

Study on Ethanol-Water Batch Distillation

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Abstract

This research was concerned about studying variables in simple distillation system, based on conservation of mass and energy principle as well as developing mathematical model of mass flow rate of ethanol-water mixture via MATLAB. The variables were steam pressure, temperature, concentration of ethanol-water and mass flow rate. In summary, the concentration of distilled ethanol-water mixture was decrease due to the decreasing of concentration of ethanol in the pot still. The results from calculation tended to be the same as results from experiment, the average deviation of initial concentration of ethanol-water mixture, reactant mixture, in pot still 10% and 30%v/v between experiment and calculation was 6.64 and 11.80%. For the mass flow rate of distilled ethanol-water mixture is decrease due to the decreasing of ethanol concentration in pot still and the distillation temperature is increase to boiling temperature of water. And the average deviation of mass flow rate of distilled ethanol-water mixture of experiment which differs from Yuthapong-Thanakom model calculation, 2.02% differ from theory calculation, 2.22%. The results from this research are expected to be useful for developing distillation system in the future.

Keywords: Pot-Still, Ethanol-Water mixture, Distillation, Mass flow rate, Binary mixture

1. Introduction

The stainless steel Pot-Still is widely used in food, chemical and medicine industry. On the other hand, Copper Pot-Still is widely used in alcohol beverage industry in which requires high quality distillation process, for instance, Whiskey and Brandy. The advantages of copper Pot-Still are easy to form and well conductive. It is widespread in Europe such as French, Portugal, and Scotland. From many studies on related field: the study on mathematical model developing for Batch distillation with column, based on mass

balance theory and evaporated proportional, which is broadly applied to control and construct distillation process. The mathematic models for ethanol distillation are developed such as A.Bonsfills [1], and the study on whisky product process developing individual mathematical model for each of other alcohol production process And E.Cassel [2] developed mathematical model for essential oil from rosemary, stem of sweet basil and lavender as well as compared results from model with experiment. Vincente Gomic [3] did an experiment about liquid-vapor equilibrium of

ethanol especially in Azeotropic zone by adding Dimethylbenzene. Y.Pakam and T.Soontornchainacksaeng [4,5] study on parameters and system simulations the copper pot still. They simulated the systems with MATLAB and created the mathematic model for the distilled ethanol-water mixture. They also varied the gage pressure and the concentration of ethanol-water mixture. The mass flow rate of distilled ethanol-water mixture was direct variation to inlet steam gage pressure. In another case the concentration of distilled ethanol-water mixture was decrease due to concentration of ethanol-water mixture in the pot still is decreased. Gele M.de koeijer [6] who created the heat and mass transfer mathematic models in ethanol-water mixture distillation founded the error of mathematic models from the experiments because of tiny kinetic theory in distillation tower. Fawzi A. Banat [7] simulated the mass transfer in membrane distillation of ethanol-water mixture. The distillation temperature and concentration of ethanol-water mixture were varied with diffusion paths. Claudio A. Faundez [8] who created the vapor-liquid equilibrium mathematic model of substances in alcoholic distillation processes from wine used the binary mixture model. The results of substances in this research consisted of water + ethanol + cogener but the cogener could be negligible. Zbigniew T. Fidkowski [9] optimized batch distillation by the minimized the cycle time in order to minimum energy for separation in continuous distillation. Jyun-Yang Yao [10] simulated the dynamic simulations in Batch distillation for separating mixtures with minimum-boiling azeotrope. one separating acetone and methanol using water as entrainer and the other

separating isopropyl alcohol (IPA) and water using dimethyl sulfoxide (DMSO) as entrainer. M.E.T Alvarez [11] experimented a packed batch distillation column. These data that were taken from separation of ethanol/water mixture was studied at different reflux ratios. It was verified that the higher ethanol concentration was obtained in one hour of distillation and the accumulate ethanol concentration was above to 80% (mole) when operated at constant reflux. This research is possible to obtain high yields and concentrations of ethanol in a short time. Bai Peng [12] control temperature method of cyclic total reflux batch distillation by introducing middle and top temperature in batch distillation, isopropanol/1-propanol and ethanol/1-propanol. The results indicate that the operation state switch of total reflux and total withdrawal with this method in cyclic total reflux batch distillation is completely feasible and the product yield is high. Daniel Osorio [13] simulates for wine distillation that is 40% faster than the rigorous solution of the DAE system. The model can be applied to the distillation of other spirits or complex mixtures, as well as in other separation processes in which the recovery of aromas is essential.

The objective of these research variable concerning in distillation system, concentration and flow rate of ethanol in Pot-Still, in order to lay the foundation of distillation system.

2. Materials and methods

Materials and methods referred in this research include simple distillation principle and heat transfer theory based on conservation of mass and energy principle as follows.

2.1 Simple distillation principle

There are 3 important parts in distillation system for separating two substances are pot still, tube and condenser. The assumptions of pot still distillation consist of the heat transfer from condensation of saturated vapor to ethanol-water mixture in the pot still with unmixed, pot still surface is adiabatic, and condenser is the counter flow shell and tube heat exchanger, the distilled ethanol-water mixture flow in the tube and the cooling water flow in the shell.

The working principles of this distillation system are as follows: fill ethanol-water mixture with desired concentration into pot still, release saturated vapor into coil in order to transfer heat from itself to ethanol-water mixture until it is boiled, the distilled ethanol-water vapor goes through tube to condenser which is cooled by water, the concentration of remaining ethanol-water mixture is decreased and distillation temperature is increased to 100°C. The schematic representation is illustrated in Fig 1

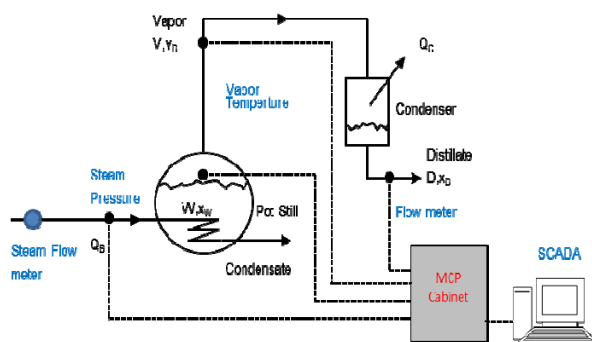


Fig.1 Schematic representation of Batch distillation system

2.2. Principle of mole fraction and pressure calculation

The mole fraction between ethanol and water, there are the range of boiling point of ethanol and water as 78°C and 100°C.

The equation for partial pressure from modified Raoult's law [14] for nonideal solution is used as shown in equation (1)

$$P = x_1 \gamma_1 P_1^{sat} + (1 - x_1) \gamma_2 P_2^{sat} \quad (1)$$

For the activity coefficient, γ_i , can be calculated from Van Laar equation [14] and can be calculated from equation (2) and (3) as follows

$$\ln \gamma_1 = A_{12} \left(\frac{A_{21}(1 - x_1)}{A_{12}x_1 + A_{21}(1 - x_1)} \right)^2 \quad (2)$$

$$\ln \gamma_2 = A_{21} \left(\frac{A_{12}x_1}{A_{12}x_1 + A_{21}(1 - x_1)} \right)^2 \quad (3)$$

Where x_1 mole fraction of ethanol in liquid state, x_2 mole fraction of water in liquid state, γ_1 activity coefficient of ethanol, γ_2 activity coefficient of water.

The theory calculation result of vapor-liquid equilibrium in ethanol-water mixture and the research of Rieder - Thomson [15] are closely the same as shown Fig 2

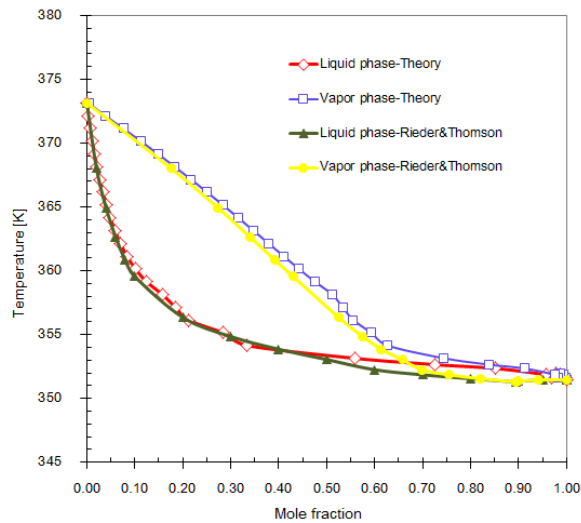


Fig.2. The comparison of vapor-liquid equilibrium between theory calculation and Rieder – Thomson

From Fig 2, the vertical axis is temperature, and mole fraction of ethanol is shown as the horizontal axis in which the point on the left-hand side of axis means pure water and the point on the right-hand side of axis means pure ethanol. The lower line means the mole fraction of liquid ethanol in any temperature. The upper line means the mole fraction of vapor ethanol in any temperature. Azeotropic point means that the mole fraction of ethanol is 0.9 in which the ethanol cannot be distilled anymore by typical distillation process.

The increasing temperatures affect to the mole fraction both liquid and vapor phase. The increasing temperatures range 351-355K effect to the decreasing mole fraction in liquid phase from 1.00 to 0.25 around 0.188/K and in vapor phase from 1.00 to 0.60 around 0.10/K. The increasing temperatures range 355-373K effect to the decreasing mole fraction in liquid phase from 0.25 to 0.00 around 0.014/K and in vapor phase from 0.60 to 0.00 around 0.033/K. Both of liquid and

vapor phase, the rate of decreasing mole fraction per temperature range 351-355K is greater than range 355-373K because of the concentration of ethanol in water.

The theory calculation results of vapor-liquid equilibrium which are differ from Rieder & Thomson in liquid and vapor phase are 18.21% and 12.65% respectively. These results can be used to calculate the ethanol-water mixture distillation. To illustrate how Fig 2 works, for example, draw the line from horizontal axis at mole fraction equals 0.08 to the lower line then draw the line horizontally from intersection point to upper line which are both in the range of 360K, after that draw the straight line back to horizontal axis. The illustration is inferred that when the reactant with initial mole fraction equals 0.08 is heated to its boiling point (360K), the temperature remain constant until it turns into vapor state so that the point from intersection between the line drawn from upper line and horizontal axis is the remaining mole fraction. The Rayleigh Distillation equation[14], the relationship between total moles remaining in the Pot-Still and the mole fraction of the more volatile component in the Pot-Still, is shown in equation 4.

$$\ln \left(\frac{H_i}{H_f} \right) = \int_{x_f}^{x_i} \frac{1}{y-x} dx \quad (4)$$

where H_i is initial moles of liquid originally in pot still, H_f is final moles of liquid remained in pot still, x_i is initial mole fraction in liquid composition in pot still, x_f is final mole fraction in liquid composition in pot still, x is mole fraction in liquid phase, y is mole fraction in vapor phase

2.2. Heat transfer theory

According to Phase-Change process of ethanol-water mixtures when exothermic and endothermic phenomenon take place, the equation is developed by conservative of energy principle. The energy that is used for changing state is called latent heat of vaporization. The saturated vapor that is issued in the coil and exits in saturated liquid transfers the heat conduction to boil the initial ethanol-water mixture. The vapor of ethanol-water mixture that rises up to condensing unit is the product of ethanol-water mixture. [16,17,18]

3. Methodology

The purpose of this research is to study on experimental results from alteration in concentration ethanol-water mixture and mass flow rate within copper pot still in order to conduct mathematical model and compare to theoretical results. The hypotheses of this research include: the properties of each of substance is ideal, and potential and kinetic energy are neglected. The boundaries of this research are as follows.

3.1 Concentration of ethanol-water mixture

3.1.1 Define variables as an initial concentration 10, 20, 30, and 40%v/v, and then study the effect from experiment.

3.1.2 Repeat experiments in which have 10 and 30%v/v initial concentration 5 times in order to average the results for comparing with theoretical results.

3.2 Mass flow rate of distilled ethanol-water mixture

3.2.1 Define variables as an inlet absolute pressure, 131.325, 141.325, 151.325, 161.325 kPa, for 30%v/v concentration and then calculate

the mass flow rate for comparing with theoretical results.

3.2.2 Develop mathematical model of mass flow rate and then calculate

3.2.3 Repeat an experiment in which has 30% initial concentration 5 times in order to average the results for comparing with theoretical results.

4. Developing mathematical model of mass flow rate of distilled ethanol-water mixture

The conservative of heat and mass are used to simulate in MATLAB. Measure the basic variables, mass flow rate of inlet saturated vapor, in absolute pressure term, 131.325, 141.325, 151.325, 161.325 kPa in order to develop the relationship equation between rate of heat transfer and absolute pressure by curve fitting method.

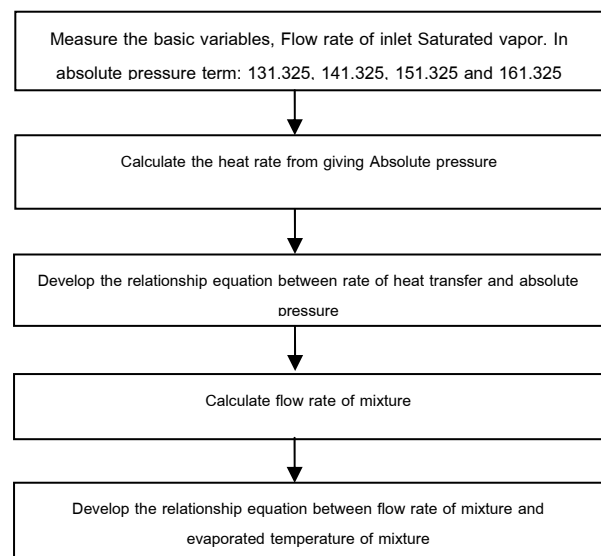


Fig.3 Flow diagram

The mass flow rate of ethanol-water mixture by applying conservative of mass and energy principle and develop the relationship equation between mass flow rate and distillation

temperature by curve fitting method as shown in equation (5) and (6)

$$\frac{dm}{dt} = \varphi \int_{T_1}^{T_2} \frac{1}{T^{1.855}} dT \quad (5)$$

$$\varphi = 14.26 \int_{P_1}^{P_2} P^{0.177} dP \quad (6)$$

Where P is atmosphere pressure, T is temperature, m is mass of mixture, t is time.

Boundary condition

$$\varphi(131.325) = 3813.7, \quad \dot{m}(358.15) = 0.090, \\ \dot{m}(373.15) = 0.083$$

$$\varphi(141.325) = 4057.8, \quad \dot{m}(358.15) = 0.096, \\ \dot{m}(373.15) = 0.088$$

$$\varphi(151.325) = 4427.1, \quad \dot{m}(358.15) = 0.105, \\ \dot{m}(373.15) = 0.096$$

$$\varphi(161.325) = 4850.3, \quad \dot{m}(358.15) = 0.115, \\ \dot{m}(373.15) = 0.112$$

Where Coefficient, φ , that depends on inlet absolute pressure: 131.325, 141.325, 151.325, and 161.325 kPa is used for conducting relationship equation among mass flow rate, heat transfer rate, and distillation temperature

5. Results and discussion

The concentration of ethanol-water mixture in pot still is compared between theoretical and experimental results. Likewise, mass flow rate of distilled ethanol-water mixture is compared between mathematical model and experimental results.

5.1 Concentration of ethanol-water mixture, reactant mixture, in Pot-Still

The results from the experiment show that boiling point and distillation temperature in each of concentration ethanol-water mixture are different depending on initial concentration. In this experiment, the initial concentration was 10, 20, 30, and 40%v/v. As a result, boiling point of ethanol-water mixtures were 366, 362, 359, and 358K respectively. In addition, the initial concentrations of distilled ethanol-water mixtures were 50, 66, 75, and 80%v/v respectively. The concentration of ethanol-water mixture in pot still direct variation to concentration of distilled ethanol-water mixture, the distillation temperature is close to the boiling temperature of water according to the high amount of water in mixture. Moreover, the concentration of distilled ethanol-water mixture is low according to the low amount of ethanol in reactant mixture. In brief, Initial concentration of reactant mixture strongly influences to other properties of mixture

The results from experiment shows that when pot still is heated to boiling temperature of water, 373K, the ethanol run out during temperature is in range of 358-366K, the concentration of distilled ethanol-water mixture decrease by 2.5-4.0%/K. During temperature is in range of 366-371K, the concentration of distilled ethanol-water mixture decrease by 4.00-7.78%/K. during temperature is in range of 366-371K, the concentration of distilled ethanol-water mixture decrease by 7.78-11.61%/K. As mentioned in 2.1.

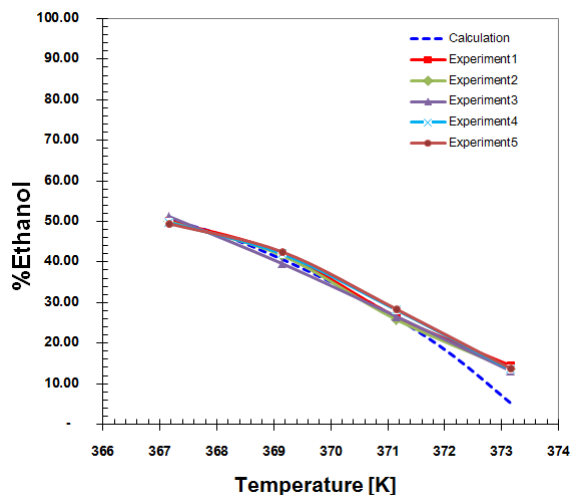


Fig.4. Relationship between the concentration of distilled ethanol-water mixture and initial concentration 10%v/v

From the Fig 4, the result from calculation is 50.18%v/v which is close to the result from experiment, 50%v/v. Nevertheless, there is an error at 373K because this process has to be stopped at 13.72%v/v concentration of distilled ethanol-water mixture due to the limitation of worthiness in energy and quality of product. Hence, the error between experiment and theory is about 6.64 %v/v.

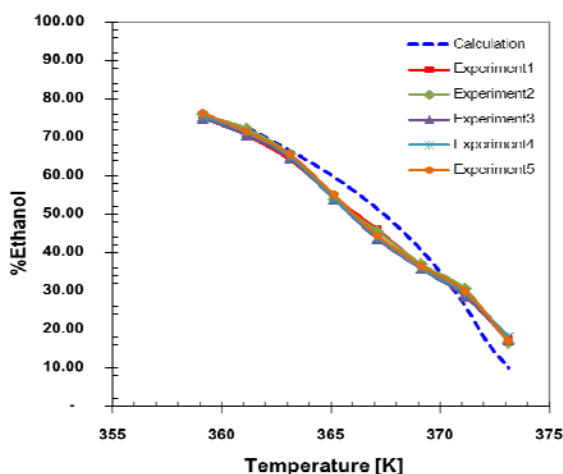


Fig.5. Relationship between the concentration of distilled ethanol-water mixture and initial concentration 30%v/v

From the Fig 5, the result from calculation is 75.70%v/v which is close to the result from experiment, 70%v/v. Nevertheless, there is an error at 373K because this process has to be stopped 17.18%v/v concentration of distilled ethanol-water mixture due to the limitation of worthiness in energy and quality of product. Hence, the error between experiment and theory is about 11.80%v/v

5.2 Results of mass flow rate of distilled ethanol-water mixtures

The mass flow rate of distilled ethanol-water mixture depends on concentration of ethanol in pot still, stream pressure, and distillation temperature. When steam is inputted with high absolute pressure, the mass flow rate of distilled ethanol-water mixture is increased correspondingly. In addition, when steam is inputted continuously during distillation temperature, the mass flow rate of distilled ethanol-water mixture is decreased due to the increasing of distillation temperature as shown in Fig 6-Fig 9.

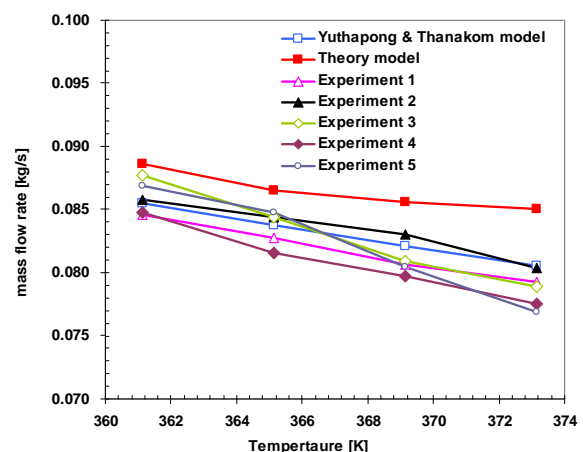


Fig 6. Relationship between the mass flow rate of distilled ethanol-water mixture and distillation temperature at absolute pressure of saturated vapor 131.325 kPa.

From the Fig 6. The absolute pressure of saturated vapor 131.325 kPa., distillation temperature at 361, 365, 369 and 373K, the mass flow rate of distilled ethanol-water mixture of theory calculation is 0.0886, 0.0865, 0.0856, 0.0851kg/s, or 0.1146, 0.1089, 0.1063, 0.1050L/s respectively. The mass flow rate of distilled ethanol-water mixture of Yuthapong-Thanakom model calculation is 0.0855, 0.0838, 0.0821, 0.0805kg/s or 0.1061, 0.1015, 0.0971, 0.0927L/s respectively. The average mass flow rate of distilled ethanol-water mixture 5 experiments is 0.0860, 0.1010, 0.0970, 0.0958kg/s or 0.1074, 0.1010, 0.0970, 0.0958 L/s respectively. The 5 experiments that differ from theory calculation average 3.61% differ from Yuthapong-Thanakom model average 0.56%

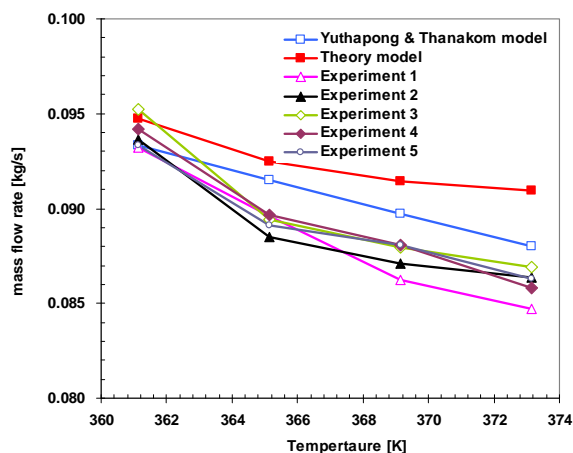


Fig 7. Relationship between the mass flow rate of distilled ethanol-water mixture and distillation temperature at absolute pressure of saturated vapor 141.325 kPa.

From the Fig 7. The absolute pressure of saturated vapor 141.325 kPa., distillation temperature at 361, 365, 369 and 373K, the mass flow rate of distilled ethanol-water mixture of theory calculation is 0.0947, 0.0925, 0.0914, 0.0909kg/s or 0.1311, 0.1250, 0.1222, 0.1208L/s

respectively. The mass flow rate of distilled ethanol-water mixture of Yuthapong-Thanakom model calculation is 0.0934, 0.0915, 0.0897, 0.0880kg/s or 0.1274, 0.1224, 0.1176, 0.1129L/s respectively. The average mass flow rate of distilled ethanol-water mixture 5 experiments is 0.0939, 0.0893, 0.0875, 0.0860kg/s or 0.1289, 0.1163, 0.1115, 0.1076 L/s respectively. The 5 experiments that differ from theory calculation average 3.50% differ from Yuthapong-Thanakom model average 1.94%

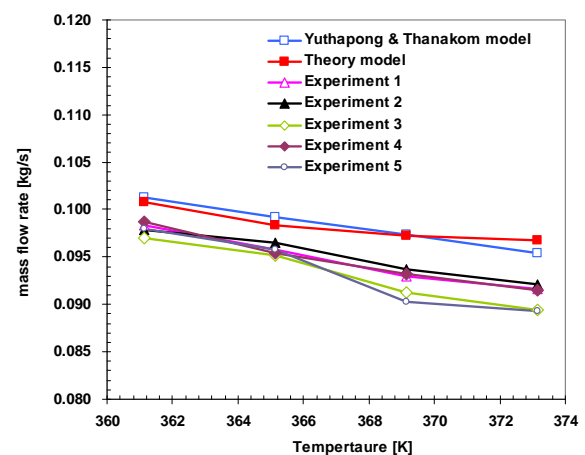


Fig 8. Relationship between the mass flow rate of distilled ethanol-water mixture and distillation temperature at absolute pressure of saturated vapor 151.325 kPa.

From the Fig 8. The absolute pressure of saturated vapor 151.325 kPa., distillation temperature at 361, 365, 369 and 373K, the mass flow rate of distilled ethanol-water mixture of theory calculation is 0.1008, 0.0984, 0.0973, 0.0968kg/s or 0.1475, 0.1410, 0.1381, 0.1366L/s respectively. The mass flow rate of distilled ethanol-water mixture of Yuthapong-Thanakom model calculation is 0.1013, 0.0993, 0.0973, 0.0954 kg/s or 0.1488, 0.1433, 0.1381, 0.1330L/s respectively. The average mass flow rate of distilled ethanol-water mixture 5 experiments is

0.0980, 0.0960, 0.0960, 0.0950 kg/s or 0.1399, 0.1338, 0.1351, 0.1308 L/s respectively. The 5 experiments that differ from theory calculation average 2.21% differ from Yuthapong-Thanakom model average 2.19%

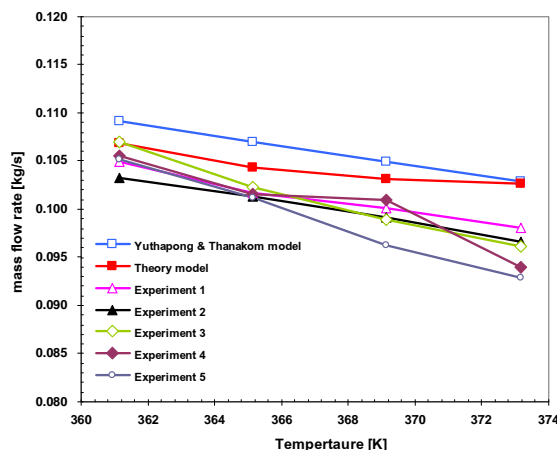


Fig 9. Relationship between the mass flow rate of distilled ethanol-water mixture and distillation temperature at absolute pressure of saturated vapor 161.325 kPa.

From the Fig 9. The absolute pressure of saturated vapor 161.325 kPa., distillation temperature at 361, 365, 369 and 373K, the mass flow rate of distilled ethanol-water mixture of theory calculation is 0.1069, 0.1043, 0.1032, 0.1026 kg/s or 0.1640, 0.1571, 0.1539, 0.1524 L/s respectively. The mass flow rate of distilled ethanol-water mixture of Yuthapong-Thanakom model calculation is 0.1092, 0.1070, 0.1049, 0.1029 kg/s or 0.1701, 0.1643, 0.1586, 0.1531 L/s respectively. The average mass flow rate of distilled ethanol-water mixture 5 experiments is 0.1094, 0.1059, 0.1042, 0.1002 kg/s or 0.1708, 0.1613, 0.1567, 0.1459 L/s respectively. The 5 experiments that differ from theory calculation average 1.79% differ from Yuthapong-Thanakom model average 1.13%

6. Conclusion

The initial concentration of ethanol-water mixture, reactant mixture, is inversely proportional to boiling temperature. When reactant mixture is heated from boiling temperature of ethanol to boiling temperature of water, the concentration of distilled ethanol-water mixture is decrease due to the increasing of distillation temperature. The deviation of the result which calculated from 10 and 30%v/v initial concentration were 6.64% and 11.80% respectively.

The mass flow rate of distilled ethanol-water mixture is directly proportional to absolute pressure of saturated vapor. When saturated vapor is inputted to pot still continuously during distillation temperature range, the mass flow rate of distilled ethanol-water mixture is decrease due to the decreasing of ethanol concentration in pot still to 0%v/v and the distillation temperature is increase to boiling temperature of water, 100°C.

At the same temperature, the saturated vapor at absolute pressure at 161.325 kPa effect to the highest mass flow rate of distilled ethanol-water mixture. The average experiment at distillation temperature 361, 365, 369, 373 K which differs from theory calculation 3.61, 3.50, 2.21, 1.79% respectively differs from Yuthapong-Thanakom model calculation 0.56, 1.94, 2.19, 1.13% respectively. Yuthapong-Thanakom model has more precision than theory calculation and able to useful for developing distillation system.

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