The capability of buoyancy weighing bar method in determining the separation time of biodiesel and glycerol at various concentrations

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Abstract. This study aims to examine the ability of the buoyancy weighing bar method in determining the separation time of biodiesel and glycerol at various concentrations. The comparison of biodiesel and glycerol concentrations studied are 99%:1%; 98%:2%; 97%:3%; 96%:4%, while the type of rod used is made of iron with a diameter of 10 mm and length of 210 mm. This research is conducted at room temperature and using gas chromatography analysis result as a comparison. The results are also compared with aluminium rods with diameter of 5 mm and 15 mm diameter, also compared with aluminium rods with length of 160 mm, 110 mm, and 60 mm diameter. From the results obtained, it is known that the buoyancy weighing bar method can predict the separation time at various concentrations of biodiesel and glycerol.

1. Introduction
The process of separation between two fluids that do not mix can be done in various ways. One method used to separate two liquids with different densities is to utilize gravity. The buoyancy weighing bar method is the latest method that utilizes gravity to determine the separation time between two fluids which do not mix [1, 2]. In this buoyancy weighing-bar method, the method used is to hang a rod on the suspension, then the mass of this rod will be measured along with the heavier mass transfer to settle and the lighter mass will float. This is done until the mass of the rod is constant which indicates that the separation process is complete [3, 4, 5]. Various variations are made to determine the accuracy of the buoyancy weighing-bar method. In this study, the samples tested are biodiesel (methyl ester) and glycerol, and this study aims to examine the ability of the buoyancy weighing bar method in determining the separation time of biodiesel and glycerol at various concentrations. The mixture of biodiesel and glycerol forms two liquid phases which are not fused, the dispersed phase and the continuous phase. The dispersed phase is the phase that plays a role in the formation of bubbles and the continuous phase is the phase that forms the matrix in which the bubbles are suspended [6]. Two types of emulsions are formed namely glycerol in biodiesel where bubbles from glycerol are dispersed in biodiesel, and biodiesel in glycerol where bubbles from biodiesel are dispersed in glycerol [7].

The emulsion formed depends on the volume of the continuous phase and the dispersed phase. Increasing the viscosity of the continuous phase will increase the stability of the emulsion. The larger the bubble size and the less continuous phase volume, the emulsion will be less stable [8]. In a stirred tank, the rate of mass transfer between the liquid-liquid phase system depends not only on the dynamic motion of the two mixed liquids but rather on the size of the bubbles. Bubble size can be formed from bubble splitting or bubble fusion. Bubble splitting occurs in turbulent flow, whereas the merging of bubbles depends on the frequency of collisions and the efficiency of merging between bubbles. The greater the dispersed phase fraction, the greater the collision [9]. The small bubble size produced by homogenization can increase the dispersed phase. As a result, viscosity is increasing and emulsifier absorption can increase. Emulsification also requires proper homogenization time. The intensity and duration of the mixing process depend on the time needed to dissolve and distribute it evenly [9].
Damage or destabilization of the emulsion occurs through three main mechanisms namely creaming, flocculation and coalescence. Creaming is a separation process that occurs due to upward or downward movements, this occurs because of the gravitational force on phases that have different densities. Flocculation is an aggregation of bubbles. In flocculation, there is no concentration of film between surfaces so that the number and size of globules remain. Flocculation will accelerate creaming. Coalescence is the merging of bubbles into larger bubbles. At this stage, there is a concentration of film between surfaces so that the bubble size changes [9].

2. Methods
The material used in the study is glycerol ($\rho = 1260 \text{ kg/m}^3$) and biodiesel ($\rho = 867 \text{ kg/m}^3$) [10]. The equipment used in the study is the FS AR-210 analytical balance with an accuracy of 0.0001 which have a hook below to hang the weighing bar. This weighing bar is used to detect the migration of biodiesel and glycerol bubbles, where glycerol will settle while biodiesel will float. The rod is made of aluminum ($\rho = 2826.654 \text{ kg/m}^3$) with a rod length of 210 mm and a diameter of 10 mm. The concentration ratios of biodiesel and glycerol are varied by 99%: 1%; 98%: 2%; 97%: 3%; 96%: 4%.

Gas chromatography is used as a comparison to determine the purity of biodiesel separation. A special stirrer is used to homogenize suspensions at the beginning of the experiment.

The study is carried out by mixing biodiesel and glycerol at 298 K (room temperature) to form two layers with variations in the ratio of the concentration of biodiesel and glycerol. The two layers formed consist of biodiesel at the top and glycerol at the bottom. The buoyancy weighing bar method is used to find out that biodiesel and glycerol are completely separated. All mixtures are stirred before measurement. To prepare the suspension, 1000 ml of the mixture is put into a glass cylinder. After stirring with a special stirrer, the rod is put into suspension then the rod masses are recorded. The length of measurement is about 10 minutes and data of rod masses are recorded at 1- second interval.

3. Results and discussion

3.1. Effect of liquid concentration
Figure 1 shows the ability of the buoyancy weighing-bar method to determine the separation time of biodiesel and glycerol at various concentration ratios. The cylinder rod used is a type of aluminum with a length of 210 mm and a diameter of 10 mm.

![Figure 1. The ability of the buoyancy weighing-bar method to determine the separation time of biodiesel and glycerol at various concentration ratios.](image-url)
In figure 1 it can be seen that at a concentration ratio of 99%: 1%, the mass of the rod is constant faster than in the other concentration ratios. This is followed by a comparison of 98%: 2%, 97%: 3%, and the last 96%: 4%. At a concentration ratio of 99%: 1%, the mass of the rod is constant at 150 seconds, at a concentration ratio of 98%: 2%, the mass of the rod is constant at 90 seconds, at a concentration ratio of 97%: 3%, the mass of the rod is constant at 370 seconds, and at a concentration ratio of 96%: 4%, the mass of the rod is constant at 500 seconds. This phenomenon shows that the mass migration that floats and sinks according to the concentration ratio. The higher the concentration of biodiesel in glycerol, the separation process will require a longer time. At the beginning of the separation process, larger bubbles will float first, so that the decrease in rod mass also occurs quickly. The longer the duration of the separation process, the decrease in rod mass will also be slower until eventually constant. This constant condition indicates that the separation has been completed because all the light bubbles (biodiesel bubbles) have floated above glycerol [11]. In the suspension analysis at 200 seconds using gas chromatography, the biodiesel contents obtained are 98.1265%, 98.6815%, 98.6705%, 98.33678% respectively for a ratio of 99%: 1%, 98%: 2%, 97%: 3% and 96%: 4%.

3.2. Effect of rod diameter
Figure 2 shows the effect of rod diameter in detecting the separation time of biodiesel and glycerol at a ratio of 99%: 1% biodiesel and glycerol concentrations. The rod used is aluminum with a cylindrical shape with a diameter variation of 5 mm, 10 mm and 15 mm.

![Figure 2](image_url)

**Figure 2.** Effect of cylinder rod diameter on separation time at a ratio of 99%: 1% biodiesel and glycerol concentration.

Figure 2 shows that rod with a diameter of 15 mm gives more accurate predictions of separation time compared to diameters of 5 mm and 10 mm. It can be seen that the constant point achieved using a rod with a diameter of 15 mm is longer than a rod with a diameter of 5 mm and a diameter of 10 mm. The constant point achieved using a rod with a diameter of 5 mm and a diameter of 10 mm is about 150 seconds, while the constant point achieved using a rod with a diameter of 15 mm is 250 seconds.
In the biodiesel test using gas chromatography at the 200 seconds, it is found that the biodiesel content is 98.1265 %, which means that before the 200th-second, the mass of the rod should not be constant.

3.3. Effect of rod length

Figure 3 shows the effect of rod length to detect the separation time of biodiesel and glycerol at a ratio of 99 % : 1% biodiesel and glycerol concentrations. The rod used is aluminum with a diameter of 10 mm.

![Figure 3. Effect of length of rod on separation time at a ratio of 99%: 1% biodiesel and glycerol concentration.](image)

In Figure 3, it can be seen that the length of 210 mm reaches a constant point later than the length of the rods of the 160 mm, 110 mm and 60 mm. Based on the data analysis results using gas chromatography in the comparison of the concentration of biodiesel and glycerol 99% : 1% at 200 seconds, it is found that the biodiesel content is 98.1265%. This means that the mass of the bar in this condition should not be constant, especially less than 200 seconds.

The bar with the length 210 mm is constant at about 150 seconds, the bar with the length 160 mm is constant at about 110 seconds, the bar with the length 110 mm is constant at about 30 seconds, and the bar with the length 60 mm is constant at about 10 seconds.

So it can be concluded that the rod with a length of 210 mm gives more accurate predictions of separation time compared to the length of 160 mm, 110 mm and 10 mm.

4. Conclusion

The conclusion obtained in this study is that buoyancy weighing bar method can predict the separation time of biodiesel and glycerol at various concentrations. Different concentrations do not significantly affect the ability of buoyancy weighing bar to detect the separation of biodiesel and glycerol, although the concentration comparison affects the length of time for glycerol-biodiesel separation. Rod with a diameter of 15 mm gives more accurate predictions of separation time compared to diameters of 5 mm and 10 mm, and rod with a length of 210 mm gives more accurate predictions of separation time compared to lengths of 160 mm, 110 mm, and 60 mm.
References

[1] Tambun R, Sibagariang Y and Manurung J 2018 Influence of the weighing bar size to determine optimal time of biodiesel-glycerol separation by using the buoyancy weighing-bar method *IOP Conference Series: Materials Science and Engineering* **309** pp 012067

[2] Tambun R, Sibagariang P, Alphantaria B and Rambe A M 2018 Droplet size distribution measurement of water in kerosene by the buoyancy weighing-bar method *MATEC Web of Conferences* **154** pp 01016

[3] Motoi T, Ohira Y and Obata E 2010 Measurement of the floating particle size distribution by a buoyancy weighing-bar method *Powder Technology* **201**(3) pp 283-288

[4] Obata E, Ohira Y and Ohta M 2009 New measurement of particle size distribution by a buoyancy weighing-bar method *Powder Technology* **196**(2) pp 163-168

[5] Tambun R, Nakano K, Shimadzu M, Ohira Y and Obata E 2012 Sizes influences of weighing bar and vessel in the buoyancy weighing-bar method on floating particle size distribution measurements *Advanced Powder Technology* **23**(6) pp 855-860

[6] Liu D H, Liptak B G 1997 *Environmental engineers' handbook* CRC press.

[7] Beckett S T 2012 *Physico-chemical aspects of food processing* Springer Science & Business Media

[8] Maaß S, Gäbler A, Zaccone A, Paschedag A R and Kraume M 2007 Experimental investigations and modelling of breakage phenomena in stirred liquid/liquid systems *Chemical Engineering Research and Design* **85**(5) pp 703-709

[9] McClements D J 2015 *Food emulsions: principles, practices, and techniques* CRC press

[10] Irvan, Trisakti B, Hasibuan R and Joli M 2018 Fermentative utilization of glycerol residue for the production of acetic acid *IOP Conf. Ser: Mater. Sci. Eng.* **309** 012126

[11] Mao Y, Yong L, Tao H, Shimin W and Yiqian X 1998 In-situ measurement of droplet size distribution by light scattering method *Wuhan University Journal of Natural Sciences* **3**(4) pp 418-422.