

Comprehensive analysis of Studies based on Precipitation of Silica by CO₂ Dissolution in Sodium Silicate

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ABSTRACT

The work described aims to extract silica from rice husk ash (RHA) as a cost-effective and environmentally friendly alternative to conventional methods. The high silica content in RHA makes it a valuable source for silica extraction. Additionally, by utilizing RHA instead of quartz, which is conventionally used, the environmental impact and energy requirements can be reduced. The process of extracting silica from RHA involves digesting it with sodium hydroxide (NaOH) and then dissolving carbon dioxide (CO₂) in the resulting sodium silicate solution. The dissolution of CO₂ in the solution is crucial because CO₂ is a greenhouse gas and its capture and storage are important for mitigating climate change.

The experimental work focused on studying the dissolution of CO₂ in sodium silicate solution at different temperatures. A 1-meter high absorption column with a square cross-section of 0.20 meters was constructed for this purpose. The CO₂ was introduced into the absorption column through a sparger, leading to the formation of bubbles. As the bubbles ascended the column, their diameter and acceleration gradually decreased. Eventually, the bubbles reached the surface of the column and escaped. The goal was to determine the ideal temperature for complete dissolution of CO₂ in the sodium silicate solution. High-speed video analysis using a Photron Fast Cam SA4 camera was performed to analyze the bubble behavior at different temperatures. The experimental data showed that at temperatures of 50°C and 60°C, complete dissolution of CO₂ was not achieved. However, at temperatures higher than 60°C, the bubble size reduced to zero by the time it reached the top of the column, indicating complete dissolution of CO₂ in the liquid. Based on the experiments, it was concluded that 70°C is the ideal temperature for achieving complete dissolution of CO₂ in the sodium silicate solution.

This research provides insights into the dissolution of CO₂ in sodium silicate solution and identifies the optimal temperature for achieving complete dissolution. By utilizing rice husk ash as a raw material for sodium silicate production and capturing CO₂ in the process, the work contributes to the valorization of agricultural residues and the reduction of greenhouse gas emissions.

Keywords: CO₂ Dissolution, Precipitation, Silica, Rice husk ash

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I. INTRODUCTION

The work described aims to understand the dissolution of carbon dioxide gas in sodium silicate solution and determine the suitable temperature for its dissolution. Carbon dioxide is a greenhouse gas that contributes significantly to climate change, and it is important to capture and store it to mitigate its effects on the climate and human health. Rice husk, an agricultural residue that is abundantly available, contains a high amount of ash, with silica being the major constituent, accounting for approximately 90% [1-3]. Extracting silica from rice husk ash not only provides a value-added product but also addresses the issue of disposing of large amounts of ash. In this work, the focus is on studying the dissolution of carbon dioxide in sodium silicate solution. Sodium silicate can be derived from quartz (silica rock) as well as rice husk ash. By dissolving carbon

dioxide in sodium silicate solution, silica can be precipitated as a solid, and the remaining sodium carbonate can be treated with calcium hydroxide to regenerate sodium hydroxide [5].

Understanding the dissolution of carbon dioxide in sodium silicate solution is crucial for capturing and storing carbon dioxide effectively. The researchers aim to determine the appropriate temperature at which carbon dioxide can be dissolved most efficiently. By studying the dissolution process and identifying the optimal temperature, this work contributes to the development of methods for capturing and storing carbon dioxide. It utilizes rice husk ash, an abundant agricultural residue, and provides insights into the utilization of silica and the reduction of greenhouse gas emissions.

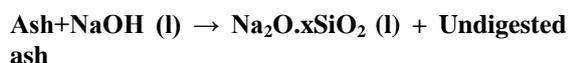
II. MATERIALS

In the process of producing sodium silicate solution from rice husk ash (RHA), the first step is digestion. This step involves digesting the RHA with caustic (sodium hydroxide) under specific conditions. The purpose of digestion is to extract silica from the ash and form a sodium silicate solution.

During digestion, the RHA is mixed with caustic, and the mixture is subjected to specific conditions, such as temperature and reaction time, to facilitate the extraction of silica. The caustic reacts with the silica present in the ash, resulting in the formation of sodium silicate in the solution. The digestion process allows for the dissolution of the silica and its conversion into a soluble form.

Once the digestion process is complete, the solution is filtered to remove any residual undigested ash that may be present. Filtration helps ensure that the clear filtrate is obtained, which contains the desired sodium silicate solution without any solid impurities.

The clear filtrate, which consists of the sodium silicate solution obtained through digestion, is then ready for the next step in the process, which is precipitation..



Precipitation

In the precipitation step of producing sodium silicate solution from rice husk ash (RHA), the focus is on precipitating silica from the sodium silicate solution obtained through the digestion process. The following steps are involved in the precipitation process:

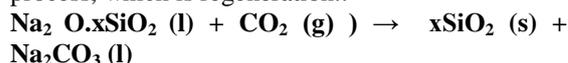
Carbon Dioxide Addition: The sodium silicate solution is subjected to the addition of carbon dioxide gas at a specific flow rate. This carbon dioxide gas is passed through the solution under design conditions. Continuous stirring is employed during this operation. The purpose of adding carbon dioxide is to induce the precipitation of silica from the solution.

Precipitation: As carbon dioxide is infused into the sodium silicate solution, a reaction occurs between the carbon dioxide and the sodium silicate. This reaction leads to the precipitation of silica as a solid. The carbon dioxide interacts with the sodium silicate to form solid silica particles, which gradually separate from the solution.

Filtration and Washing: After precipitation, the resulting precipitated silica is separated from the solution by filtration. Filtration helps to remove the solid silica particles from the liquid. The precipitated silica is then washed with

water to remove any soluble salts or impurities that may be present.

Drying: Once the precipitated silica is washed, it is dried to remove any remaining moisture. Drying can be achieved through various methods such as air drying or using drying ovens. The dried silica is obtained as a solid product. The filtrate, which contains sodium carbonate resulting from the reaction between carbon dioxide and sodium silicate, is taken for the next step in the process, which is regeneration..



Regeneration

In the regeneration step of producing sodium silicate solution from rice husk ash (RHA), the focus is on regenerating the sodium hydroxide and utilizing the byproduct sodium carbonate. The following steps are involved in the regeneration process:

Reaction with Calcium Compound: The sodium carbonate obtained from the precipitation step reacts with a calcium compound (such as calcium hydroxide) to form calcium carbonate and sodium hydroxide. The calcium compound can be obtained from various sources, including commercially available calcium hydroxide or by utilizing the calcium hydroxide obtained from the previous regeneration cycle. The reaction is typically carried out in aqueous solution.

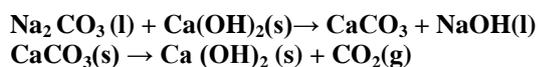
Filtration: The resulting solution after the reaction is filtered to separate the solid calcium carbonate from the aqueous sodium hydroxide. Filtration helps to remove the insoluble calcium carbonate, leaving behind the sodium hydroxide solution.

Washing and Drying: The separated calcium carbonate is washed with water to remove any impurities and soluble salts. After washing, the calcium carbonate is dried to remove any remaining moisture. Drying can be done using methods like air drying or using drying ovens.

Reuse or Value Addition: The dried calcium carbonate can be utilized in two ways. It can be calcined (heated) to obtain calcium oxide, which can be reused in the regeneration process to react with sodium carbonate again, completing the cycle. Alternatively, the calcium carbonate can be sold as a separate product, providing an additional value stream.

The regenerated sodium hydroxide solution obtained from the filtration step is then ready to be used in the digestion process again, starting the sodium silicate production cycle anew. This regeneration step ensures the efficient utilization of resources and provides options for

value addition with the byproduct calcium carbonate..



Precipitated Sodium silicate has industrial application in food, paint, cosmetic industry etc.

Movement of Carbon Dioxide in Sodium Silicate Solution

In the study of carbon dioxide bubble movement in sodium silicate solution, a specially designed absorption column was constructed. The column had a height of 1 meter and a square cross-section measuring 0.20 meters. The column was made of toughened glass, which provided both visibility and the ability to withstand high temperatures up to 200°C.

To maintain the desired temperature, two flat heating coils and two thermocouples were fixed at two sides of the absorption column. This allowed for precise temperature control during the experiment.

To introduce carbon dioxide gas into the system, a sparger with a diameter of 0.1 mm was used. The sparger was fixed at the bottom of the absorption column and served as the point of gas entry. The carbon dioxide gas passed through the sparger and entered the sodium silicate solution.

The study was conducted at various temperatures, likely to observe the effects of temperature on carbon dioxide dissolution. By analyzing the movement of carbon dioxide bubbles in the absorption column, researchers could gain insights into the behavior of the gas within the sodium silicate solution.

To capture the movement of the carbon dioxide bubbles, a high-speed camera, specifically a PHOTRON Fast Cam SA4 video camera, was used. The camera allowed for slow-motion analysis, enabling detailed observation and analysis of the bubble dynamics within the absorption column.

Overall, this experimental setup and analysis aimed to provide a better understanding of how carbon dioxide bubbles behave and move within the sodium silicate solution, particularly in relation to temperature variations..

Steps

The experiment involving the movement of carbon dioxide in the sodium silicate solution can be summarized into the following steps:

- **Preparation:** Ten liters of sodium silicate solution are filled into the absorption column. The heating coil is switched on to reach the desired temperature. The camera is positioned in front of the absorption column to capture the bubble movement.
- **Temperature Setpoint:** Once the temperature reaches 50°C, the carbon dioxide gas is introduced into the absorption column through the sparger. At this point, the camera is switched on to start recording.
- **Bubble Formation and Movement:** As the carbon dioxide enters the absorption column, bubbles of varying diameters are formed. These bubbles start moving towards the top of the column. The time taken for the bubbles to reach the top is approximately 5 to 6 seconds. The movement of the bubbles is recorded using the camera.
- **Precipitated Silica Removal:** After the experiment, the precipitated silica, which has settled in the solution, is removed from the absorption column. This step ensures the separation of the solid silica particles from the sodium silicate solution.
- **Fresh Sodium Silicate Solution:** For each trial, a fresh batch of sodium silicate solution is used. This ensures consistent conditions for the experiments conducted at different temperatures, such as 50°C and 70°C.
- **Repeat Procedure:** The entire procedure is repeated for different temperatures of interest, such as 50°C and 70°C. This allows for comparative analysis of the bubble movement and dissolution characteristics at different temperatures.

By following these steps and conducting multiple trials at different temperatures, the researchers can gather data on the behavior of carbon dioxide bubbles in the sodium silicate solution and determine the most suitable temperature for achieving complete dissolution of carbon dioxide.

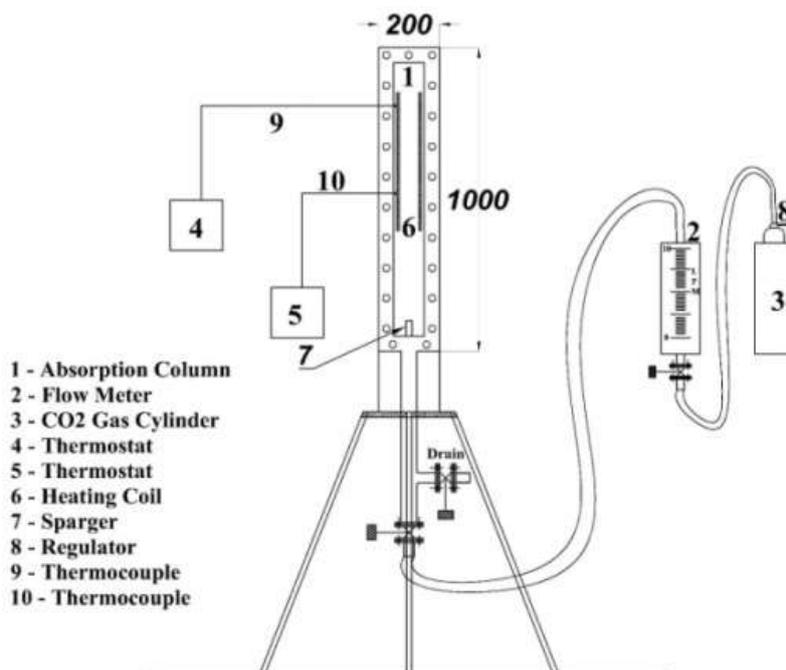


Fig. 2: Line Diagram of Absorption Column.

III. RESULTS AND DISCUSSION

The experimental work aimed to study the diameter, positioning, and acceleration of carbon dioxide bubbles in the sodium silicate solution. The carbon dioxide gas was introduced into the absorption column through a sparger, initiating bubble formation. The following observations were made:

- Initial Parameters: At the start of the experiment, a bubble with a diameter of 5 mm was selected at 0.04 seconds. The bubble had an initial speed of 450 mm/sec and was positioned at 18 mm within the column.
- Bubble Behavior: As the bubble moved up in the column, its diameter and acceleration gradually decreased. At the liquid's surface, the bubble reached a diameter of 0.125 mm and an acceleration of 1.9083 mm/sec.
- Time and Dissolution: The diameter and acceleration of the bubble decreased over time until the bubble reached the surface of the column and escaped. From these observations, it was evident that complete dissolution of carbon dioxide was not achieved at a temperature of 50°C with an initially selected bubble diameter of 5 mm.

- Repeat Experiment at 70°C: In order to investigate the effects of a higher temperature, the experiment was repeated at 70°C. The results are presented in Table 2 and illustrated in Figures 6, 7, and 8.
- Bubble Analysis at 70°C: For the temperature of 70°C, a bubble with an initial diameter of 3.25 mm was selected. Frame-wise analysis was conducted, starting from frame number 18 and continuing for an interval of 25 frames. The analysis showed that the bubble's diameter and velocity decreased over time. It was observed that the bubble size reached zero around 3 seconds at a height of 50-60 cm, indicating complete dissolution of carbon dioxide in the liquid.

Based on these findings, it can be concluded that at a temperature of 70°C, complete dissolution of carbon dioxide in the sodium silicate solution was achieved with an initially selected bubble diameter of 3.25 mm. The experiment provided insights into the dynamics of bubble dissolution, including the relationship between bubble diameter, velocity, and time, as well as the position of the bubble within the column.

Table 1: Analysis for Change in Diameter, Position and Velocity of the Bubble at 50°C.

Exp. scale mm	Comp. scale mm	Exp to Comp scale ratio	d1(As per comp)								
			(frame no)	T(sec)	dx(mm)	dy(mm)	do Avg	d1Act	S(mm)	δS(mm)	V(mm/Sec)
10	20	0.5	10	0.04	13	7	10	5	18	18	450
10	20	0.5	35	0.14	11	8	9.5	4.75	22	40	157.14
10	20	0.5	60	0.24	14	5	9.5		26	66	108.33
10	20	0.5	85	0.34	11	6	8.5	4.25	24	90	70.588
10	20	0.5	110	0.44	12	5	8.5	4.25	26	116	59.091
10	20	0.5	135	0.54	11	6	8.5	4.25	26	142	48.148
10	20	0.5	160	0.64	11	6	8.5	4.25	23	165	35.938
10	20	0.5	185	0.74	10	6	8	4	25	190	33.784
10	20	0.5	210	0.84	10	6	8		22	212	26.19
10	20	0.5	235	0.94	9	7	8		25	237	26.596
10	20	0.5	260	1.04	9	7	8		22	259	21.154
10	20	0.5	285	1.14	9	6	7.5	3.75	20	279	17.544
10	20	0.5	310	1.24	8	6	7	3.5	20	299	16.129
10	20	0.5	335	1.34	8	5	6.5	3.25	18	327	13.433
10	20	0.5	360	1.44	6	5	5.5	2.75	20	347	13.889
10	20	0.5	385	1.54	6	5	5.5		19	366	12.338
10	20	0.5	410	1.64	6	5	5.5		19	385	11.585
10	20	0.5	435	1.74	6	4	5	2.5	10	395	5.7471
10	20	0.5	460	1.84	5	4	4.5	2.25	18	413	9.7826
10	20	0.5	485	1.94	5	4	4.5		16	429	8.2474
10	20	0.5	510	2.04	5	4	4.5		19	448	9.3137
10	20	0.5	535	2.14	4	4	4	2	16	464	7.4766
10	20	0.5	560	2.24	4	4	4		14	478	6.25
10	20	0.5	585	2.34	4	4	4		15	493	6.4103
10	20	0.5	610	2.44	4	3	3.5	1.75	14	507	5.7377
10	20	0.5	635	2.54	4	3	3.5		15	522	5.9055
10	20	0.5	660	2.64	4	3	3.5		13	535	4.9242
10	20	0.5	685	2.74	3	3	3	1.5	12	547	4.3796
10	20	0.5	710	2.84	3	2.5	2.75	1.375	13	560	4.5775
10	20	0.5	735	2.94	3	2.5	2.75		13	573	4.4218
10	20	0.5	760	3.04	3	2	2.5	1.25	14	587	4.6053
10	20	0.5	785	3.14	2	2	2	1	14	601	4.4586
10	20	0.5	810	3.24	2	2	2		12	613	3.7037
10	20	0.5	835	3.34	2	2	2		14	627	4.1916
10	20	0.5	860	3.44	2	2	2		11	638	3.1977
10	20	0.5	885	3.54	2	2	2		13	651	3.6723
10	20	0.5	910	3.64	1.5	1	1.25	0.625	12	663	3.2967
10	20	0.5	935	3.74	1	1	1	0.5	14	677	3.7433
10	20	0.5	960	3.84	0.5	0.5	0.5	0.25	12	689	3.125

10	20	0.5	985	3.94	0.5	0.5	0.5	0.25	13	702	3.2995
10	20	0.5	1010	4.04	0.5	0.5	0.5	0.25	13	715	3.2178
10	20	0.5	1035	4.14	0.5	0.5	0.5		12	727	2.8986
10	20	0.5	1060	4.24	0.5	0.5	0.5		11	738	2.5943
10	20	0.5	1085	4.34	0.5	0.5	0.5		11	749	2.5346
10	20	0.5	1110	4.44	0.5	0.5	0.5		10	759	2.2523
10	20	0.5	1135	4.54	0.5	0.5	0.5		10	769	2.2026
10	20	0.5	1160	4.64	0.5	0.5	0.5		10	779	2.1552
10	20	0.5	1185	4.74	0.5	0.5	0.5		10	789	2.1097
10	20	0.5	1210	4.84	0.5	0.5	0.5		11	800	2.2727
10	20	0.5	1235	4.94	0.25	0.25	0.25	0.125	11	811	2.2267
10	20	0.5	1260	5.04	0.25	0.25	0.25		10	822	1.9841
10	20	0.5	1285	5.14	0.25	0.25	0.25		10	832	1.9455
10	20	0.5	1310	5.24	0.25	0.25	0.25		10	842	1.9084

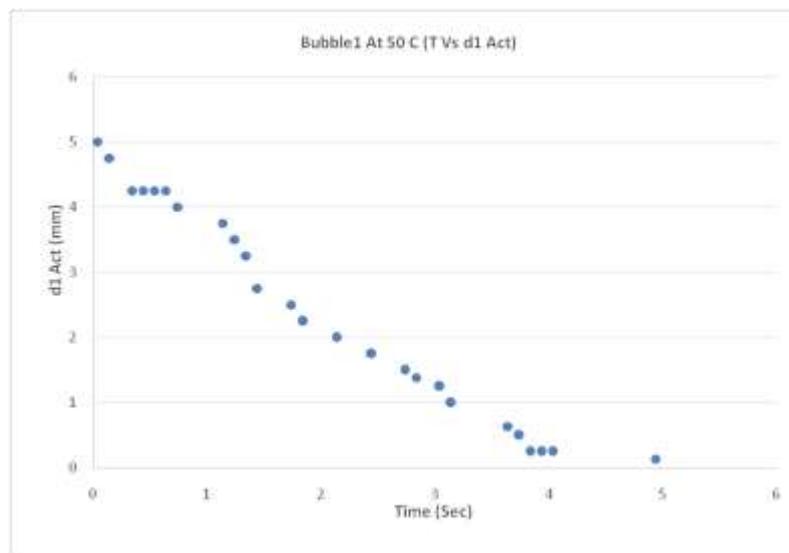


Fig. 3: Decrease in Diameter of Bubble.

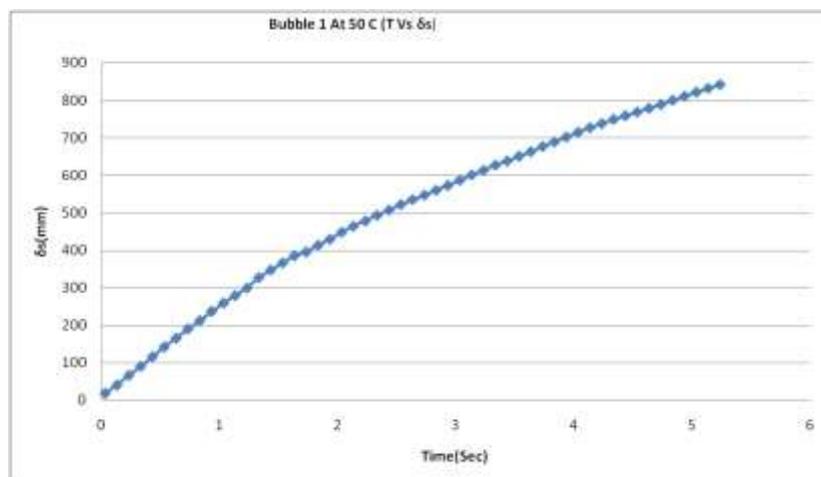


Fig. 4: Position of Bubble.

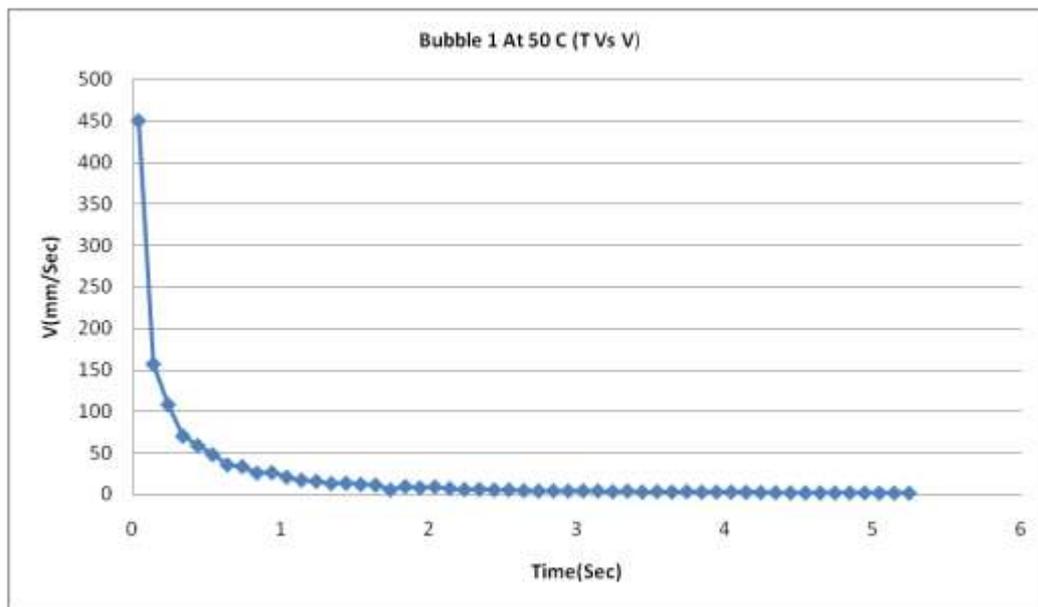


Fig. 5: Decrease in Velocity of Bubble.

Table 2 shows the analysis data for temperature 70°C and for the initial bubble diameter of 3.25 mm. Frame wise analysis was started from the frame number 18 and continued analysis for up to last frame for an interval of 25

frames. The diameter of the bubble and velocity of the bubble decreases with increase in time, velocity of the bubble depends on the diameter of the bubble and also we found that the position of the bubble with respect to time.

Table 2: Analysis for Change in Diameter, Position and Velocity of the Bubble1 At 70° C.

Exp. scale (mm)	Comp. scale (mm)	Exp to Comp scale ratio	Frame No.	t (sec)	Bubble 1						
					d1 (As per comp)			d1 actual	S (mm)	δs	S/t (mm/s)
					dx (mm)	dy (mm)	do avg				
10	20	0.5	18	0.072	7	6	6.5	3.25	20	20	277.7778
10	20	0.5	43	0.172	6	6	6	3	29	49	168.6047
10	20	0.5	68	0.272	7	5	6	3	26	75	95.58824
10	20	0.5	93	0.372	7	5	6	3	27	102	72.58065
10	20	0.5	118	0.472	6	5	5.5	2.75	26	128	55.08475
10	20	0.5	143	0.572	5.5	4.5	5	2.5	24	152	41.95804
10	20	0.5	168	0.672	5	4	4.5	2.25	22	174	32.7381
10	20	0.5	193	0.772	4	4	4	2	23	197	29.79275
10	20	0.5	218	0.872	4	4	4	2	22	219	25.22936
10	20	0.5	243	0.972	3.5	3.5	3.5	1.75	21	240	21.60494
10	20	0.5	268	1.072	3	3	3	1.5	19	259	17.72388
10	20	0.5	293	1.172	2.5	2	2.25	1.125	1	260	0.853242
10	20	0.5	318	1.272	1.5	1.5	1.5	0.75	14	274	11.00629
10	20	0.5	343	1.372	1.5	1	1.25	0.625	15	289	10.93294
10	20	0.5	368	1.472	1	1	1	0.5	17	306	11.54891
10	20	0.5	393	1.572	0.75	0.75	0.75	0.375	15	321	9.541985
10	20	0.5	418	1.672	0.5	0.5	0.5	0.25	14	335	8.373206
10	20	0.5	443	1.772	0.5	0.5	0.5	0.25	13	348	7.336343
10	20	0.5	468	1.872	0.5	0.5	0.5	0.25	14	362	7.478632
10	20	0.5	493	1.972	0.5	0.5	0.5	0.25	11	373	5.578093
10	20	0.5	518	2.072	0.25	0.25	0.25	0.125	12	385	5.791506
10	20	0.5	543	2.172	0.25	0.25	0.25	0.125	12	397	5.524862
10	20	0.5	568	2.272	0.25	0.25	0.25	0.125	10	407	4.401408
10	20	0.5	593	2.372	0.25	0.25	0.25	0.125	10	417	4.215852

10	20	0.5	618	2.472	0.25	0.25	0.25	0.125	11	428	4.449838
10	20	0.5	643	2.572	0.125	0.125	0.125	0.125	10	438	3.888025
10	20	0.5	668	2.672	0.125	0.125	0.125	0.125	10	448	3.742515
10	20	0.5	693	2.772	0.125	0.125	0.125	0.125	10	458	3.607504
10	20	0.5	718	2.872	0.125	0.125	0.125	0.125	10	468	3.481894

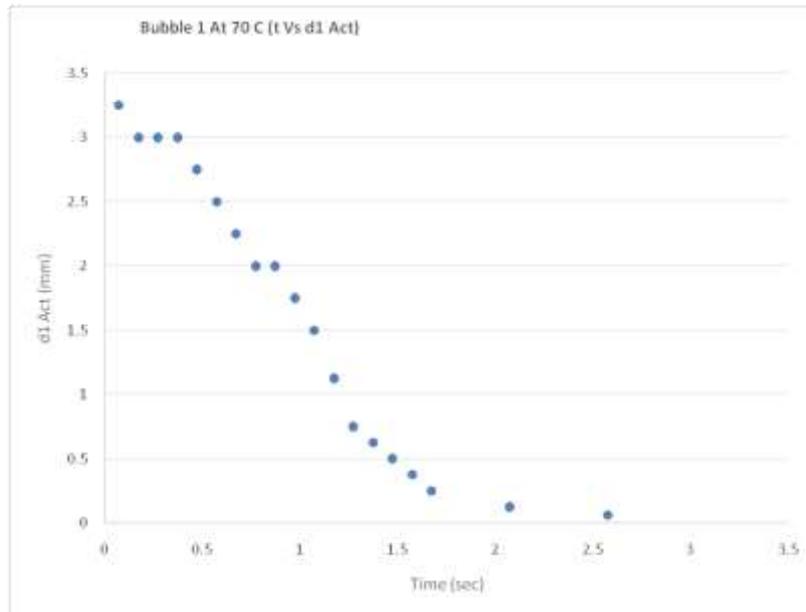


Fig. 6: Decrease in Diameter of Bubble.

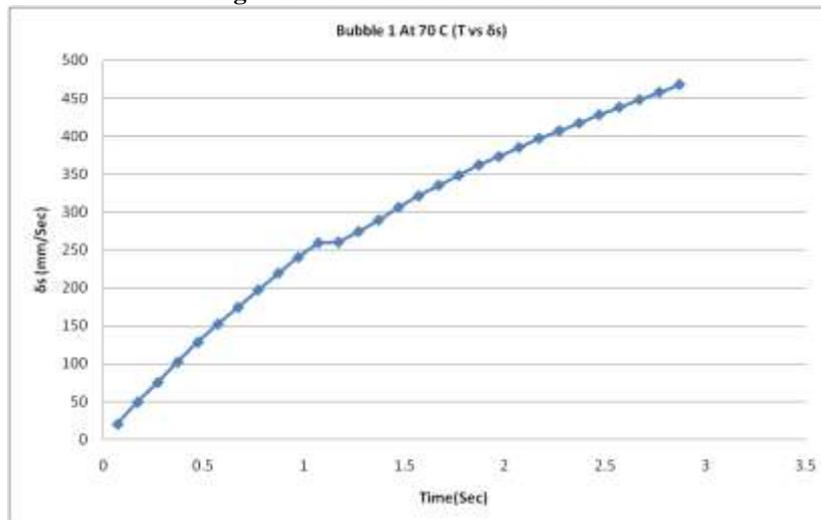


Fig.7: Position of Bubble.

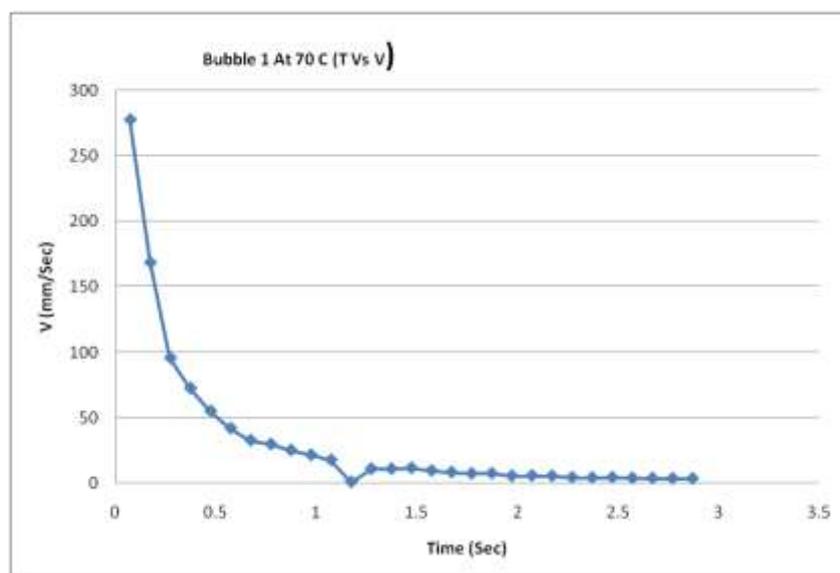


Fig.8: Decrease in Velocity of Bubble.

IV. CONCLUSION

The gas absorption column was designed and constructed with specific dimensions, including a length of 1 meter and a breadth of 0.20 meters. The column had a glass surface, which allowed for visibility during the experiments. Additionally, heating and sparging arrangements were incorporated into the column to control the temperature and introduce carbon dioxide gas. The experiment was conducted at two different temperatures: 50°C and 70°C, using a ratio of 1:2 (presumably referring to the ratio of rice husk ash to sodium hydroxide). At 50°C, it was observed that the bubbles escaped from the surface of the sodium silicate solution, indicating that this temperature was not suitable for achieving complete dissolution of carbon dioxide in the given experimental conditions. On the other hand, from the results of the experiment, it was determined that 70°C was ideal for achieving complete dissolution of carbon dioxide in the sodium silicate solution. At this temperature, the desired dissolution of carbon dioxide was achieved, indicating its effectiveness in the given experimental setup. Overall, the experiment demonstrated that a temperature of 70°C was optimal for achieving the desired dissolution of carbon dioxide in the sodium silicate solution within the parameters and conditions of the study.

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