UPWARD SINGLE DROP MASS TRANSFER IN WATER

CHE213: Mass Transfer and Separation Processes

Unit Operations Lab: Group 24
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INTRODUCTION

- Mass transfer from a single drop is essential for understanding liquid-liquid extraction operations.
- The mass transfer behavior during a drop's journey reflects interfacial dynamics, internal circulation, and solute resistance across phases.
- This experiment originally helped estimate mass transfer coefficient, effects of drop size, etc.
- Such single-drop studies offer fundamental insights applicable to large-scale extraction operations.
- They also help validate theoretical models under controlled and simplified conditions.
- These insights are vital for designing efficient extractors in chemical industries where droplet behavior governs throughput and selectivity.

EXPERIMENTAL SETUP

- In the standard setup, a column is filled with pure Methyl Iso Butyl Ketone (MIBK) (continuous phase). Aqueous acetic acid solution is introduced dropwise from the top, forming the dispersed phase.
- Our innovation reverses the system: the column is filled with water, and a 2N acetic acid–MIBK solution is injected from the bottom at a constant rate. This creates rising organic-phase droplets in water, simulating an extraction scenario often encountered in industry.

INNOVATIVE ASPECTS

- Our experiment explores mass transfer under buoyancy-driven condition (induced by density difference) instead of gravity-driven condition. This helps quantify how factors like drop formation (as the drop is now formed inside the water) , residence time, and interfacial dynamics influence the overall mass transfer coefficient in upward-moving systems.

- In the traditional setup, whole column is filled with MIBK but our setup uses water to fill the column which can save us a significant amount. We have done a cost analysis for UOP lab. We know, Column Dimensions: Diameter = 7 cm (est.), Height = 70 cm, Volume = 2693.92 cm^3.
  MIBK Density = 0.802 g/cm^3 → Mass = 2.160 kg, Cost = ₹200/kg → Cost per experiment ≈ ₹432 , Annual Cost (24 runs): ₹432 × 24 = ₹10,368

METHODOLOGY

- Prepare an aqueous solution of acetic acid and MIBK and to determine its concentrations by titration with 0.5 NaOH.
- Fill the syringe with solution prepared above and place it in the syringe pump to and connect the syringe with a small diameter pipe to the column's needle and this will function as dispersed phase.
- Fill the column with water. Note- Syringe pump needs to be started before we start filling the column with water . Fill the column with water upto the mark.
- Now, after some time the drops should start emerging from below, if the drops aren't emerging the needle may be damaged. Therefore, the needle has to be handled with care.

The diameter of the needle = 1.6 mm

METHODOLOGY

- After approximately 1 min of liquid running (from burette/syringe reading). Count the number of drops. From this, the drop radius can be calculated.
- Knowing the length of the column and the residence time for a drop, its upward velocity can be calculated.
- After sufficient liquid has been pumped from the syringe pump, (for our experiment we chose this as 30 ml) we can stop the flow.
- The acetic acid + MIBK will float above water and can be taken out by a dropper into a beaker and then we have to determine its normality.
- After this we have to empty the column by removing all the contaminated water from it and refill it.
- Now, we can change the flow rates and calculate mass transfer coefficients.

THEORY

- A rising liquid drop allows internal circulation, with nonzero tangential velocity at the interface. This internal circulation enhances mass transfer by continuously regenerating the interfacial area—a process wherein new interfacial area is created at the front of the drop and destroyed at the rear.
- As the drop ascends under buoyant force, it reaches a terminal velocity dependent on its size and the balance of gravitational, buoyant, and drag forces. This terminal velocity is impacted by internal circulation and interfacial mobility. The drag force on a rising drop can still be described by the general force balance:

$$\frac{4}{3}\pi r^3(\rho_d - \rho_c)g = \frac{1}{2}C_D\rho_c A v^2$$

, the correlations for Cd were used as used in traditional experiment.

THEORY

- Mass transfer occurs from the interior of the MIBK drop (dispersed phase) to the surrounding aqueous phase (continuous phase) due to a concentration gradient of acetic acid. The resistance to mass transfer occurs in three stages: 1) From the bulk of the drop to the interface, influenced by internal circulation. 2)Across the liquid–liquid interface, usually assumed negligible. 3) From the interface into the bulk of the continuous phase.
- These following correlations were obtained from experiments and works quite good for the case in which drop is falling. To find the mass transfer coefficients we applied similar correlations here as the traditional system.

$$k_c = 0.023 U_t (N_{Sc})_c^{-1/2}$$

$$k_d = 0.023 U_t (N_{Sc})_d^{-1/2}$$

k_c = Individual continuous phase mass transfer co-efficient, m/s

k_d = Individual dispersed phase mass transfer co-efficient, m/s

$$, N_{sc} = \frac{k_d}{\rho_d D_d}$$

OBSERVATIONS

- We did the entire experiments for three different flow rates 70, 80 , 90 ml/hr and the results can be seen below. Each was continue till 30ml of liquid was pushed through syringe pump.

FLOW RATE	NO. OF DROPS/MIN	RESIDENCE TIME OF A DROP	DIAMETER OF A DROP (in mm)	HEIGHT OF THE COLUMN FILLED (in cm)	VELOCITY (in cm/s)
70 ml/hr	93	8.75	1.548	68	7.771
80 ml/hr	104	8.12	1.565	69	8.498
90 ml/hr	111	7.63	1.599	66	8.651

RESULTS

- In syringe, acetic acid+ MIBK in each case has a normality of 2N.
- The normality of NaOH used for titration in each case is 0.5N and the indicator used in titration was phenolphthalein.
- For 1st case, flow rate = 70 ml/hr : It required 2.5 ml of NaOH to titrate 10.1 ml of acetic acid + MIBK solution collected from top. Therefore, normality after mass transfer of acetic acid into water is 0.124.
- For 2nd case, flow rate = 80 ml/hr : It required 2.4 ml of NaOH to titrate 9.9 ml of acetic acid + MIBK solution collected from top. Therefore, normality after mass transfer of acetic acid into water is 0.121.
- For 3rd case, flow rate = 90 ml/hr : It required 2.3 ml of NaOH to titrate 9.8 ml of acetic acid + MIBK solution collected from top. Therefore, normality after mass transfer of acetic acid into water is 0.117.
- The decreasing normality is suggesting that mass transfer coefficient is increasing.

RESULTS

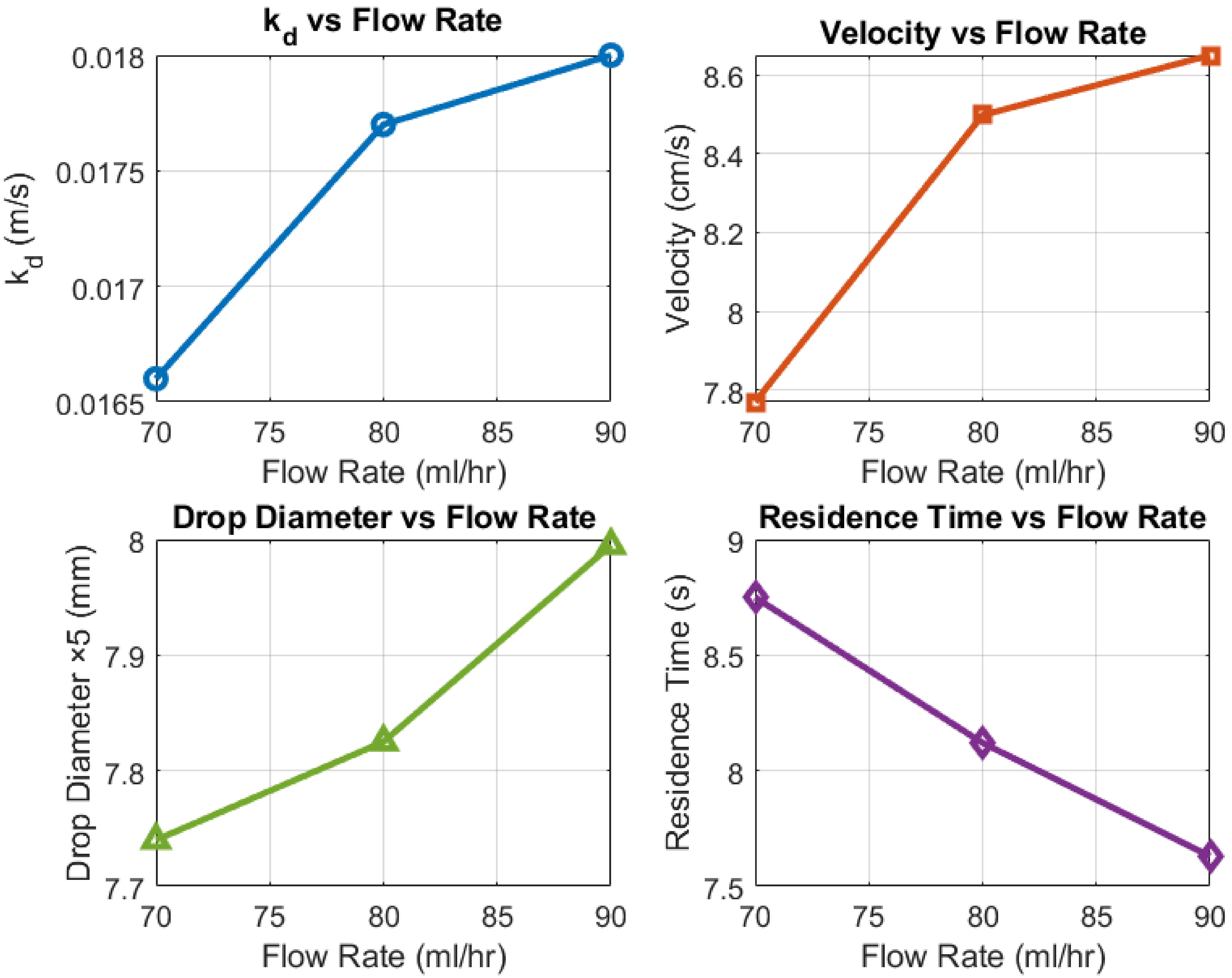
Calculating overall dispered phase mass transfer coeffcient:

- By using the correlations we can can calculate kd's (individual dispersed phase mass transfer coefficient (in m/s)) for each case.
- kd= $\left(0.0233 \cdot U_t \cdot \rho_d^{0.5} \cdot D_d^{0.5}\right)^{0.66}$, where Ut is terminal velocity (which is assumed to be constant), density of dispersed phase is 0.92 g/cm^3 and diamater of drop changes

Flow Rates (in ml/hr)	kd (dispersed phase mass transfer co-efficient)
70	0.0166
80	0.0177
90	0.0180

- With increasing flow rates the mass transfer coefficients should **decrease** as the residence time is decreasing. But this doesn't happen due to increase in surface area of the drop, which enhances mass transfer, moreover high flow rate also contributes to the increased mass transfer.

- These are the final results plotted, mainly it shows how different parameters change in this experiment with flow rate.



Errors and Issues with the Experiment

- First of all, we suggest two changes in the setup, it weren't possible for us due to time constraints. There should be a stopper above the needle we used to inject acetic acid, because whenever the column is filled and we try to refill the syringe with acetic acid the water goes in the bucket and the level of the liquid decreases.
- There should be a tapering funnel at the top with a control valve so that we can easily take out the acetic acid solution from the top instead of a dropper what we did, this can lead to much smaller errors.
- The syringe should be placed above the water level in the column, because the flow can be in reverse direction if there's empty space in the syringe and when no acetic acid solution is being pumped in the column.

CONCLUSION

- The inverted-phase experiment offers a valuable extension to the classical "Mass Transfer from a Single Drop" setup by simulating a practical scenario where a lighter organic phase is dispersed into a heavier aqueous phase. The results confirm that drop dynamics—such as increased terminal velocity, flow rates, drop size, etc. —affect the mass transfer coefficient.
- Despite these limitations, the experiment successfully demonstrates mass transfer behavior in upward droplet systems and provides a strong foundation for further design optimization in extraction processes.
- Future Work: To improve , adding a top-mounted collection funnel with a controlled valve, optimizing syringe positioning, and a stopper above needle. In future, we could Integrate high-speed video capture for accurate drop tracking, residence time, and shape analysis and build a CFD simulation for this system.

THANK YOU