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Improvement in recovery of Dimethyl formamide (DMF) by Short path distillation unit

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Abstract: N, N-dimethyl formamide (DMF) is a well known solvent of many hydrophobic organic compounds. Main consumer of DMF is the polymer industry, used in production and processing of polyamide and acrylic fibers, polyurethane products, synthetic leather, films, coatings. Effluents from such type of industry containing 10-25wt. % DMF was produced and needed to dispose and recover the DMF before discharge, used recovered DMF as raw material in same industry. The conventional method for recovering of DMF is distillation, the disadvantage of which is large energy consumption because of need of 3-4 distillation columns. The general aim of our work is to suggest process modifications that enable the capacity increase of an existing DMF-water separation, decrease the energy and cost.
Keywords: N, N Dimethyl formamide, Short path distillation unit, vacuum system.

I. INTRODUCTION

N, N-Dimethyl formamide (DMF) is a well-known solvent of many hydrophobic organic compounds. Production and processing of polyamide and acrylic fibers, films, coatings and wire enamels all require the usage of DMF. It is also used as crystallization medium in the pharmaceutical industries but the main consumer of DMF is the polymer industry.

Effluents come out from such type of industry containing which having dimethyl formamide ,Water, Dimethyl amine, Formic acid, Other volatile and non volatile impurities depending upon which industry's Effluent .it is hazardous in nature. It create problem related to abdominal paining, vomiting and kidney, heart, liver problem according to one survey it is one of the carcinogenic solvent. According to NFPA (National Fire Protection Association) diamond degree of hazard to health is 2. It is hazardous in nature so not directly dispose into river or sea.so it must need recover the DMF.

Boiling point of DMF is **150˚C** while it starts to decompose at **100˚C.** At atmospheric pressure, boiling point of water is 100˚C so for start boiling, temperature requirement above 100˚C but at same time decomposition of DMF take place which directly decrease concentration i.e. quality of DMF. The conventional method for recovering of DMF is distillation, the disadvantage of which is large energy consumption because of need of 3-4 distillation columns also capacity is lesser. Distillation is not possible at atmospheric pressure so for such type of separation, vacuum distillation is one of the options in the case of distillation.

The solvent spinning process that is used for the production of polyamide fibers uses large amounts of DMF. The polymer is dissolved in DMF resulting in a solution that is suitable for extrusion through a spinneret. The extrusion is followed by the precipitation of the polymer fiber with the help of an aqueous bath. In the last step of the technology the DMF is to be recovered from an aqueous solution, so it can be used again as a solvent.

A. Experiment

We performed experiment on short path distillation unit which is combination of three system
(1) Heater and condenser (2) rotor and (3) water jet ejector (vacuum system). Here we fixed the vacuum 200 mm Hg based on VLE data and fixed rotor speed 13 rpm. As condensing media we use the ice in condenser.

By taking 15, 20, 30 mole % dilute DMF, distilled in SPDU till bubbling is not completed. Temperature of heater kept about at its boiling point±1˚C at given pressure. After completion of bubbling ,remove the solution from system measure the the concentration and again used previous residue as feed in next stage again increase the temperature and same procedure carried out till 0.99% concentration is not obtain.

B. Sample Analysis.

We need to measure concentration of distillate and residue at the end of experiment because short path distillation unit is batch type. we use 10%,20%,……,100% concentration of DMF solution and find the refractive index of each solution by using refractometer

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and draw the graph of refractive index vs. DMF mole fraction. This graph is can be used for find concentration of DMF when refractive index of distillate and residue known which is find by using refractometer.

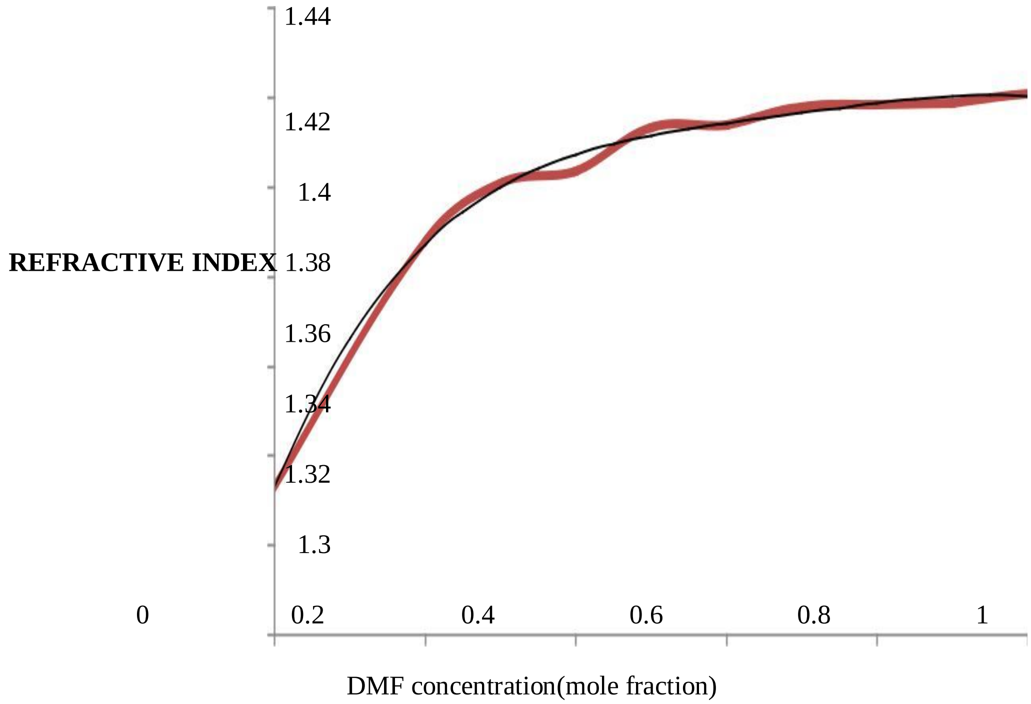


Figure 1: Refractive index VS mole fraction of DMF

Vacuum require for separation of DMF-water Binary system can be fixed based on VLE data (T-XY) diagram which is obtained from CHEMSEP simulator.

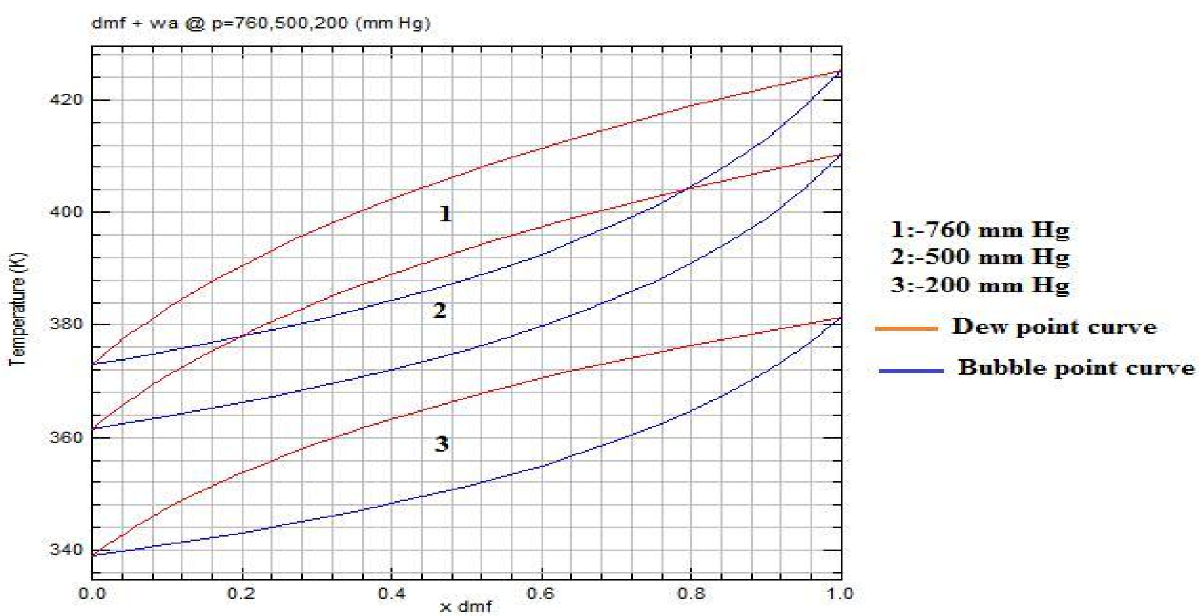


Figure 2: Experimental data of DMF-water system

From above diagram it is clear that operate 1st column at 200mmHg absolute pressure without decomposition with maximum recovery.

C. Experimental Result

Here we derived data for ,15%20%,30% and generate the graph as shown in result for comparison. We showed only experimental data of 15 mole% DMF solution.

			Residue			Distillate	
T(°C)	P(absolute)	vol.(ml)	R.I.	Mole fraction	vol. (ml)	R.I.	Mole fraction
80	200	104	1.381	0.17	11	1.344	0.04
90	200	78	1.397	0.253	27	1.349	0.058
93	200	63	1.399	0.27	13	1.354	0.074
96	200	42	1.414	0.6	17	1.364	0.109
98		30	1.421	0.99	15	1.38	0.17

Table 1: Experimental data of DMF-water system

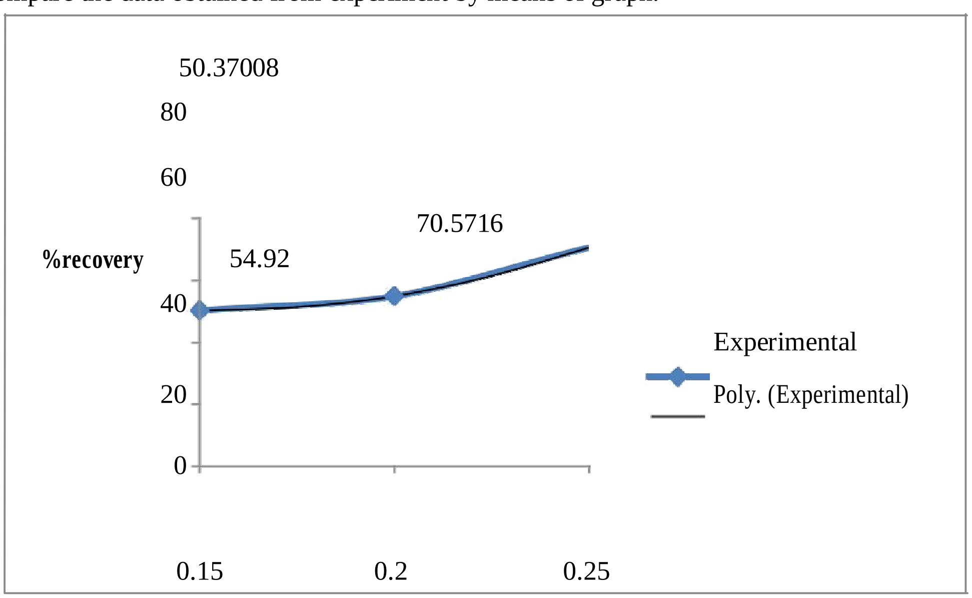
Similarly we collected all distillate coming from above experiment and again used as feed again, it is distilled till upto feed concentration is not achieved for checking possible in improvement in recovery of DM

			Residue			Distillate	
T(°C)	P(absolute)	vol. (ml)	R.I.	Mole fraction	vol. (ml)	R.I.	Mole fraction
80	200	52	1.367	0.12	13	1.345	0.044
87	200	29	1.3715	0.135	12.5	1.339	0.02
90	200	24	1.391	0.224	12.5	1.343	0.036

Table 2: Experimental data of DMF-water system

II. RESULT AND DISCUSSION

Here we compare the data obtained from experiment by means of graph.



Feed molefraction

Figure 3: %recovery vs. mole fraction of feed

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%Recovery of DMF from effluent increases with increase in mole fraction of feed.

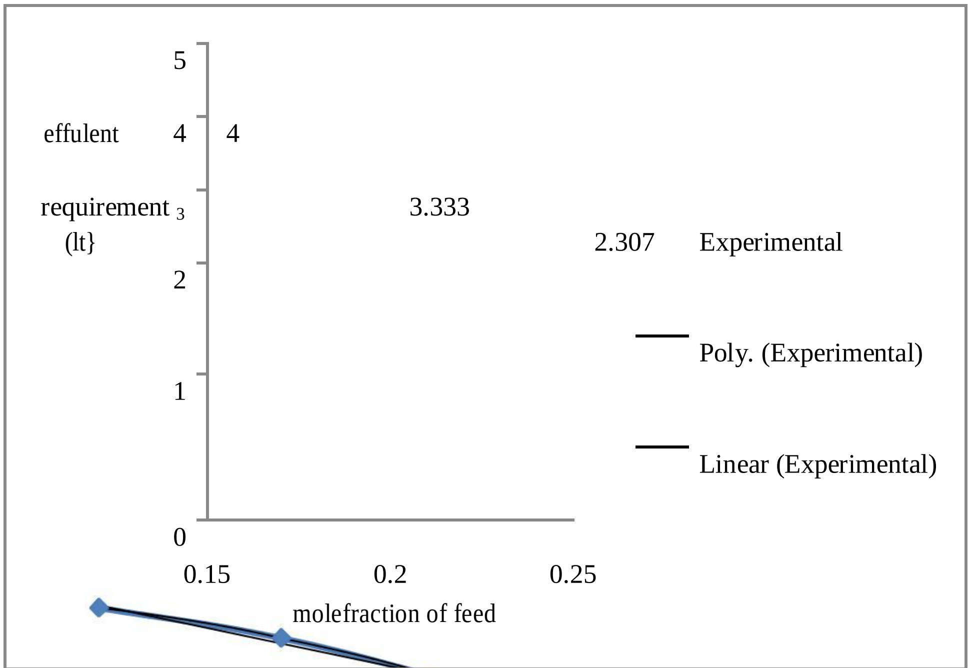

Figure 4: Effluent requirement (lt) vs. mole fraction of feed

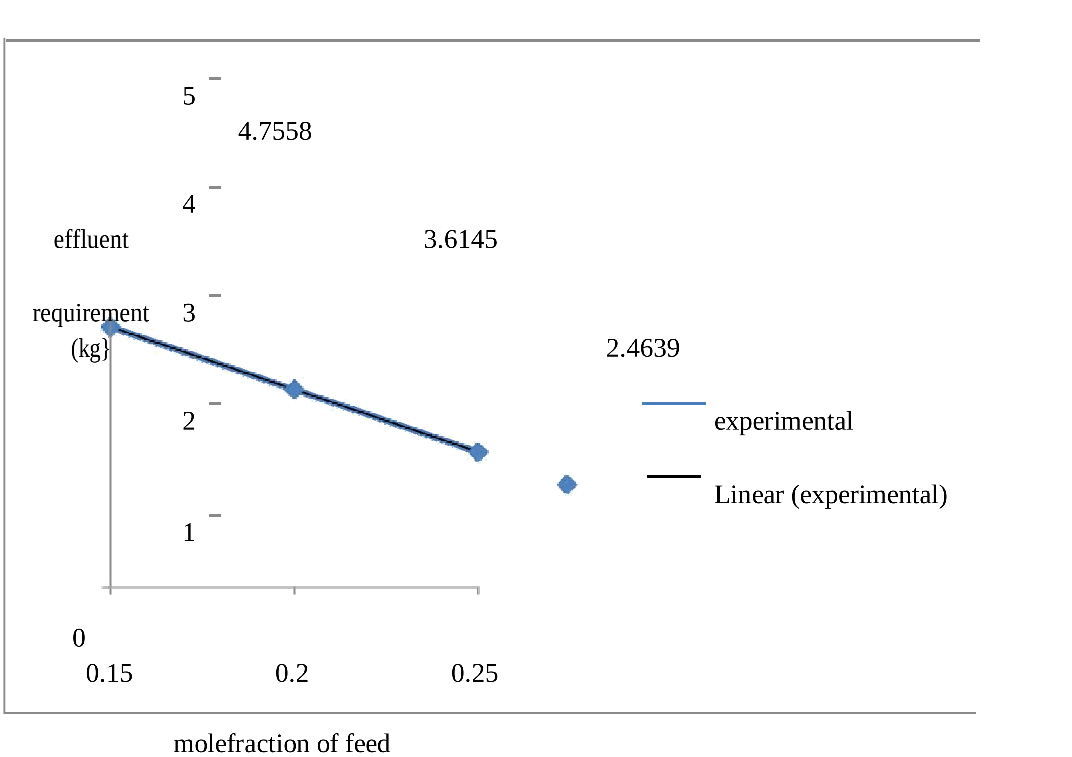

Figure 5: Effluent requirement (Kg) Vs. mole fraction of feed

1) Amount of effluent requirement for production of 1 kg (or liter) DMF decrease with increase in feed concentration which is derived based on material balance in Excel.

A. Improvement In Recovery Of Dmf From Effluent

It is done by addition of 1 extra column operated at vacuum for increase the concentration of distillate coming out from main column upto feed concentration and recycle it to main column.

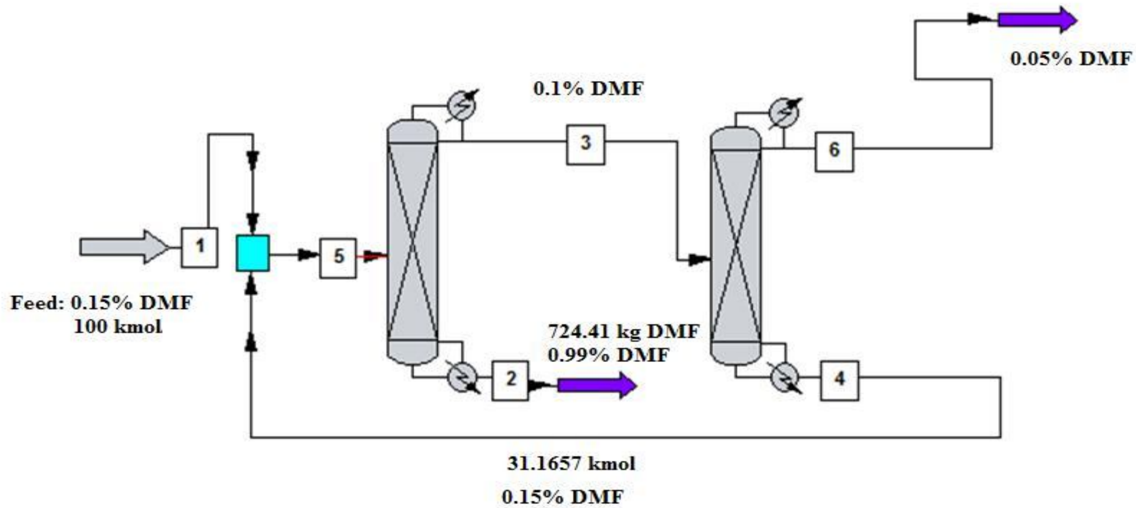

Figure 6: DMF-water separation system with extra unit

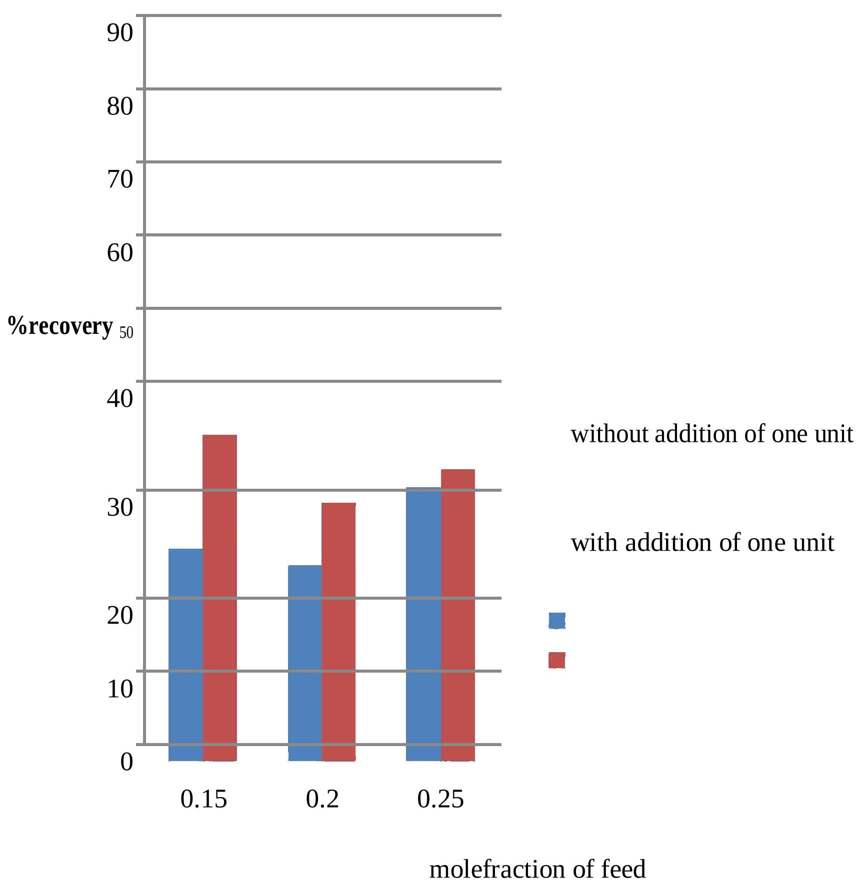

Figure 7: %recovery Vs molefraction of feed

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By addition of extra unit, % recovery of DMF is increases and increment in recovery is more for less concentration of feed compare to high concentration feed. It is mainly because of as feed concentration increases, separation become easier and maximum separation of DMF taking place in 1^{st} stage while for less concentration feed separation of DMF is lesser in 1^{st} unit so extra unit required is more preferable.

III. CONCLUSION

Short path distillation method is best feasible method for separation of DMF-water separation. % recovery can be increased by addition of one distillation column which is increase concentration of main column distillate. Because of increase in feed flow rate by means of reflux, pure DMF product flow rate is also become increase that ultimately increase capacity of column means it increase cost of equipment in term of fixed cost. Indirectly it decreases water pollution because of maximum recovery of DMF from process effluent.

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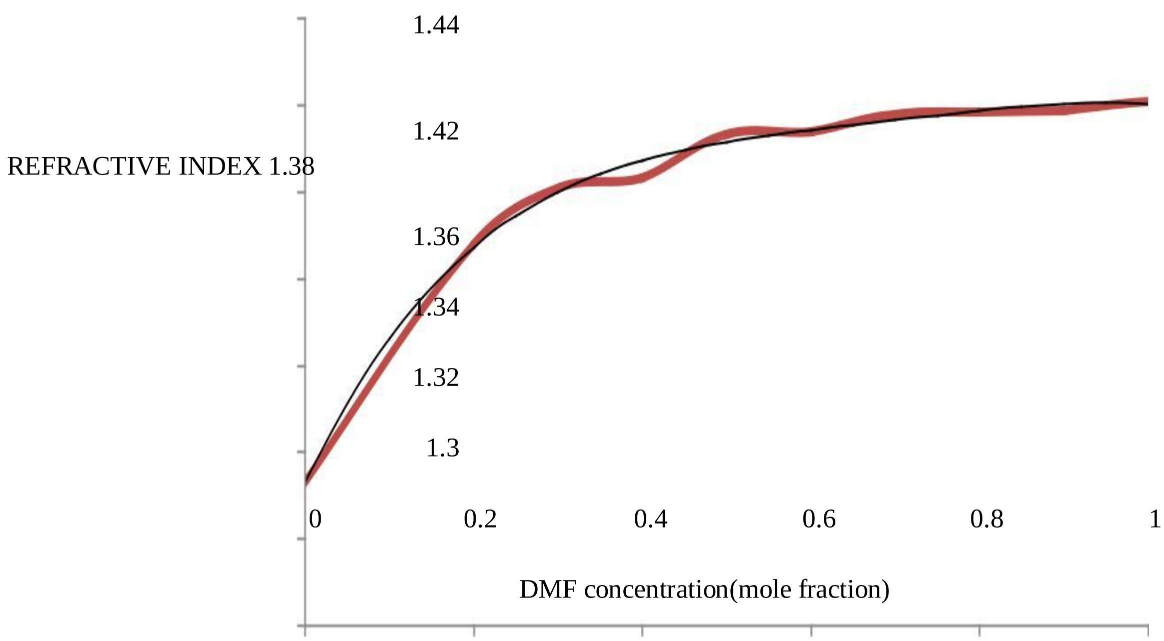

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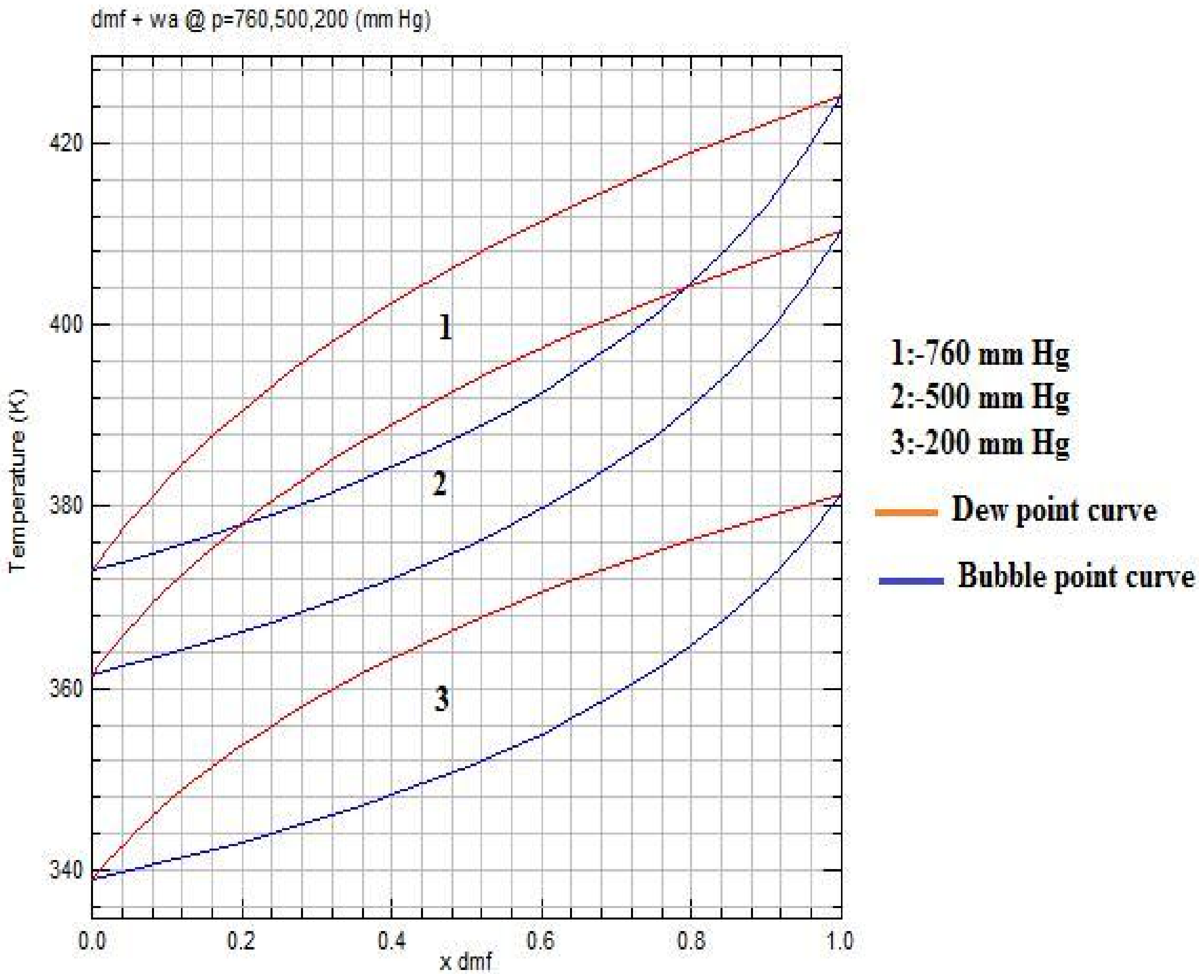

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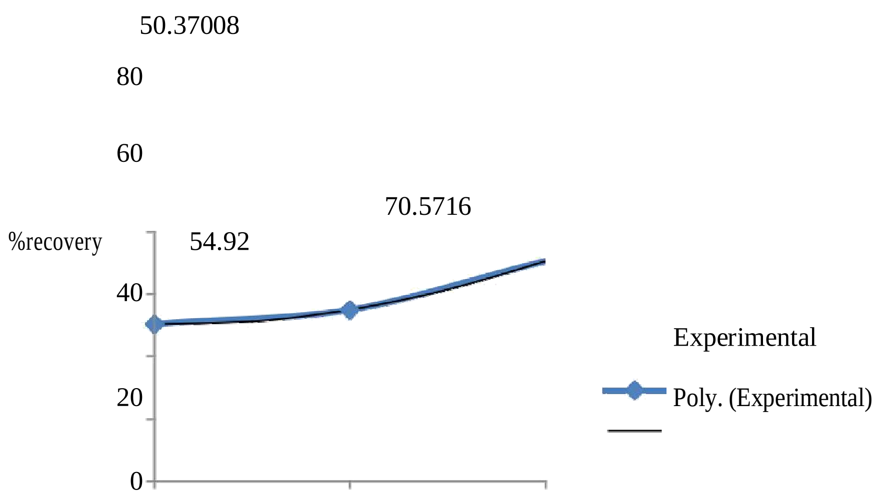

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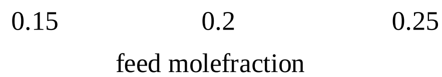
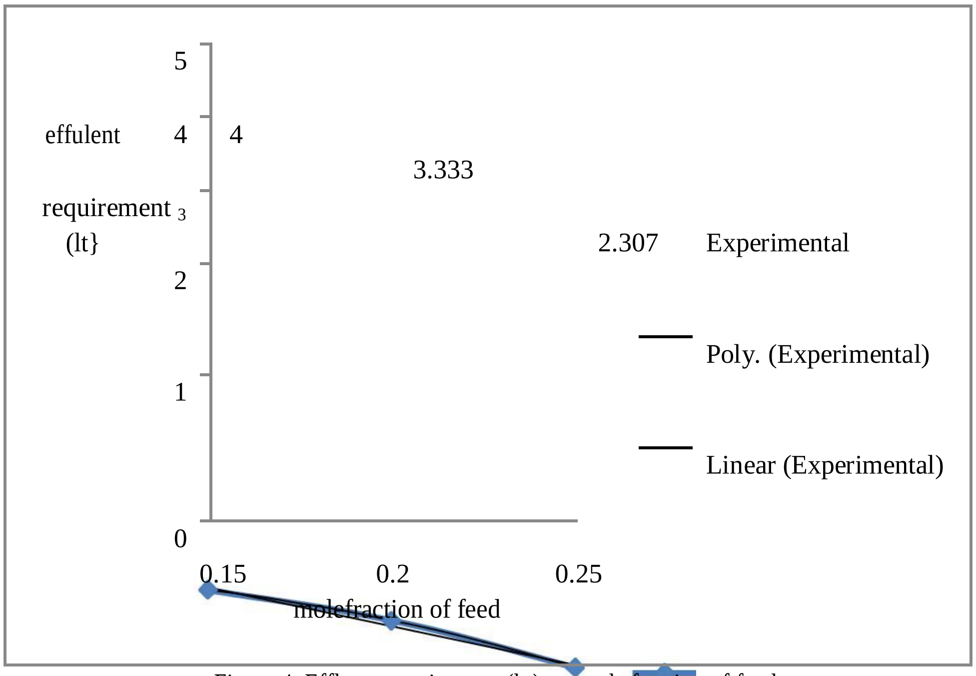


Figure 3: %recovery vs. mole fraction of feed



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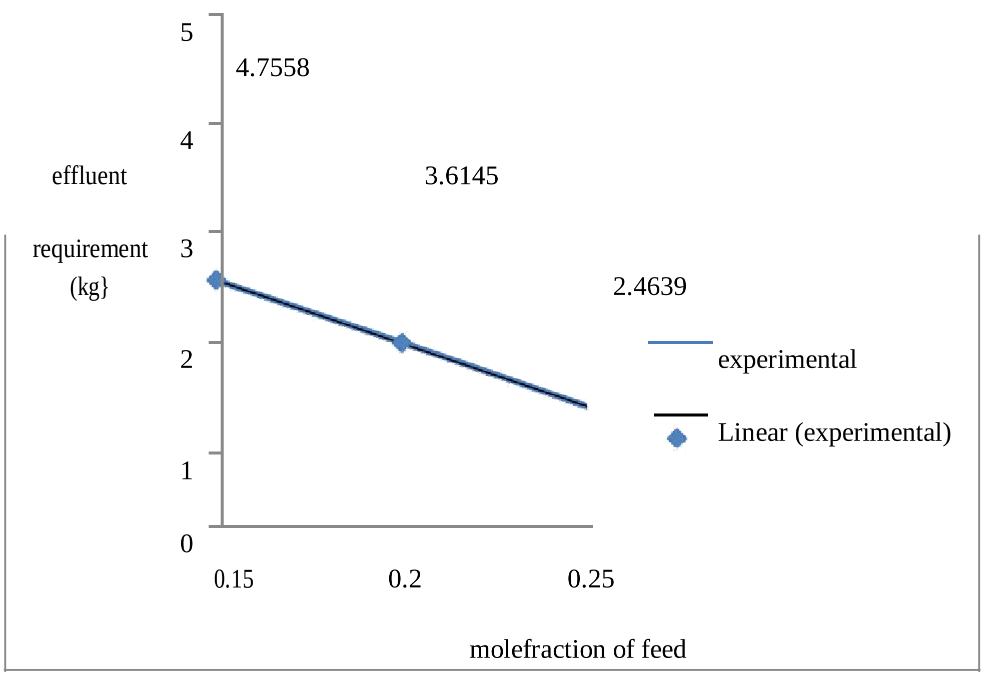

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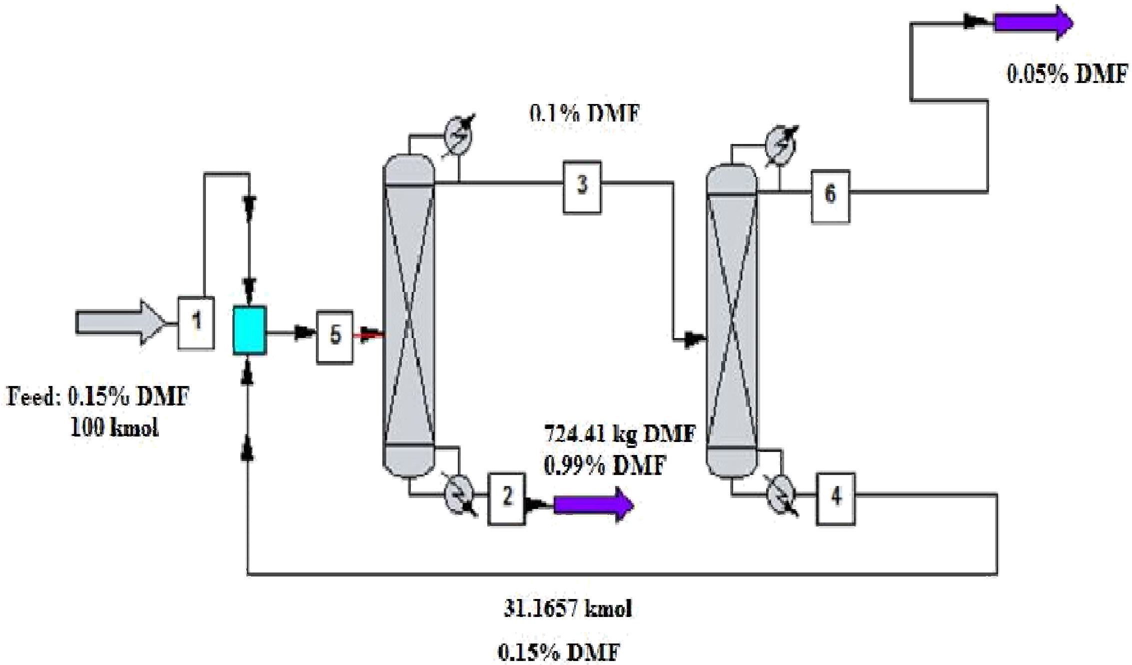

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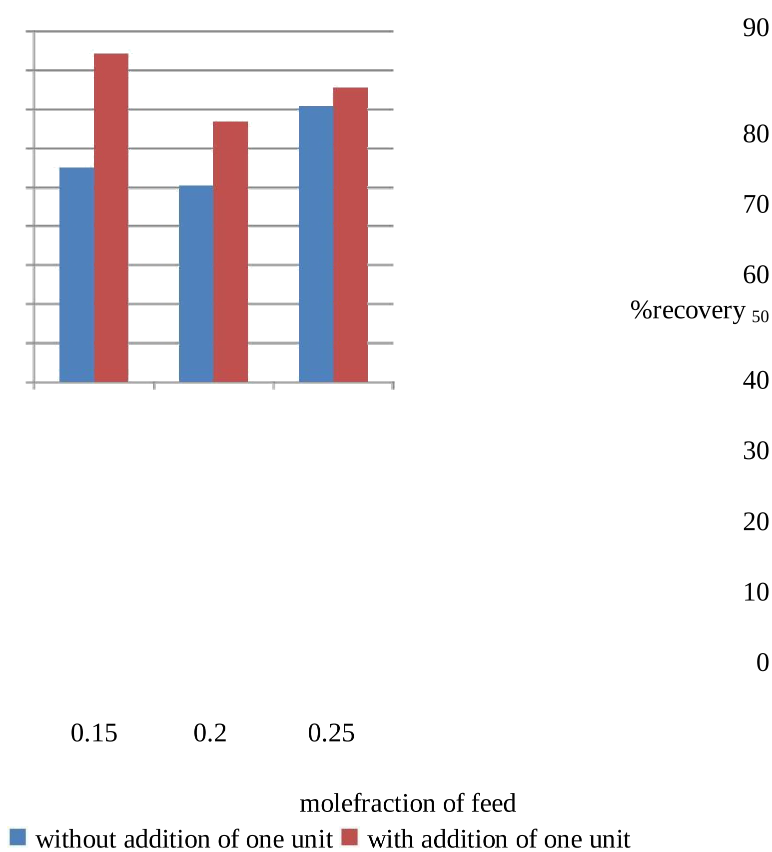

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