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Evaluating the effect of added electrolyte and surfactant on the performance of Bubble Column Bioreactor

Ameya Harmalkar[1]‡, Abhishek Dhand[1]‡, Manu Vashishtha[2,3,*]
[1] *Department of Chemical Engineering, Institute of Chemical Technology, Mumbai, India.*
[2] *Department of Chemical Engineering, Indian Institute of Technology – Bombay, India*
[3] *Department of Humanities, Dr. B. R. Ambedkar Institute of Technology, Andaman & Nicobar Island, India*
**- Corresponding Author: manu.vash@gmail.com*
‡- Equal Contribution

Abstract:*In the present study, the reactive absorption of CO_2 in aqueous NaOH and Na_2CO_3-$NaHCO_3$buffer solution has been utilized to investigate the mass transfer coefficient (k_La). Bubble column reactors can serve as potential bioreactors for the commercial production of proteins, antibiotics and enzymes with tunable operational parameters. CO_2 induced bubble reactors have a prospect of application in CO_2 sequestration for algal/microbial conversions, where absorption of CO_2 in systemic media is essential.In order to study the hydrodynamics for the design of biological systems, the rates of absorption of pure carbon dioxide into the buffer solution have been measured using a Bubble Column reactor. The effect of the mass transfer coefficient k_La was investigated with the flow rate of the gas; ionic strength, nature of ions, viscosity and surface tension of the buffer solution. The effect of added surfactants on the performance of bubble column reactor was evaluated. Attempts have also been made to determine the operating regime of the reaction. The study aims to determine the mass transfer coefficient of the reactive absorption in a bubble column, and investigates the use of crucial parameters on the rate of absorption of the reaction for extrapolation to the design of bubble columns for biological systems. Recommendations have also been made for correlations and suggestions for further work have been incorporated.*

Keywords**:** *Bubble column, Mass transfer coefficient, reactive absorption, CO_2 sequestration, surfactant.*

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I. Introduction

Absorption of CO_2 into aqueous solutions of alkali hydroxides and carbonates have been studied by various investigators not only for theoretical interest but also because of the industrial importance of the reaction.[1] Gas Absorption has various applications in the chemical industry such as the removal of CO_2 and H_2S from natural gas or synthesis gas by absorption in solutions of amines or alkaline salts. Another example is the washing of ammonia from a mixture of ammonia and air by means of liquid water. The solute is subsequently recovered from the liquid by distillation, and the absorbing liquid can be either discarded or reused.

Sometimes the solute is removed from a liquid by by bringing the liquid into contact with an inertgas; such an operation, the reverse of gas absorption, is desorption or gas stripping. Aerated drinks have CO_2 dissolved in water at high pressure, leading to sparkly taste on consumption. Additionally, gas absorption as a process can be used.

In a bubble column, gas is dispersed in liquid phase at the bottom of a column with the help of a distributor. Bubble column reactors are preferred for slow or very slow gas liquid reactions. Because, in such cases, the overall rate of reaction depends primarily on the liquid hold-up which is very high in a bubble column. Industrial examples of bubble column reactors are Liquid phase air oxidation of acetaldehyde in presence of $KMnO_4$, air oxidation of p-nitrotoluenesulphonic acid, carbonylation ofmethanol to acetic acid, oxychlorination of ethylene for the production of vinyl chloride, production of proteins in a fermenter from methanoletc. [1,2] The major disadvantage of bubble column is the complete backmixing in liquid phase which decreases the concentration of liquid and hence slows down the rate of reaction. To overcome this, baffles, packings or trays are used.

Investigations of bubble columns from the viewpoints of both environmental problems and industrial use can be found in the literature. [2] They were favored because of their simple construction, higher heat and mass transfer coefficients, higher removal efficiency, and effective control of the liquid residence time. In addition, bubble columns may be operated in either batch mode or continuous mode, depending on the requirements, as in processes such as liquid-phase methanol synthesis or continuous-mode Fisher-Tropsch synthesis with a liquid superficial velocity that is lower than the gas superficial velocity by at least an order of magnitude or more. In this manner, the gas flow controls the fluid dynamics of the individual phases of these systems. This in turn controls liquid mixing and inter-phase mass transfer, which subsequently influence conversion and selectivity.[3,4]

The absorption of gas into a solvent will take place as soon as the gas comes in contact with the liquid solvent. The gas reagent moves first from the bulk of the gas to the interface, diffuses through the film resistance of the interface and then, enters the bulk. In reactive absorption however, the reaction of the gas reagent with the solvent can be dependent on a variety of factors, majorly on whether the reaction is kinetically controlled or mass transfer controlled.

The absorption of CO_2 into strongly alkaline solutions is often treated as absorption accompanied by a reversible second order reaction.[1-3]The absorption proceeds through the gas film and the liquid film encompassing various film and bulk media resistances. To determine the regime of the reaction, the CO_2 flow rates have been varied to understand the effect of the gas phase reagent on the absorption. If there is a considerable change in the rate of absorption, then it can be concluded that the reaction is in the second regime .i.e. Mass Transfer controlled regime, else if it remains independent of the changes then we can conclude that it is the fourth regime .i.e. Instantaneous reaction regime.

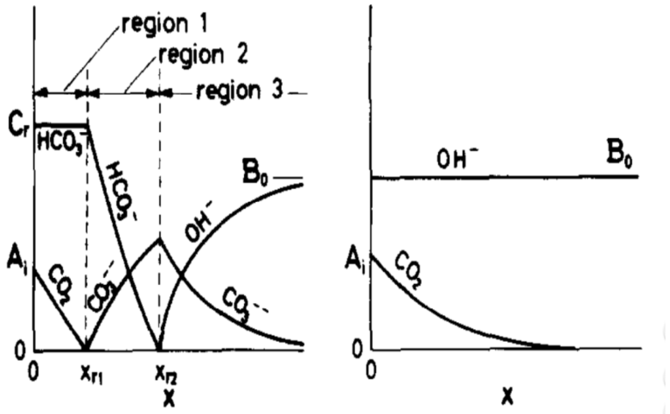

Figure 1: Concentration profile of CO_2 absorption in buffer under a) instantaneous reaction regime b)pseudo-first order regime.

The Henry's constant is a defining factor for solubility, however, its value will be different in the buffer and must be accounted for. [5] The Danckwerts[4] theory has been utilized to evaluate the Henry's constant in the buffer which has been utilized for the determination of gas phase concentration of CO_2 in the reaction.

II. Materials And Methods

The column, made of glass, was fixed on the support stand with Teflon tubing. The air compressor and CO_2 flow from the cylinder were switched on. The gas flow was adjusted such that 10% $CO_2$and remaining air was sparged inside the column. Gas flow rates are measured using calibrated rotameters. All the glassware, Burette (50 ± 0.1 mL),pipette (10 ± 0.2 mL), conical flask (approx. 25mL), procuredfrom Merck, were washed thoroughly, rinsed and then oven dried before using. The buffer solution was then added to the column ensuring no weeping takes place. The timer is started simultaneously. Sufficient time was provided for gas liquid contact to allow steady state to reach. The initial height of buffer in the column Hi (in cm) and Dispersion height H (in cm) is noted. The sample was collected from the column for analysis after the reaction time, t (in min). The aforementioned procedure was repeated for different gas flow rates and different concentrations of the buffer solution. Volumetric titrations were performed to estimate the moles of CO_2 reacted.

Table 1: Equipment least count and calibration data

Equipment	Least Count	Description
Measuring Cylinder	10 mL	Used to measure the volume of water in the column
Burette	0.1 mL	Used to measure the titrant volume
Pipette	0.1 mL	Used to measure the analyte volume
Column Scale	0.1 cm	Used to measure the height of the interface

Chemicals such as NaOH, HCl, $BaCl_2$ and other chemicals used in the experiment, were procured from the SD Fine chemicals. Suitable amount of sample (10ml) was mixed with excess 0.2 N $BaCl_2$ and 25ml 0.5N

NaOH (standardized solution). $BaCl_2$ is added to precipitate the intermediate $BaCO_3$ formed. The resultant analyte was titrated against 0.5N HCl (standardized solution) using phenolphthalein indicator (end point- pink to colorless) to obtain titration reading (T2) which corresponds to concentration of bicarbonate. An initial sample of the buffer solution is analyzed for the total alkalinity.

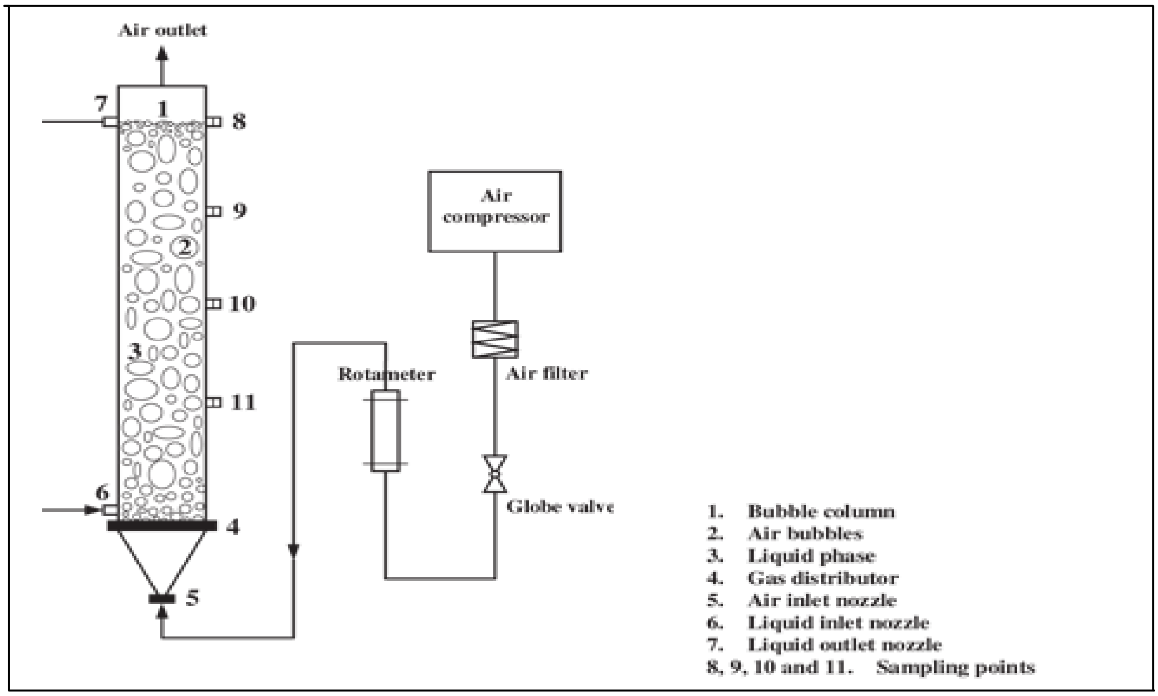

Figure 2: Schematic Representation of the Experimental Set-up

III. Results And Discussions

The experiment investigates the chemical absorption of CO_2 in a buffer solution in a Bubble column, and also attempts to analyze the effect of gas flow rates on the reactive absorption. For the determination of regime of reaction, concentration of the buffer has been varied by subsequent dilution. Regime determination is crucial as that allows us to approximate a Reaction regime for determination of the mass transfer co-efficient. It is well established that the liquid in a reasonably well agitated contactor is perfectly mixed. [6]

In a recent investigation by authors *Hanhart, Westerterp and Kramers*[5],it has been reported that the gas phase in an agitated dispersion in bubble columns can be considered to be perfectly mixed if there is extensive back-mixing. The present study was composed of two parts:

a) Identification of the Regime.
b) Investigation of the dependence of gas flow rates .i.e. superficial gas velocity on the Rate of Absorbance, and subsequently, on the mass transfer coefficient.

3.1. Identification of the reaction regime in a Bubble column

The reaction in bubble column is dependent on the amount of agitation in the media as reported by *Pandit and Joshi* which evidently rules out the Regimes 1 and 3 from the picture. The regime equations for the second and fourth regime are as follows:

$$R_A a = k_l \underline{a}\{[A^*] - [A_o]\} \qquad \textbf{...(1)}$$

$$R_A a = k_l \underline{a}[A^*] \times \left[1 + \frac{[B_o]}{Z\,[A^*]}\sqrt{\frac{D_B}{D_a}}\right] \qquad \textbf{...(2)}$$

The regime equations for Mass transfer controlled and Instantaneous Reaction regimes portray the dependence on reactant A and B. In the present study, for determination of the reaction regime, we have varied the concentration of reagent B .i.e. bulk liquid, by dilution of the buffer. The reactions are:

$Na_2CO_3 + H_2O + CO_2 \rightarrow 2NaHCO_3$
$NaHCO_3 + NaOH \rightarrow Na_2CO_3 + H_2O$

$Na_2CO_3 + BaCl_2 \rightarrow NaCl + BaCO_3$

The absorption of CO_2 into the Na_2CO_3-$NaHCO_3$ buffer leads to the conversion of Na_2CO_3 into $NaHCO_3$. The net amount of $NaHCO_3$ can be obtained by addition of NaOH and $BaCl_2$ followed by titration with HCl. Initial concentration of $NaHCO_3$ can be obtained by titration of the sample buffer before reaction, thus the amount of Na_2CO_3 consumed can beobtained, which is an indirect measure of amount of CO_2 consumed.

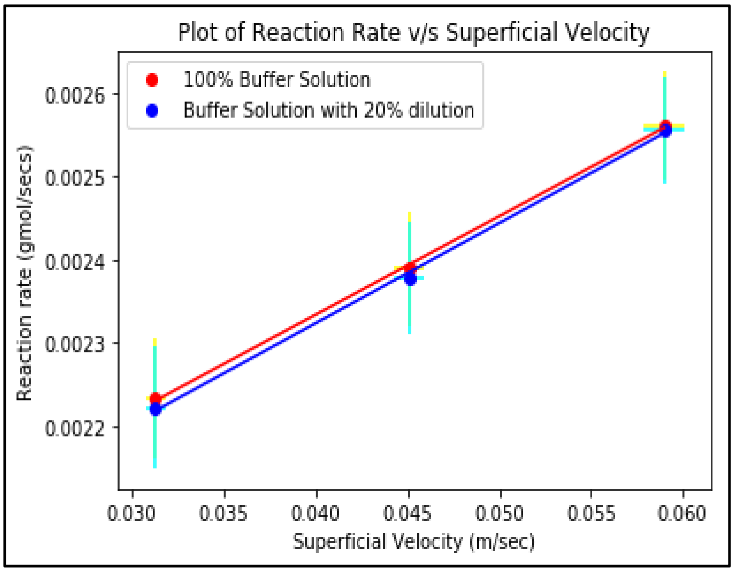

Figure 3 : Dependence of Reaction rate on Buffer Concentration

It can be delineated from the graph shown in ***Figure 3*** that the reaction rate is almost ***independent of the dilution in buffer*** for variation in superficial velocities of the gas and hence, we can assert that the reaction is in ***Regime 2***. The regressed fit and error bars for both the cases with and without dilutions are straight line almost coinciding with each other with the error ranges into consideration.

3.2. Dependence of mass transfer coefficient on superficial velocity

The mass transfer coefficient of the reactive absorption exhibits strong influence on the concentration of A .i.e. the Gas phase reagent CO_2. As the flow rate of CO_2 increases, the superficial velocity increases, the bubble size of the dispersion decreases resulting in higher interfacial area. The mass transfer coefficient follows a directly proportional relationship with the superficial gas velocity, and it slightly increases with increasing superficial velocity.

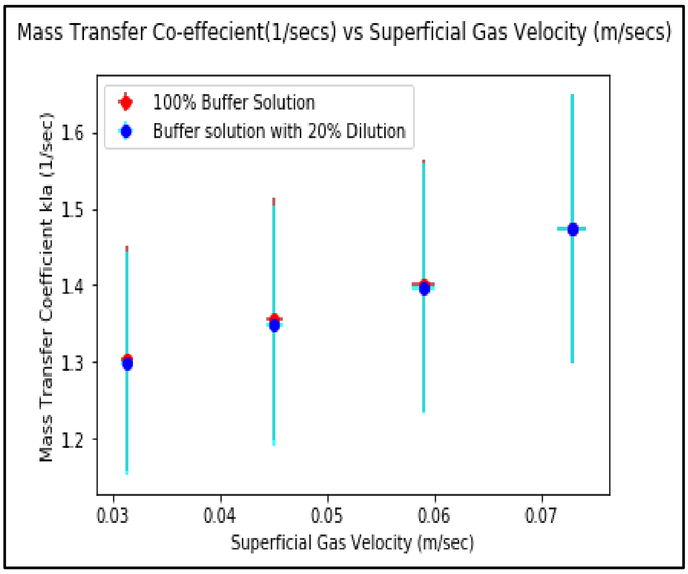

Figure 4 : Variation of Mass transfer Coefficient with Superficial velocity

With the conclusion that the reaction occurs in regime 2, we carry out further analysis of the reading obtained. By studying the variation of mass transfer coefficient with superficial gas velocity in Figure 4 for regime 2, we see that as the superficial velocity increases, the mass transfer coefficient increases. Since the performance of a bubble column depends on k_La, by increasing the gas superficial velocity, we can achieve good mass transfer rates. This is because, as the gas superficial velocity increases, the boundary layer film at the interface decreases, leading to higher k_L. Also, as the gas velocity increases, the bubble breaking increases due to increased turbulence, leading to formation of bubbles of smaller diameter, thereby increasing the interfacial area per unit volume, a.

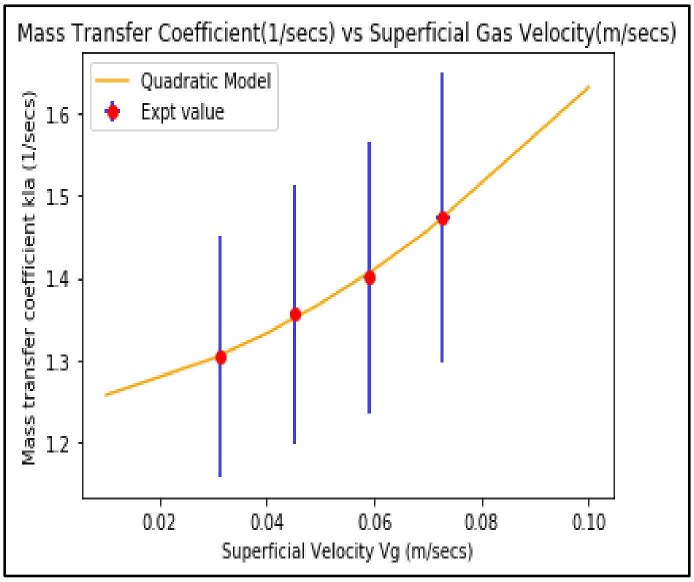

Figure 5 : Best fit for variation of mass transfer Coefficient

It is also evident from the figure that major dependence of the reaction rate is with theamount of CO_2 into the system rather than the buffer concentration. The uncertainty analysis for the Quadratic model was fitted and the values of the coefficients are as follows:

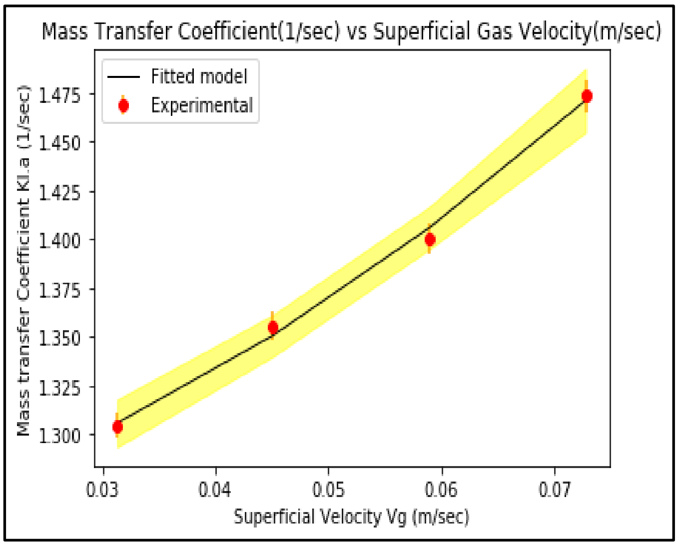

Figure 6: Quadratic fitting of the curve with uncertainty analysis

The quadratic fitting with the uncertainty analysis have been portrayed in the Figure 6. The shaded region denotes the region of uncertainty in the plot and the red markers indicate the experimental values with error bars.It is evident from ***Figure 6*** that the uncertainty is the curve is acceptable and hence the quadratic best fit can be assumed to be acceptable.

3.3. Effect of addedelectrolyte on rate of absorption

The inorganic substances, often salts of mineral acids or other electrolytes and ionic solutions, are repelled from the interface where they adsorb negatively and cause very small increase of the surface tension. Despite the negative surfactants like salts produce only a very small increase in surface tension, their influence of on the behaviour of bubbly mixtures can be enormous.

Bubble coalescence times are influenced by both electrolyte concentration and the bubble approach speed as reported by Del Castillo et al. [14] Addition of electrolyte removes the stable region and shifts the transition between transient stability and instant coalescence to higher speeds which was in agreement with the model developed by Yaminsky et al. [16] Most studies have revealed that coalescence occurs easily in pure water and that with increasing concentrations of added electrolytes, there is a transition to coalescence inhibition. This transition concentration is sharp and occurs over a narrow range which is characteristic of a particular electrolyte, usually at concentrations of the order of 0.1 M. Marrucci et al. developed an expression for the amount of salt required to immobilize the gas-liquid interface in bubble coalescence. This amount of salt depends on the magnitude of the change in surface tension with concentration at the interface or surface activity. [15]

The effect of addition of electrolyte (NaCl) on the rate of absorption and thus on the mass transferco-efficient was analyzed. The presence of electrolyte is supposed to suppress the chances ofbubble coalescence. This is because similarly charged particles will repel bubbles and force themto stay apart. This would lead to presence of smaller bubbles resulting in more gas-liquid contactand hence higher mass transfer co-efficient. However, experimentally, it was observed that theeffect was not very significant. The possible reason may be because the effect of electrolyte ismuch less prominent than the effect of liquid film drainage between 2 bubbles for coalescence.The Rate of reaction remains almost constant for increasing electrolyte concentration. The addition of electrolytes, especially kosmotrophic salts like NaCl, is paramount in biological contactors where pH of the solution influences the rates of reaction. In CO_2 induced systems, interfacial area is often important as it lies in Regime 2 and hence can make great contributions to productivity of the equipment and economics as a whole.

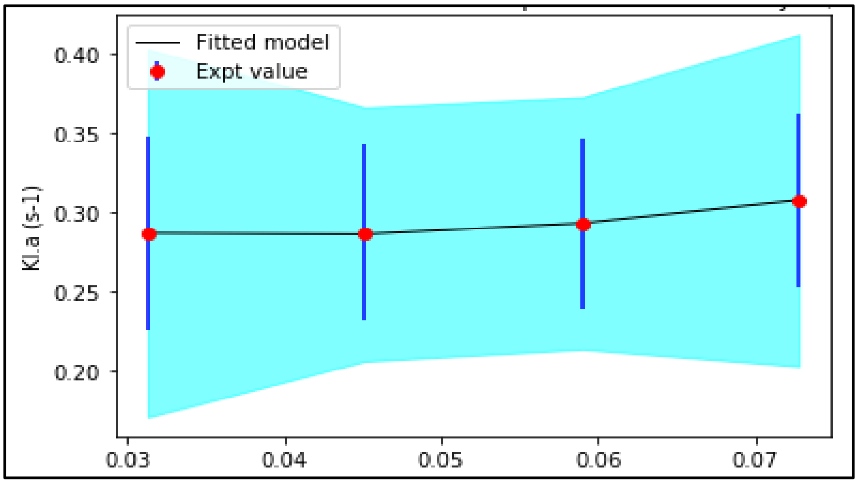

Figure 7: Variation of Mass transfer coefficient with Concentration of electrolyte

3.4. Effect of Surfactants

The presence of a surfactant affects the gas–liquid mixture in many different ways, on many different temporal and spatial scales: bubble formation, bubble rise, bubble interactions, axial and radial profiles, mixing and dispersion, overall voidage and flow regimes, and consequently, mass transfer.

The gas holdup generally increases when a surfactant is present. When increasing the surfactant content, the voidage rises to a saturation value, where it levels off, being insensitive to further surfactant addition. The reason for the voidage increase is seen threefold. First, smaller bubbles are formed in surfactant solutions. Second, the bubble coalescence is suppressed and the small bubbles remain small. Third, the boundary condition on the bubble surface is changed from slip to no-slip and the bubbles experience a larger drag. All these reasons lead to a lower bubble speed u, which in turn results in a higher voidage. The reason for the voidage saturation is seen in the fact that the capacity of the surfactant to reduce the bubble size and to rigidify the bubble surface in a given bubbly mixture is exhausted, so adding more of it exerts no effect.Ruzicka et al. (2008) found experimentally, that the homogeneous regime is stabilized by low surfactant concentration and destabilized by high concentration. [29]

Raymond et al. and Koide et al. concluded that the decrease in the liquid-phase mass transfer coefficient k_L on the addition of surfactants is primarily due to changes in hydrodynamic characteristics of the system. [31,32] Surfactants adsorbed at the gas-liquid interface retard surface flow by the surface tension gradient at the interface and hence decrease k_L value of a single bubble. Along this line of thought, Koide et al. proposed a model to estimate k_L of a single bubble in aqueous solutions of surfactants. Bubble columns have received attention as bioreactors. Liquids in bioreactors contain surfactants, antifoam agents or both. Yagi et al. studied experimentally the effects of surfactants, antifoam agents and sterilized cells on the gas holdup s and the volumetric liquid-phase mass transfer coefficient k_La in a bubble column, and showed that both ε and k_La were decreased by the addition of surfactants or antifoam agents to water. [33] On the other hand, Kelkar et al. showed that voidage (ε) was increased by the addition of normal alcohols to water and that the degree of increase in ε was higher in aqueous solutions of alcohols with longer carbon chain length. [30]

Roustan et al. (2005) evaluated the effect of surfactants in a bubble column reactor and reported the various parameters on which the performance depends: [9]

a)***Bubble Diameter (D_B)***: At low gas flow rates (Q_G< 1mL/s), the bubble diameters obtained with cationic and anionic surfactant solutions are lower than those obtained with tap water. As proposed by *Loubière and Hébrard* (2004), these results are due to the differences observed in terms of dynamic surface tensions and to their consequences on the balance between the surface tension and the buoyancy forces during the bubble growth and detachment. [10] At high gas flow rates, the differences in terms of bubble diameters are directly linked to static surface tension values. The bubble diameter is no more controlled by the force balance at detachment, but rather by the power dissipated in the liquid, conditioning the bubble break up and coalescence phenomena. This clearly proves that a modification of surfactant concentration (i.e., of surface tension value and of surface coverage ratio at equilibrium) affects the generated bubble diameters.

b) ***Bubble formation frequency***: The bubble formation frequencies obtained with surfactants are on average larger than those with water, except for low gas flow rates where the smallest frequencies are observed for cationic surfactants. In the case of water and anionic surfactant solutions, the bubble formation frequency reaches a constant value above critical gas flow rates whereas it increases continuously for cationic surfactants.

c) ***Terminal rising velocity of bubble***: The results show that the terminal rising bubble velocity is affected by some modifications of surfactant concentrations.

d) ***Interfacial Area***: The interfacial area increases roughly linearly with the gas flow rate irrespective of the liquid phase. The interfacial areas related to surfactant solutions are significantly larger than those of water.

e) ***Volumetric mass transfer coefficient***: The variation of the volumetric mass transfer coefficient (k_La) with the gas flow rate for the different liquid phases indicates that the volumetric mass transfer coefficient increases with the gas flow rate irrespective of the liquid phase. The values of k_La vary between 0.00035 and 0.003 s^{-1} for gas flow rates varying between 0.3 and 3.5 mL/s. The volumetric mass transfer coefficients of both surfactant solutions are significantly smaller than those of water. The volumetric mass transfer coefficient increases with the gas flow rates whatever the liquid phases and the k_La values for both surfactants are significantly smaller than those of water. The liquid-side mass transfer coefficient remains roughly constant for a given liquid phase irrespective of bubble diameter. The physico-chemical properties such as liquid surface tension and surface coverage ratio (s_e) prove to be the important parameters to consider for predicting the variation of the k_La values.

IV. Applications

Bubble column reactors are extensively used as bioreactors in which micro-organisms are utilized in order to produce industrially valuable products such as enzymes, proteins, antibiotics, etc. [7] Table 2 below summarizes the various applications of bubble column reactors as bioreactors.

Table 2: Biochemical applications of bubble column reactor

Sr. No.	Bioproduct	Biocatalyst	References
1	Thienamycin	Streptomyces cattleya	[17]
2	Glucoamylase	Aurebasidium pullulans	[18]
3	Acetic acid	Acetobactoraceti	[19]
4	Monoclonal antibody	Hybridoma cells	[20]
5	Plant secondary metabolites	Hyoscyamusmuticus	[21]
6	Taxol	Taxus cuspidate	[22]
7	Organic acids	Eubacteriumlimosum	[23]
8	Ethanol fermentation	Saccharomyces cerevisiae	[24]

Large scale industrial application of bubble column bioreactors utilize 'tower fermenters' for the production of alcoholic liquids, in particular beer. The commercial production of beer in tower fermenters has been carried out in continuous operation over a period of many years. [8] These bioreactors are also applied in vinegar production. The aeration in a bubble-column reactor provides the required oxygen for an aerobic microorganism and also mixing. [28] Proper aeration provides suitable gas holdup, a higher residence time of the gas in the liquid, and a high gas-liquid interaction area available for mass transfer. Newer applications of such bio- reactors without mechanical agitation have been worked out for single cell protein (SCP) production. [25-27] Hosseini et al. (2003) used a bubble column reactor for production of SCP from cheese whey. [11] They also studied the effect of the gas flow rate and L/D ratio on biomass production and COD reduction and reported that high shear forces, addition of excess antifoam to the medium and increased gas flow rate imposed an inverse effect on the mass-transfer coefficient whereas by increasing L/D, the gas holdup decreases, and so the mass-transfer coefficient also decreases. Since the planned commercial units are larger than the usual bioreactors, by the application of stirred tank bioreactors, one would run into several difficulties due to the necessity for heat removal in external cooling loops with sufficiently high pumping rates, and to the intensive aeration as well as agitation required. Their high energy requirement would prevent the economical operation of a large commercial plant. To avoid these difficulties, especially the external cooling of the fermentation medium which is necessary to remove the great amount of heat produced by mechanical stirring, new pneumatic bioreactors (bubble column reactors with and without recycling) are developed. Different modifications of bubble column bioreactors, e.g. multistage tower reactors with a mechanical stirrer have also been fabricated. Chen at al. (2015) modeled fermentation of synthetic gas to renewable liquid fuels such as ethanol and 2,3-butanediol using metabolic and transport models of fermenting bacteria Clostridium ljungdahlii in a bubble column reactor. [12] On the other hand, Wu and Merchuk (2002) simulated biomass production of marine red microalga Porphyridium sp. in a bubble-column photobioreactor and studied the effects of shear stress on growth rate. [13]

V. Conclusions

The absorption of CO_2 in the Na_2CO_3-$NaHCO_3$ buffer is investigated to obtain the regime of reaction and analysis of the variation in mass transfer coefficient and rate of absorption with variation of CO_2 have been studied. The operating regime of the reaction is concluded to be ***Regime 2*** .i.e., Mass transfer controlled Regime as the reaction is independent of the dilution in the liquid phase (buffer). From the *Figure 3* and *Figure 4* it can be inferred that the mass-transfer coefficient and the Rate of Absorption, both have a directly proportional relationship with CO_2. Overall rate is mass transfer controlled, affected by the gas flow rate and not by the liquid phase concentration. Liquid phase mass transfer coefficients were calculated. The obtained values of the liquid side mass transfer coefficient for reactive absorption of CO_2 gas in the $Na_2CO_3/NaHCO_3$ buffer solution in the bubble column increased with increasing gas flow rates.

The absorption of CO_2 in buffer solutions is paramount in biological contactor which involve salting out and salting in phenomenon with kosmotropes. A plethora of biological microbes function in a CO_2 induced media, and the reaction rates of such microbes can be tuned with the absorption rates of CO_2 in the buffer. The use of bubble column for biological systems can be manipulated by operation parameters resulting in an effect on the shear stress and interfacial area.

Further studies can involve the addition of surfactant to the solution to study whether absorption is affected as a whole as surfactants are supposed to influence the bubble shape and size. Interfacial area of the bubbles is a crucial parameter for the determination of the mass transfer along with the mass transfer coefficient. Sparger variations can be carried out and the effect of sparger size variations on the overall functioning and regime of the column can be studied.

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