FLOODING PHENOMENA IN TWO IMMISCIBLE LIQUIDS SYSTEM: CONCEPTUAL APPROACH

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ABSTRACT

The use of the liquid separating equilibrium has recently become favourable as the temperature window for the biodiesel synthesis does not require reaching the distillation thermodynamic state. This study is mainly to characterize the flooding properties in liquid-liquid system of palm oil and ethanol. The key parameters, the Sauter diameter of ethanol and oil droplet in the excess oil or ethanol phase, coalescence factor of excess volume in various ratios, and hydrodynamics in cylindrical viscometer carried on. Firstly, to measure the Sauter diameter of ethanol in excess oil done in laboratory by self-modified syringe tube by releasing droplet form the bottom of measuring cylinder. For the second experiment, several ratios of oil to ethanol prepared mixed vigorously for 20 minutes at 45^0C. The time for settlement and height of two layers formed recorded. Finally for the third experiment, several ratios of oil to ethanol prepared and mixed vigorously for 20 minutes at 45^0C and the mixture tested for the viscosity, shear rate and rpm in viscometer. Average, 0.002m Sauter diameter of ethanol and oil droplet measured at $7.29\text{x}10^{-3}$ m/s and 0.73 m/s of velocity, suggesting that the aperture of the plate would be at least 0.005m if either ethanol or oil to be the dispersed phase. In the meantime, the result of liquids mixing shows that the solubility of ethanol increased proportionally with the volume of ethanol at 45^0C. At low concentration of ethanol, however, the slope decreased due to the formation of emulsion phase. The key parameters that influences the flooding in the hydrodynamics are viscosity and agitation speed. To the flooding point of view, low viscosity would make the flow of the liquid flow easier. The increase of agitation speed increases the chance of flooding in the viscometer.

FENOMENA BANJIR DALAM SISTEM DUA CECAIR YANG TIDAK BERCAMPUR: KONSEP PENDEKATAN

ABSTRAK

Baru-baru ini penggunaan keseimbangan dalam memisahkan cecair telah menjadi sasaran kerana suhu untuk menghasilkan biodiesel tidak perlu mencapai keadaan termodinamik stabil. Terutamanya, kajian ini adalah untuk mencirikan sifat banjir dalam sistem cecair-cecair minyak dan etanol. Parameter utama yang dikaji adalah diameter Sauter etanol dan minyak, factor tautan jumlah yang berlebihan dalam nisbah yang pelbagai dan kajian hidrodinamik dalam viscometer. Pertama, untuk mengukur diameter Sauter etanol dalam minyak berlebihan yang dilakukan di makmal dengan tiub yang diubah suai untuk megukur titisan etanol. Bagi eksperimen kedua, beberapa nisbah minyak kepada etanol disediakan bercampur selama 20 minit pada suhu 45^{0}C. Masa untuk membentuk dua lapisan dan ketinggian lapisan tersebut dicatatkan. Akhirnya, beberapa nisbah minyak kepada etanol disediakan dan bercampur untuk 20 minit pada suhu 45^{0}C dan campuran itu diuji dengan alat viscometer untuk mengukur kelikatan, kadar rich, rpm dan sebagainya. Purata diameter Sauter untuk etanol adalah 0.002m dan hajalu etanol adalah $7.29\text{x}10^{-3}$ m/s. Diameter Sauter untuk minyak adalah 0.002m dan halajunya adalah 0.73m/s. Pada masa yang sama, hasil cecair yang dicampurkan menunjukkan bahawa kebolehlarutan etanol meningkat berkadaran dengan jumlah etanol pada 45^{0}C. Pada kepekatan etanol yang rendah, cerun menurun disebabkan oleh pembentukan fasa emulsi. Parameter utama yang mempengaruhi banjir di hidrodinamik adalah kelikatan dan kelajuan pergolakan viscometer. Kelikatan rendah akan membuat aliran cecair lebih mudah. Walaubagaimanapun, peningkatan kelajuan pergolakan meningkatkan peluang banjir di viscometer.

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LIST OF SYMBOLS

^0C	Degree Celsius
mL	Mililiter
L	Liter
S	Second
Re	Reynolds number
v	Viscosity
D_{32}	Sauter diameter
%	Percentage

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CHAPTER 1

INTRODUCTION

1.1 INTRODUCTION

This chapter mainly emphasizes on the general idea of this study along with problem statement, objectives, scope, and significant of the study.

1.2 BACKGROUND OF STUDY

Flooding phenomena is the hydraulic capability of a countercurrent extractor forced by revolution of one liquid phase into the discharge stream of the other. The point at which an extractor floods is a function of the design of the internals as this affects the holdup characteristics of the extractor, the solvent-to-feed ratio and physical properties as this affects the liquid-liquid dispersion behavior, the agitation intensity, the specific input. The latter often is expressed in terms of the volumetric flow rate per cross-sectional area and liquid velocity. Flooding limitations set a restriction for the maximum flow rate in equipment, based on counter current two phase flow, such as extraction columns.

While immiscible liquid refers to the property where two liquids are not capable of combining to form a homogeneous mixture. In this study, palm oil is used that came from triglycerides group where ethanol is used that came from alcohol group to conduct the experiments. Though classified as immiscible, in actual fact there is still some degree of mutual solubility. For example, again using the example of benzene and water a small amount of benzene will be dissolved in the water phase and vice versa. Since by definition, immiscible liquids do not interact with each other in any way whatsoever, they will evaporate completely independent of each other. An important outcome is that the boiling point of an immiscible mixture must be lower than that of either of its components.

Flooding phenomena usually happens in extraction column where a small portion of dispersed phase comes out in the top of the column. Ideally extractors are designed to operate near flooding to maximize productivity. In practice, however many new column extractors are designed to operate at 40 to 60 percent of the predicted flood point because of uncertainties in design, process impurity uncertainties, and allow for future capacity increases. This practice varies from one type of extractor to another and one designer to another. In a static extraction column, countercurrent flow of the two liquid phases is maintained by virtue of the difference in their densities through the equipment. Only one of the liquids may be pumped through the equipment at any desired velocity of flow rate (dispersion phase); the maximum velocity of the other phase is the fixed by the flood point. If an attempt is made to exceed this hydraulic limit, the flooding will occur.

1.3 PROBLEM STATEMENT

Usually, liquid-liquid flow systems are operated close to flooding point. In a reactive liquid flow system with two immiscible or partially immiscible liquids, the flooding would also be due to the appearance of emulsion media either in the dispersed phase or in the continuous phase. The phenomenon will definitely be a significant impact the mass transfer limitation of the process. Despite the typical hurdle with the liquid-liquid flow systems the process also faces the uncertainty of mixture environment. The determination of significant factors using an appropriate factorial design would help optimize the operational condition of the column during reactions.

1.4 RESEARCH OBJECTIVES

To elaborate the flow behavior and potential of flooding of the liquids, the following are the objectives:

1.4.1 To determine the Sauter diameter and velocity of dispersed droplet either in excess ethanol or excess oil.

1.4.2 To investigate the effect of various volumetric ratios of two liquid phases on the time elapsed for liquid-liquid settlement.

1.4.3 To characterize the hydrodynamic properties for various ethanol to oil ratio using the cylindrical viscometer

1.5 SCOPE OF STUDY

This is a preliminary study before going into the actual rig. All experiments and testing were done under batch modes in the bench-scale rigs. Even so, the results can be used to discuss flow system in a conceptual manner by means of dimensionless number in hydrodynamics. Firstly, the oil is fresh palm oil and the ethanol is the one which 95% grade of quality (technical grade) as the economically available feedstock. This grade of purity easily gets and can produce a significant result. Then, the temperature of mixing is 45^0C, optimum temperature for enzymatic reaction.

1.6 SIGNIFICANCE OF STUDY

Now, the biodiesel productions are getting more attention so that the cost for fuel can be reduced. The immiscibility of reactants in the biodiesel synthesis widens the readily problematic kinetics of reversible reaction. Hence, the extraction part may play main role in biodiesel synthesis. The liquid-liquid extractive reactor for biodiesel is still new where the hydrodynamics of two immiscible liquid systems is necessary to be studied to elucidate possible limitations including the flooding.

CHAPTER 2

LITERATURE REVIEW

2.1 GENERAL CONSIDERATIONS

The limit of stable counter current liquid-liquid flow is closely related with flooding as such it is an important design criterion in a different of process equipment such as falling film absorbers, evaporators, reflux, and heat pipes and in some features of nuclear reactor safety. Flooding results the limit of steady counter current flow. There are some uncertainties about the precise mechanism although flooding has been studied for number of decades. Generally flooding occurs when large numbers of waves formed near the liquid outlet (Suzuki and Ueda, 1977; McQuillan et al., 1985; Govan et al., 1991).

One of the extraction column that are suitable for analysis is Oldshue and Rushton column(Ramtahal, 2001). Most counter current multistage extraction processes have made use of a series of mixer and settlers. In these systems two relatively immiscible phases are brought into intimate contact by mixing in one vessel and then allowed to separate in another non agitated vessel; this combination constitutes a stage. Two phases pass counter currently from stage to stage through the system, hence the name multistage counter current operation (Oldshue and Rushton, 1952). Mixer settler arrangements with individual mixing and settling tanks and connecting piping and pumps are complicated and in some cases are large and bulky, but because of their capacity and simple mechanism they enjoy considerable use.

2.2 FLOODING IN TWO IMMISCIBLE LIQUIDS SYSTEM

Diverse range of processes and equipment were encountered by flows of two immiscible liquids. Accurate prediction of immiscible liquids flow characteristics such as flow pattern, and liquid-liquid holdup is important in many engineering applications. However, the method used to explored liquid-liquid flow is not same as gas-liquid flows. So, various concepts and phase laws that applied to gas-liquid cannot be readily applied to liquid-liquid systems. Firstly, flow patterns, were observed in liquid-liquid systems. Mostly, visual techniques such as photographic, videos or sudden changes in average pressure drop used to identify the flow pattern of a system. Nowadays, conductivity measurements, high frequency impedance probes or Gamma densitometer for local hold-up sampling, or local pressure fluctuations and average hold-up measurements used to study the flow patterns (Al-Moosawy and Al-Hattab; 2008).

Flow pattern in a liquid-liquid systems is depend on wall wetting properties of the liquid and surface tension forces of specified liquid. The role of gravity in liquid-liquid system is neglected. However, different flow pattern may result by changing the tube material hydrophobic or hydrophilic. Moreover, as a result of relatively low density change, wetting effects and surface tension become significant, and the interface shape like concave, plane and convex also affect the flow patterns.

2.3 EFFECT OF AGITATION SPEED ON FLOODING

The throughput of this column decreases with increase in agitation speed. The rise of agitation speed carries to higher shear stress and to intense drop breaking. It appears that the numbers of drops in the column increases due to decrease of relative velocity between the dispersed phase and continuous phase decreases and consequently the values of holdup will increase and the column becomes unstable. Moreover, as the agitation speed increases, the centrifugal rotation in the settler become significant and a large number of the dispersed drops passes through the settler without coalescing and consequently a decrease in maximum throughput follows. The holdup at flooding decreases with increasing agitation speed. It can also be observed that increasing the continuous phase flow rate reduces the dispersed phase flow rate at which flooding occurs and increases the values of holdup at flooding.

2.4 INTERFACIAL TENSION ON FLOODING

When the continuous phase is an aqueous solution of low viscosity, the rising velocity of the dispersed drops depends on the drop diameter, while the interfacial tension is the physical property which has the greatest effect on the drop size. Therefore, the holdup of dispersed phase and consequently the flooding point are expected to vary with interfacial tension. The decrease in interfacial tension results in the decrease of maximum throughput. The dispersed phase holdup at flooding increases with decreasing interfacial tension(Meisam Torab-Mostaedi & Ghannadi-Maragheh, 2009). Similar trends were also found by Rincon-Rubio et al. They observed that the holdup at flooding decreases with interfacial tension, when flooding occurred by phase inversion in a Wirz extraction column.

Kamei et al (1954) are one of the other researchers that perform a study on effect of surface tension in flooding phenomena. Flooding increases when surface tension increases. Moreover, the study of surface tension effect on flooding is complicated. This statement opposed by Ousaka et al. (2006) and Chung et al (1980) where they said that surface tension had a stabilizing effect on flooding. Their statement has been proved with experimental data which shows that instability of interfacial area and decrease of flooding velocity with decrease of surface tension. When studies related to tube inclination, it is concluded that the surface tension considerably affects the flooding mechanisms at high liquid flow rate, in that upper flooding changes to lower flooding as the surface tension decreases (Deendarlianto et al., 2010). Moreover, the effect of surface tension is minimal on flooding for low liquid flow rate. When surface tension decreases, the flooding velocity also decreases. These are the factor affected by surface tension on flooding system.

2.5 DEFINITION OF SAUTER DIAMETER

The Sauter diameter of droplet and its velocity's related journals and articles compiled here for evident the flooding based on the diameter and the velocity of ethanol and palm oil. Based on Wikipedia's definition for Sauter diameter also known as Sauter mean diameter is an average of particle size. It was originally developed by German scientist J. Sauter in the late 1920's. It is defined as the diameter of a sphere that has the same volume/surface area ratio as a particle of interest. Mathematically, Sauter diameter is defined according to equation:

$$\text{Sauter diameter} = d_{32} = \frac{\sum n_{pi} d_{pi}^3}{\sum n_{pi} d_{pi}^2}$$

where n_{pi} represents the number of the drops of the class i of the diameter d_p (Filippa, and Trento, 2011). The Sauter diameter is common measure in fluid dynamics as a way estimating the average particle size. It is especially important in calculations where the active surface area in important such as catalysis and application in fuel combustion and etc.

2.6 EFFECTS OF SAUTER DIAMETER

The high viscosity of the liquid causes an increase of the Sauter diameter. The viscosity has a direct effect upon the final atomisation process by which the drops are formed, and this final process directly affects the Sauter diameter. With the high viscosity of vegetable oils and biodiesels compared to ethanol, the droplets tend to be bigger. Consequently, an increase in viscosity of the liquid has an adverse effect on the fineness of atomisation; hence any change by which the viscosity is decreased will improve the atomisation of the spray. The Sauter diameter depends on different fuel properties, such as surface tension, viscosity, fuel flow rate and injection pressure as well as air ambient density (David Rochaya, 2007).

Based on Ing and Jaafar (2010), the viscosity of the liquid is directly proportional to the Sauter diameter. Other than that, the Sauter diameter increases when the density of the liquid is increase. When the density of the mixture increase, atomisation process will become slower and lead to the increase in Sauter diameter. It can be seen that viscosity, surface tension and density will affect the Sauter diameter of various biofuel blends. Those biofuel properties play an important role in affecting the spray characteristic of biofuel blends. Density has the highest influence in Sauter diameter of biofuel, followed by viscosity and surface tension. Therefore, when surface tension increases the Sauter diameter will also increase (Ing and Jaafar, 2010).

The knowledge of the drop size is of fundamental importance for the hydrodynamic behaviour of pulsed sieve plate extraction columns. It decides the fate of the rise velocity of drop, dispersed phase holdup, the residence time of the dispersed phase as well as the flooding behaviour of the extractor. Furthermore together with the holdup, it determines the interfacial area for the mass transfer and affects both the continuous and dispersed phase mass transfer coefficient (Lade, Rathod; 2012).

Then, drop size also effect the droplet break up. The smaller drops found at higher pulsation intensity, which is a result of increased droplet break up. However, a high agitation rate, the fall in the values of Sauter diameter is gradual. Droplet coalescence is enhanced at high pulsation intensity by the increasing probability of droplet collision. Thus at high pulsation intensity, the enhanced rate of coalescence overcomes the increased tendency for droplet breakdown and the drop size apparently stabilized. The effect of pulsation intensity on the mean drop size of the toluene–water system (high interfacial tension) is larger than that of the other systems (low and medium interfacial tension), because breakup of the dispersed drops into smaller ones is limited (Meisam Torab, 2011).

2.7 SAUTER DIAMETER ON FLOODING POINT

Sauter diameter plays main role in flooding phenomena. The flooding point in Sauter diameter depends on dispersed phase velocity and continuous phase velocity. Dispersed phase velocity decreases with increase in continuous phase velocity. Increasing the continuous phase velocity leads to the reduction of slip velocity between the phases. Therefore, the dispersed phase holdup increases with increase in continuous phase velocity and consequently the column becomes unstable at lower dispersed phase velocities. The maximum throughput of the column decreases due to a decrease of the drop size with increasing pulsation intensity, which results in an increasing hold-up such that the critical hold-up is reached at a lower total throughput. Further, lower interfacial tension results in lower total allowable throughput than is the case for the higher interfacial tension, due to the smaller drops (Meisam Torab, 2011).

2.8 THE IMPORTANCE OF SAUTER DIAMETER ON DESIGNING EXTRACTION COLUMN

Meisam Torab (2011) said in the design of an extraction column, besides separation performance, the hydrodynamic parameters, being hold up, flooding velocities and drop size are key parameters in order to determine the column capacity and the column diameter required to provide the desired throughput within the operating region. The equilibrium droplet size distribution in a solvent extraction column is usually represented as the average volume to surface area, known as the Sauter mean diameter.

The Sauter drop diameter is a key variable in extraction column design due to its influence on both throughput and specific interfacial area available for mass transfer and it also influences the mass transfer coefficient, the dispersed phase hold up, and flooding conditions of the column. In order to develop appropriate design procedure of a given type of extraction column, a knowledge of average drop size in terms of the operating variables, column geometry and liquid physical properties is thus of the paramount importance.

2.9 LIQUID LAYER SETTLEMENT STUDY: CONCEPTS OF LIQUID HOLD-UP

A liquid-liquid two-phase flow takes place in many industrial processes. Common examples are water separation from crude oil, dispersion separation in different solvent extraction settlers, or dispersion separation at the ends of differential direct contact mass transfer columns. Proper design of the industrial equipment is usually based on local experience gained through complicated, large scale industrial processes or on the know-how supplied by the equipment manufacturer.