(1 + e_s)g_o \varepsilon_s \right)^2 + 2\varepsilon_s^2 \rho_s d_s g_o (1 + e_s) \left( \frac{\Theta_s}{\pi} \right)^{\frac{1}{2}} \quad (19)$$

where:

$$k_{\Theta_s,dil} = \frac{75}{64} \rho_s \varepsilon_s l_s \sqrt{2\pi\Theta_s} \quad (20)$$

The collisional energy dissipation,  $\gamma_s$ , is calculated by:

$$\gamma_s = 3 \left( 1 - e_s^2 \right) \varepsilon_s^2 \rho_s g_o \Theta_s \left[ \frac{4}{d_s} \left( \frac{\Theta_s}{\pi} \right)^{\frac{1}{2}} - \nabla U_s \right] \quad (21)$$

Generally, the mixer is below turbulent flow conditions. Therefore, it is crucial to apply the suitable turbulence model to illustrate the effect of turbulent fluctuations of velocities and scalar variables for the basic conservation equations. A  $k-\varepsilon$  model was used to show the turbulent motions in both phases. In the  $k-\varepsilon$  model, the turbulent viscosity is given by:

$$\mu_{t,i}(t) = \rho_i \varepsilon_i C_{\mu} \frac{k_i^2}{\varepsilon_i} \quad (22)$$

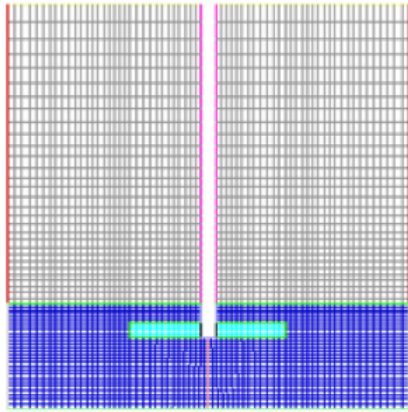
The turbulence kinetic energy,  $k$ , and its rate of dissipation,  $\varepsilon$ , can be determined from the transport equations:

$$\frac{\partial}{\partial t} (\rho_i \varepsilon_i k_i) + \nabla \cdot (\rho_i \varepsilon_i k_i U_i) = \nabla \cdot \left( \varepsilon_i \frac{\mu_t}{\sigma_k} \nabla k_i \right) + (\varepsilon_i G_k - \varepsilon_i \rho_i \varepsilon_i) \quad (23)$$

$$\frac{\partial}{\partial t} (\rho_i \varepsilon_i \varepsilon_i) + \nabla \cdot (\rho_i \varepsilon_i \varepsilon_i U_i) = \nabla \cdot \left( \varepsilon_i \frac{\mu_t}{\sigma_k} \nabla \varepsilon_i \right) + \frac{\varepsilon_i}{k} (C_{1\varepsilon} \varepsilon_i G_k - C_{2\varepsilon} \varepsilon_i \rho_i \varepsilon_i) \quad (24)$$

### 2.2.5 Numerical Procedure

The geometry of pretreatment reactor is shown in Figure 1. The numerical calculation used a two-dimensional mesh. The reactor had a diameter of 0.086 m and the height of 0.086 m. To decrease the computational effort, and due to the reactor is symmetric, the reactor was split to become a half part. Initially, the acid-dilute pretreated samples settled at the reactor bottom to a level above the impeller, then sulfuric acid was injected to the rest of the reactor. The volume fraction of rice husk pretreated in the slurry entered to the reactor of 56 %, with



**Figure 1.** Two-dimensional geometry of pretreatment reactor

the average diameter of 60  $\mu\text{m}$ . The operating conditions of reactor were the temperature of 85°C and the pressure of 1 atm. A schematic of the reactor and the initial position of slurry solution are shown in Figure 1. Within the domain, there are three fluid zones, representing the impeller zone (green colours), the region where the slurry is initially located (blue colours) and the rest of tank where NaOH solution is filled (grey colours). The simulations were started using the initial assumptions similar to the specified volume fractions. The pretreatment process was simulated for 60 min. A constant time step of 0.01 was used. The simplification assumption is pretreated rice husk entered the reactor in the form of a slurry.

### 3. RESULTS AND DISCUSSION

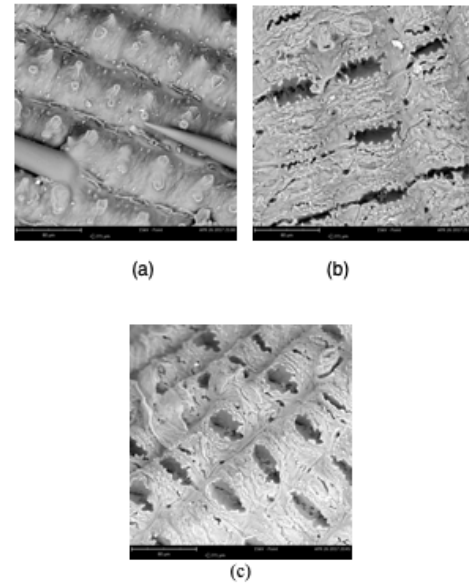
#### 3.1 Morphological of the Rice Husk Structure

Figure 2 shows the morphology of the rice husk structure (before pretreatment), b (after alkaline pretreatment), c (after alkaline-acid pretreatment). Major morphological changes of the rice husk (fig 2a) converted to fibrous clusters (fig 2b) were detected. It was shown by SEM that alterations in microstructure occurred. This is due to some lignin being eliminated by alkaline pretreatment. Lignin was also removed significantly during the alkaline-acid pretreatment (fig 2c). Thus the pretreatment of rice husk made it much more porous to enzymes. Cellulose and hemicellulose degradation occurred because of the reduction of lignin content and the alteration of its structural.

#### 3.2 Effect of the Combination of Alkaline and Acid Pretreatment on the rice husk composition

#### 3.3 Effect of Single Pretreatment on the Lignin Content of Rice Husk

Lignin is the main problem for the utilization of second generation bioethanol. The lignocellulose wall lets as an inhibitor reducing the accessibility of cellulose to be hydrolysed. The lignin content must be reduced as much as possible to provide a high efficiency of pretreatment. As shown in Fig 3, Initially,

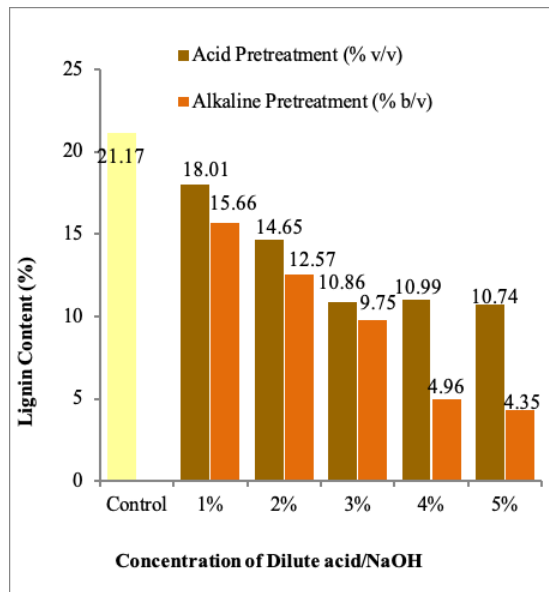


**Figure 2.** Morphology of the rice husk structure before pretreatment (a), after alkaline pretreatment (b), after alkaline-acid pretreatment (c)

the lignin content of rice husk was about 21.17%. After acid pretreatment, the lowest lignin content was 10.74%. Alkaline pretreatment produced the lowest lignin content of 4.35%. Fig 3 also illustrated that the higher concentration of either  $\text{H}_2\text{SO}_4$  or NaOH caused the increasing of lignin degradation. The highest capability of lignin degradation by acid pretreatment was about 38.38 %. Meanwhile, the alkaline pretreatment reduced the highest lignin content was about 55.34%. It can be concluded that  $\text{H}_2\text{SO}_4$  gave minor effect on lignin degradation level. On the other hand, NaOH lead the major effect on the degradation of lignin. Alkaline pretreatment broke down the lignin and glycoside bond on polysaccharide, so it can decrease the crystallinity. Previous researcher studied the lowest of lignin content is about 3.46 % for solids pretreated at the acid concentration of 2% and 90 minutes of pretreatment time.

The lowest performance of alkaline pretreatment on a HWS (hot water solubility) of about 7.34% can be enhanced to become 9.71% by using combination alkaline-acid. HWS content was about 10.39%. HWS content for both pretreatment was higher than acid pretreatment. This is due to  $\text{H}_2\text{SO}_4$  formed emulsion at acid pretreatment. Meanwhile at the combination pretreatment, before  $\text{H}_2\text{SO}_4$  form more emulsions, NaOH bonded with  $\text{H}_2\text{SO}_4$  each other to become  $\text{NaSO}_4$  which is soluble in hot water. Fig 4 shows the both pretreatment combination degraded hemicellulose of about 9.59% (alkaline-acid) and 9.27% (acid-alkaline). The highest cellulose content was about 66.75 % for acid-alkaline pretreatment. For pretreatment combination, the lowest content of lignin was about 6.09% for acid-alkaline pretreatment.

It can be concluded that combination of either alkaline-acid or acid-alkaline had similar capability in increasing HWS, the

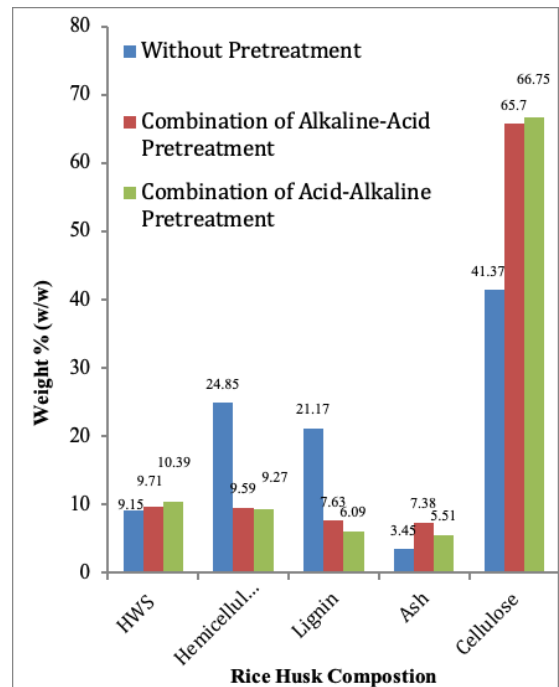


**Figure 3.** Effect of the Pretreatment on the Lignin Content of Rice Husk

degradation of hemicellulose, the enhancing of cellulose and the degradation of lignin. The difference of both pretreatment was about  $\pm 1\%$ . Globally, acid-alkaline pretreatment was better than alkaline-acid pretreatment.

**3.4 Hydrodynamic Analysis of Pressure and Volume Fraction on Acid-Alkaline Pretreatment Using CFD (Ansys Fluent 16)**

To compare the simulation result and experimental data, this study focuses on hydrodynamics of acid-alkaline pretreatment. Figure 5a shows the volume fraction of untreated rice husk. After pretreatment, the volume fraction of rice husk and reagent changed due to the mixing process. As illustrated in fig 5c and 5d, the impeller action caused the reagent in lower concentration (the blue color) and mixed with rice husk (red one). Furthermore, the rice husk at the lower level flowed to the upper one and mixed with reagent in the perfect mixing. The volume fraction of rice husk reduced because of mixing between rice husk and reagent solution. Figure 5b describes the distribution of pressure after pretreatment. The blue color represents hydrostatic pressure normally; the highest pressure is represented by red color. Increasing of the pressure caused an enhancing of the higher kinetic energy turbulent which is desired in mixing of the rice husk and the reagent. We can see from the figure the small amount of blue color above the impeller. This area had a minimum pressure of about -628 Pa which is a vortex leading the minimum mixing efficiency. The highest pressure was about 8570 Pa (red color) where the maximal mixing occurred.

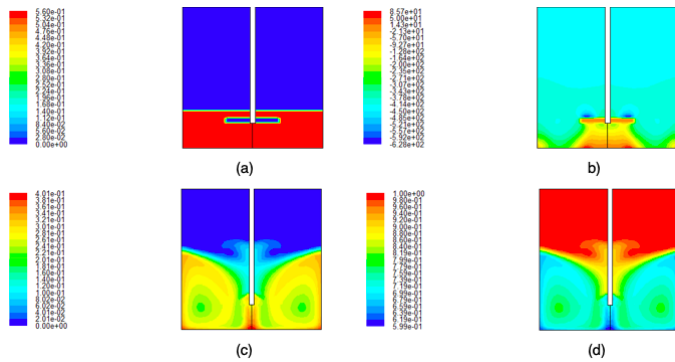


**Figure 4.** Effect of Different Pretreatment on the Composition of Rice Husk

**4. CONCLUSIONS**

A SEM-EDX investigation recorded that alterations in microstructure were observed, since a huge fraction of lignin was reduced by alkaline pretreatment. More lignin was also removed in the combined alkaline-acid pretreatment. After acid pretreatment, the lowest lignin content was 10.74%. Alkaline pretreatment produced the lowest lignin content of 4.35%. The highest capability of lignin degradation by acid pretreatment was about 38.38 %. Meanwhile, alkaline pretreatment reduced the highest lignin content to about 55.34%.

The lower performance of alkaline pretreatment on HWS (hot water solubility) of about 7.34% can be enhanced to 9.71% using combined alkaline-acid. Both pretreatment combinations degraded hemicellulose of about 9.59% (alkaline-acid) and 9.27% (acid-alkaline). The highest cellulose content was about 66.75 % for acid-alkaline pretreatment. The lowest content of lignin was about 6.09% for acid-alkaline pretreatment. It can be concluded that either the alkaline-acid or the acid-alkaline combination had similar capability in increasing HWS, the degradation of hemicellulose, the enhancing of cellulose and the degradation of lignin. The mixing area had the minimum pressure of about -628 Pa which is vortex leading the minimum mixing efficiency. The highest pressure was about 8570 Pa (red color) where the maximal mixing occurred. The rice husk flowed upward to the upper level and mixed with reagent in the perfect mixing.



**Figure 5.** Initial Volume Fraction of Rice Husk (a); Contour Pressure (b); Volume Fraction of Rice Husk after Pretreatment (c); Volume Fraction of NaOH after Pretreatment (d)

## 5. ACKNOWLEDGEMENT

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