J. Indian Chem. Soc.,
Vol. 96, May 2019, pp. 615-621

An experimental study of batch distillation of benzene-toluene binary mixture: Study of heat supply and reflux ratio effects on distillate

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Manuscript received online 27 March 2019, accepted 13 April 2019

In this study, an experimental approach was taken for optimizing the heat supply and reflux ratio for benzene-toluene binary mixture by batch distillation. 10 liters of binary mixture of benzene and toluene were taken for batch distillation. The heat supply and reflux ratio are two major and important factors for deciding the distillation process cost. Therefore the experiments were performed at various heat supply and at various reflux ratio. It was observed that heat load 1 kW produced best separation of benzene. With the different reflux ratio, the distillate also changes and with decreasing in reflux ratio distillate decrease, 3:1 found the best reflux ratio among all what experimented. The maximum separation was found 0.995 mole fraction of benzene in distillate. The distillation column setup was as a batch setup and containing eight trays. All the experiments were performed at atmospheric pressure.

Keywords: Benzene-toluene mixture, distillation column, heat supply, reflux ratio, batch process.

Introduction

There are many separation techniques used in the chemical process industries, distillation is one of them and mostly used separation technique. The principle of distillation is based on the distribution of the substances between two phases[1–4]. The two phases are liquid and gas phases, hence all the substances must be present in both phases and both phases must have different concentration. With all the complexity distillation always the most used and important technique in chemical industries[5–9]. Continuous, batch, or semi-batch, all three types of distillation methods are used in industries. The process is taken as per the requirement of product, for high quality with low quantity always batch process is preferred. The desired purity at lower cost is always a challenging issue for distillation due to its multivariable interaction, non-stationary behaviour, process nonlinearities with highly dynamic disturbances for continuous process inside the column[10]. The cost and quality of product highly and majorly depend over reflux ratio and heat duty of reboiler.

In past, many strategies were proposed by several authors to optimize the process of binary homogenous distillation. In those proposals like step-wise constant reflux ratio[11], zero and total reflux periods[12], maximum economical profit formulations[13], maximum distillate problem solution[14–16] were discussed in details. Authors[17,18] were developed a model of operating cost and operating profit of benzene-toluene batch distillation by optimized the reflux ratio. They defined optimum reflux factor as a function of capital cost and energy price. Many processes have been reported[19–22] but all not meet with the industrial process due to the high cost of operating. Benzene has wide applications primarily in the manufacturing of styrene, cumene, cyclohexane, polymers, in gasoline as a solvent and etc. and also potential air pollutants[23–25]. Toluene also has a wide range of applications in the manufacturing of phenol, polyurethane, benzyl alcohol, benzoic acid, etc.[26,27]. As there were few research articles are available for separation of benzene-toluene by batch distillation process and its feasibility. This work focused on the distillation of a homogeneous binary mixture to separate an equimolar benzene and toluene mixture. In this work, researchers established the effects of heat supply and reflux ratio variations on the separation of a pure component from the binary mixture.

Materials and methods:

Materials:

High quality lab grade chemicals were used to carry out the distillation process. Both the chemicals benzene and toluene were supplied by Riedel-de Haen and were used as received.

Experimental setup:

The experimental setup shown below in Fig. 1. The experimental setup was provided by manufacturer Armfield (UOP3BM model) was a batch process setup. It has self-contained facilities consisting of two interconnected units; a floor standing process unit and a bench-mounted control console. The column was made up of two glass sections with all eight sieve plates of diameter 50 mm. The column was vertically arranged for counter current liquid-vapour flow and column was protected from heat loss during the process. Inside the column, each plate has central supported and incorporated a weir and downcomer. These arrangements create a liquid seal between successive stages. The reboiler was manufactured from 316 stainless steel and consist of a flame proof immersion heater. A water cooled coil shell condenser mounted over top of the column and fitted with an insulated jacket. The condensate was collected in a

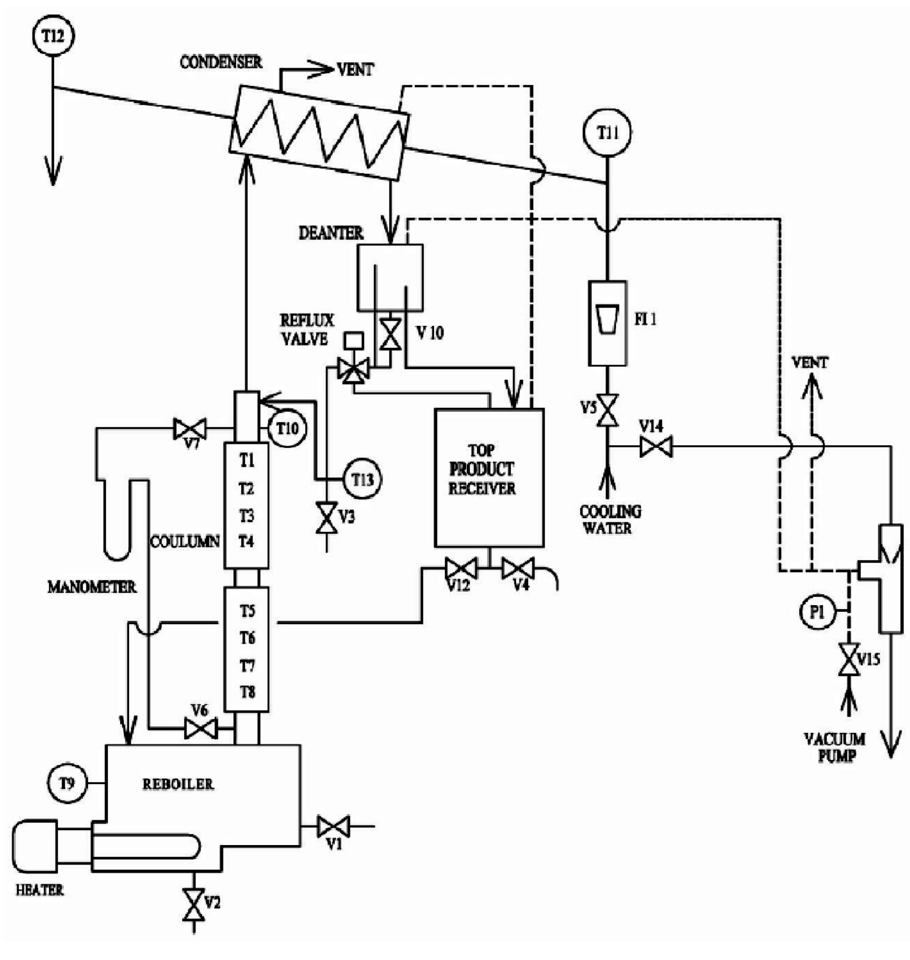

Fig. 1. Experimental setup.

phase separator (decanter) which was used also as a by-pass for reflux and final product vessel. Thirteen thermocouple sensors are used to monitor the temperature within the setup. Each sieve plate has a temperature sensor to measure the liquid temperature on plates. The total pressure drop within the column was measured by U tube manometer installed with the setup. A hand-held refractometer was used to measure the refractive index of the distillation mixture. Heat load was controlled from the console and reflux also. The temperature distribution for all thirteen points could be read from the output panel of the console.

Experimental procedure:

Ten liters mixture of toluene and benzene of 50:50% by mole has been fed into the reboiler. The heat flux supplied to boil the liquid mixture at 1 atmospheric pressure. The water as a condenser coolant was used for condensing the gaseous phase to the liquid phase. The water flow rate had controlled by rotameter, for all the runs flow rate fixed at 5000 cc. The reflux ratio has set from the console and heat flux also set from the console. The temperature was recorded from the display on the console for all the thermocouples. Each experiment ran to achieve the equilibrium condition. After achieving equilibrium condition heat supply shutdown and leave the material to cool to the room temperature. When the setup reached room temperature, the sample from the product vessel and from bottom reboiler were taken out. The cooled sample of distillate and bottom product were used for measuring the refractive index uniformly at room temperature. After the measurement, the sample again returned to the reboiler to maintain the initial concentration same in reboiler for each run. The experiments are reported for different power supplies like 0.50, 0.75, 1.00, 1.25 and 1.50 kW. At each heat supply a range of reflux ratios 3:1, 2:1, 1:1, 1:2 and 1:3 had experimented. A refractive index plot of benzene-toluene mixture has been plotted before the distillation as shown in Fig. 2.

Results and discussion

Distillation is a process where heat is needed for boiling the mixture, the heat supply variation affects the boiling time and boiling nature. With the increase in heat supply, the column gas-liquid mixture behaviour changed from gentle movement to the liquid flooding in the column followed by local violent, gentle foaming over the whole tray, violent foaming

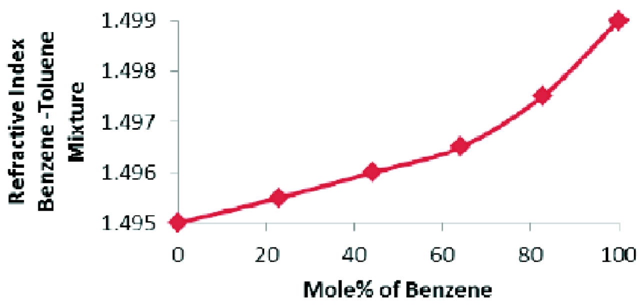

Fig. 2. Refractive index of benzene-toluene mixture in terms of benzene mole percentage.

over the whole tray. The experiments were reported for different power supplies like 0.50, 0.75, 1.00, 1.25 and 1.50 kW. It was observed that at low heat load the mole fraction of benzene was lower in the distillate, at high heat load the purity of benzene increased and showed no more variation with more heat supply. Fig. 3 below illustrates the behaviour of distillation at heat supply 0.50 kW. The column fluid flow behaviour was smooth and gentle without any violent noise.

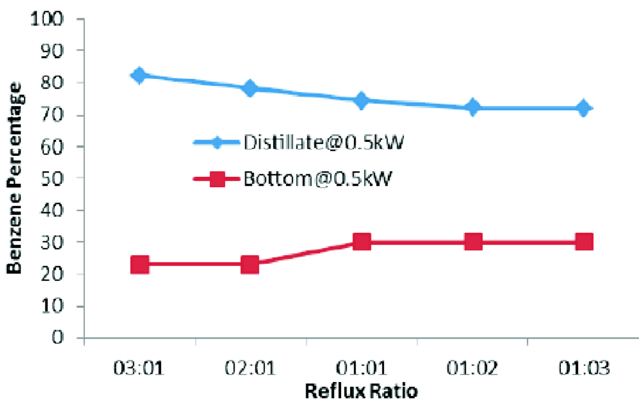

Fig. 3. Distillation of benzene-toluene mixture at 0.50 kW.

The maximum mole fraction of benzene in distillate reached around 0.80 and gradually decreased with the decrease in the reflux ratio (L/D), hence the mole fraction of benzene increased in the bottom with the decrease in reflux ratio. The Figs. 4, 5, 6 and 7 below illustrates the behaviour of distillation at heat supply 0.75 kW, 1.00 kW, 1.25 kW and 1.5 kW. With the increase in heat load from 0.50 kW to 0.75 kW, the mole fraction of benzene increased in distillate and decreased in the bottom, also the column fluid flow behaviour shifted from the gentle flow to the violent in nature over whole tray.

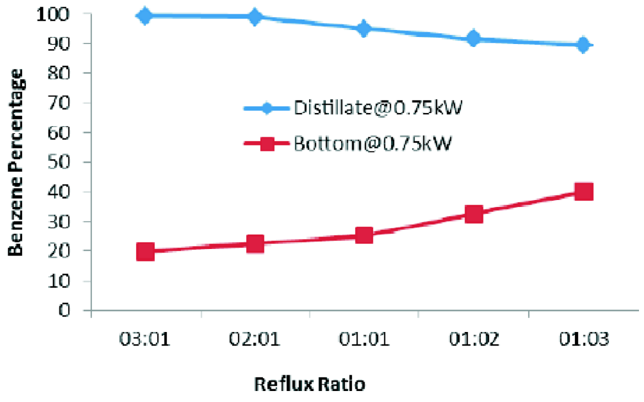

Fig. 4. Distillation of benzene-toluene mixture at 0.75 kW.

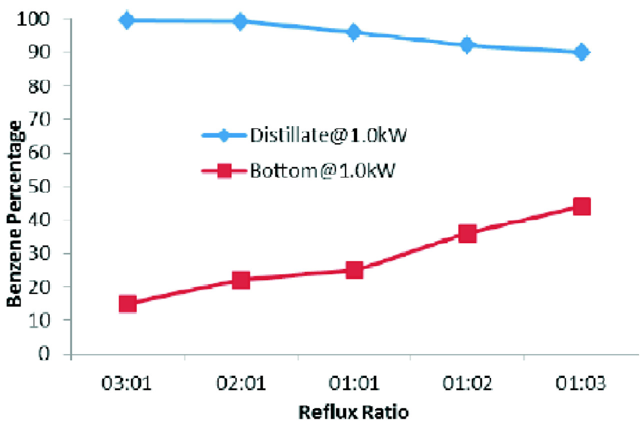

Fig. 5. Distillation of benzene-toluene mixture at 1.0 kW.

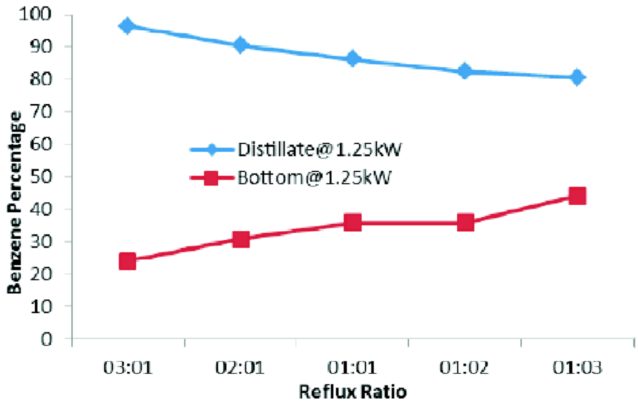

Fig. 6. Distillation of benzene-toluene mixture at 1.25 kW.

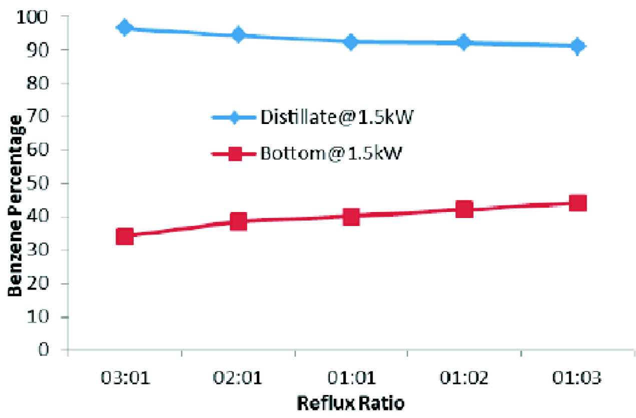

Fig. 7. Distillation of benzene-toluene mixture at 1.50 kW.

The mole fractions of benzene in the distillate and bottom are nearly the same at heat load 0.75 kW and 1.00 kW as shown in Figs. 4 and 5. There were no significant difference at any other reflux ratio, at the lower reflux ratios, the mole fraction of benzene decreased.

In further increase in heat load 1.25 kW and 1.50 kW, the mole fraction of benzene decreased in distillate and increased in the bottom as compared to heat load 0.75 kW and 1.00 kW. This behaviour was due to the increase in temperature in the column due to the high heat load reached the average temperature of the mixture. The fluid movement inside the column and over the tray become violent and foaming over the whole tray. There may two reasons behind decreasing the distillate mole fraction of benzene, first due to high reboiler temperature more toluene evaporated with benzene compared to the lower heat load. The second, due to foaming, the gas phase of benzene trapped inside the liquid phase available on each tray and convert to liquid not able to reach to the condenser. Figs. 6 and 7, as shown above, illustrates the behaviour of separation of benzene from the benzene-toluene mixture at heat load 1.25 kW and 1.50 kW respectively. The distillate mole fraction decreased with the decrease in reflux ratio more rapidly at heat load 1.25 kW, while at heat load 1.50 kW variations were less. At all the five heat loads (Figs. 3 to 7) at reflux 3:1 the distillate mole fraction of benzene are higher and as the reflux ratio decreased the mole fraction of benzene decreased. In similar fashion the mole fraction of benzene at the bottom at reflux ratio 3:1 is lowest and as the reflux ratio decreased the mole fraction of benzene increased. The Figs. 8 and 9 are a comparative analysis of all five different loads, Fig. 8 was a comparative graph of the distillate mole fraction of benzene at all five loads

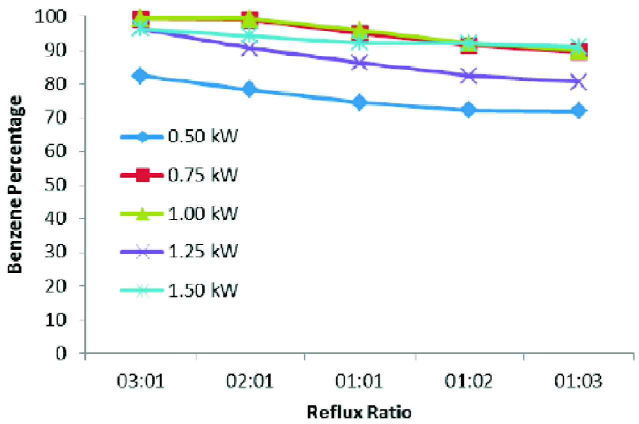

Fig. 8. Distillate mole fraction of benzene in the distillation of benzene-toluene mixture at different heat load.

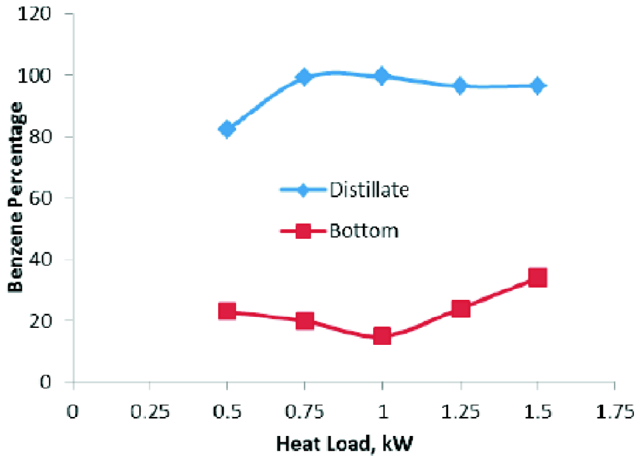

Fig. 10. Mole percentage of benzene in distillate and bottom at fixed reflux ratio 3:1.

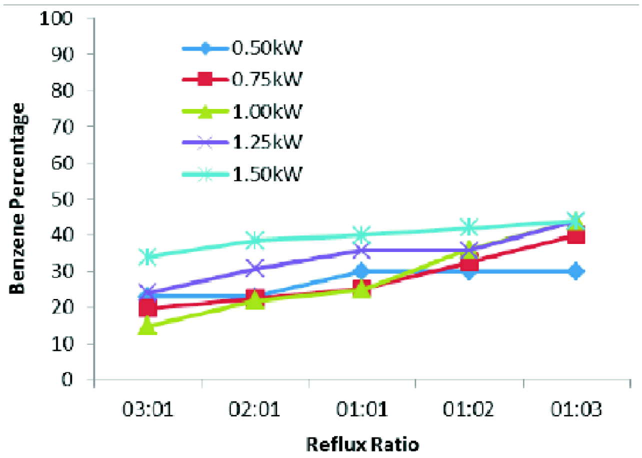

Fig. 9. Bottom mole fraction of benzene in the distillation of benzene-toluene mixture at different heat load.

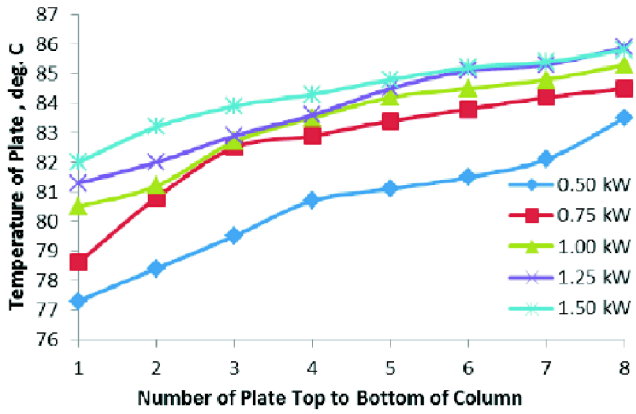

Fig. 11. Temperature distribution inside the distillation column at different heat load.

and it was very clear that there were no significant differences in the results of 0.75 kW and 1.00 kW. From Fig. 8 it was also very clear that heat load above 1.00 kW reduced the distillate mole fraction of benzene.

Fig. 9 was describing the mole fraction of benzene at the bottom or in a mixture of feed. It was very clear that at heat load 0.75 kW and 1.00 kW the presence of benzene at the bottom was similar and lowest for almost all reflux ratios. The variation of the distillate mole fraction of benzene also can be seen similar for all five loads with the reflux ratio variation in both Figs. 8 and 9. The results discussed above conclude that, the reflux ratio 3:1 showed better results compared to all other reflux ratios experimented for all five heat loads. The Fig. 10 is describing the effects of the heat load of distillation at reflux ratio 3:1. The pattern of the plot showing that the heat load near 1.00 kW is much enough to achieve a higher mole fraction of benzene at distillate. Temperatures of feed and of plates in the column were recorded, every plates and feed tank were connected with temperature measuring thermocouples. Temperatures were recorded from the display of the control console. The Fig. 11 illustrated the temperature distribution inside the distillation column at different heat load were the temperatures when the sample of distillate and bottom product were taken for benzene mole fraction analysis. The temperature distribution of heat loads 0.50 kW and 0.75 kW showed much difference between the plate 1 and 8, temperatures difference between each plate are about uniform from top to bottom of the distillation column. But for heat load 1.00 kW and upward the temperature difference of the distillation column is less and much uniformity in the temperature of different plates. The Fig. 12 showed how temperature differences change with variation of heat loads. It was very clear from the Fig. 12 that as heat load was increased the temperature difference decreased this because of the more rapid vapor formation of volatile component benzene. The behaviour of boiling of the benzene-toluene mixture was predicted very well in Fig. 13. With the increase in heat load the boiling rate increased rapidly, so the nature of fluid flow inside the distillation column shifted gentle flow to violent foaming on the whole tray. The boiling rate at heat load 1.00 kW changed the fluid movement inside the distillation column and it behaves somewhere between local violent and gentle foaming over the whole tray. Therefore

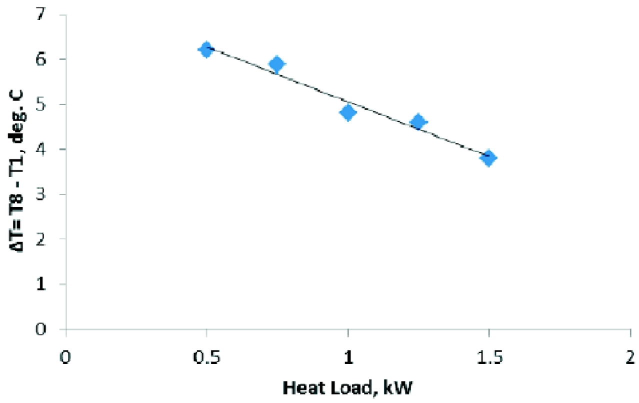

Fig. 12. Column temperature difference at different heat loads.

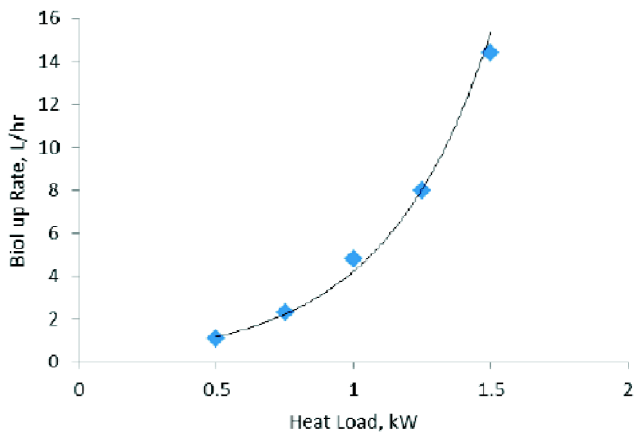

Fig. 13. Boil up rate of distillate at different heat loads.

the distillate mole fraction of benzene increased at 1.00 kW. Heat load above 1.00 kW changed the fluid movement inside the distillation column and it started to behave like violent foaming over the whole tray. Hence the distillate mole fraction of benzene decreased after 1.00 kW heat load.

To ensure the results validity few steps were taken during analysis and experiments. The value for every single point was the mean of three different carefully taken readings. An experiment of reflux ratio 3:1 at all five heat loads were repeated to ensure the repeatability of the experimental data. Fig. 14 illustrated the repeatability of experimental data and

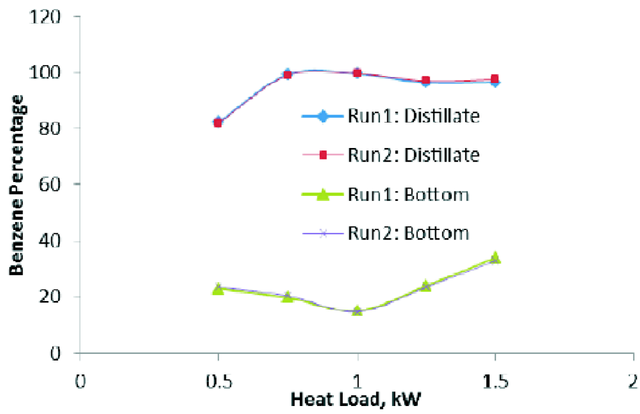

Fig. 14. Repeatability experimental result at reflux 3:1.

it was very clear that the experimental data were highly reproducible. The deviation of each reading of a refractive index from its mean was under ±0.0002. The deviation of benzene mole percentages from its mean was under ±0.025%.

Conclusions

The distillation of benzene-toluene mixture depends on heat supplied and a reflux ratio of distillation. From the result and discussion, it was clear that heat load 1 kW to 1.25 kW have an optimum result. With the different reflux ratio, the distillate also changes and with decreasing in reflux ratio distillate decrease, 3:1 found the best reflux ratio among all what experimented. From all the results and discussions it can be concluded that the batch distillation depends on heat load supplied and reflux ratio.

Acknowledgement

This work was supported by the King Khalid University-Abha, Saudi Arabia, under grant R.G.P. 1/144/40. The research team would like to express their gratitude and acknowledge to the Deanship of Scientific Research, King Khalid University, for the support in conducting this study.

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