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A Combined Approach for the Treatment of Textile Dye Bath Effluent Using CO_2 Gas

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Abstract: In this study, baking soda extraction from textile dye bath effluent has been investigated. The novel notion of employing amino acid additions to improve the standard Solvay method and thereby boost the efficiency of Na^+ recovery has been investigated. Glycine, L-arginine, and L-alanine are three amino acid additions examined for their effect on enhancing Na^+ recovery, and the best-suited additive is chosen. The dumping of brackish dye bath effluent, which has a high percentage of sodium chloride, causes textile dye baths from the textile industry. The primary goal was to remove Na^+ (sodium) from the effluent using carbon dioxide gas, which has environmental benefits. Carbon dioxide (CO_2) is the most common greenhouse gas, trapping heat and raising global temperatures, therefore contributing to climate change. The Solvay process is used to transform Na+ in salty wastewater into a valuable product. The effect of different operating variables such as NH_4OH (ammonium hydroxide) concentration, reaction temperature, carbonation time, and carbon dioxide gas flow rate on bicarbonate production was investigated. Maximum sodium recovery of about 68 percent is attained under optimal circumstances. When compared with the regular Solvay process, the modified Solvay method has a greater recovery efficiency (33 percent). Amino acid addition (arginine) improved conversion efficiency while also lowering the process's ammonia need.

Key words: Arginine, carbon dioxide utilisation, baking soda recovery, modified Solvay process.

Introduction

Carbon dioxide is widely employed in the production of greenhouse gases, which gather infrared light and convert it to heat, raising global temperatures and contributing to climate change. The combustion of fossil fuels consumes more than 80% of all energy (Lepaumier et al., 2009; MacDowell et al., 2010; Majchrowicz et al., 2009; Martinetti et al., 2009). When comparing the growth in greenhouse gas emissions, carbon dioxide has the highest concentration (Aishwarya et al., 2010a,b; Venkatesan et al., 2016b, 2015). Carbon dioxide concentrations in the atmosphere grew by 270 ppm during the pre-industrialisation era, and recently this concentration has risen by 415 ppm, which is a 0.7 percent increase in global average temperature during the previous 150 years. CO_2 concentration is gradually increasing to 1.5 ppm each year (Munoz et al., 2019; Mohammad et al., 2016; Ning et al., 2010; Portugal et al., 2009). As of July 2012, carbon dioxide had a volumetric content of 0.039 percent in the atmosphere (Karunanidhi et al., 2021; Sivakumar et al., 2014a,b; Venkatesan et al., 2020). The discharge of wastewater from textile dyeing enterprises ranges between 100 and 200 m^3/t of output (Venkatesan et al., 2012, 2016a, 2019). Salts used in reactive dyeing of cotton include sodium chloride (2580 kg/m^3), which is introduced to a dye solution to help in the depletion of different colours into cloth, whereas

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bases are used to elevate the pH from 7 to 11 in order to accomplish fixing (Sachin et al., 2020; Venkatesan et al., 2022; Woodall et al., 2019; Zhao et al., 2012). The dye bath spent liquor discharged with salts after removing the colour. The precipitation in the removal of NH_{4+} as MAP (magnesium ammonium phosphate hexahydrate; $MgNH_4PO_4{\cdot}6H_2O$) using Mg_{2+} and PO_4^{3-}. Carbon dioxide is absorbed in an ammoniated dye solution, yielding a useful product, sodium bicarbonate (Lauwhoff et al., 1984; Krishnaveni and Palanivelu, 2013; Kumar 2002a,b, 2020; Puxty et al., 2010; Ren et al., 2012; Roberts et al., 2010; Venkatesan et al., 2014, 2018). When carbon dioxide is introduced into an ammoniated brine solution, it interacts with sodium chloride to produce a precipitate of sodium bicarbonate and ammonium chlorides, as shown in the following equation:

$$NaCl + NH_4\,OH + CO_2 \longrightarrow NaHCO_3 + NH_4\,Cl$$

The Solvay process was originally developed for the production of sodium carbonate, in which a concentrated brine solution is reacted with ammonia and carbon dioxide to form soluble ammonium bicarbonate (Aronu et al., 2011; Dugas and Rochelle, 2011; El-Naas, 2011; Erga et al., 1995; Hoff et al., 2004; Jibril et al., 2001; Kilic and Kilic, 2005), which then reacts with sodium chloride to form soluble ammonium chloride and a precipitate of sodium bicarbonate via the following reactions:

$$NaCl + NH_3 + CO_2 + H_2O \longrightarrow NaHCO_3 + NH_4Cl$$
$$2NaHCO_3 \longrightarrow Na_2CO_3 + CO_2 + H_2O$$

The resulting ammonium chloride can be reacted with calcium hydroxide to recover and recycle the ammonia according to the following reaction:

$$2NH_4Cl + Ca\,(OH)_2 \longrightarrow CaCl_2 + 2NH_3 + 2H_2O$$

Experimental Section

Material

Throughout, analytical-grade compounds were used. The textile dye bath reject was manufactured in Tirupur, Tamil Nadu, India. The effluent was ammoniated with an ammonia solution obtained from Merck in India, which comprised 30% w/v NH_3. The carbon dioxide source was 99 percent analytical-grade carbon dioxide gas obtained from Supreme Engineering Services in India. L-Alanine, L-Arginine, and Glycine were employed as amino acids that were bought from Merck in India. Demineralized water was utilized to prepare all experimental solutions.

Apparatus and Procedure

A Plexiglas carbon dioxide gas absorption reactor with an inner diameter of 90 mm and an overall height of 170 mm was employed in this study. Figure 1 depicts this reactor. A textile dye solution of 300 mL from the textile industry was ammoniated and an amino acid addition was made. The combination was then treated to a continuous flow of carbon dioxide gas at a given flow rate and for a set duration of time. Carbon dioxide gas was delivered through a special feeding tube with a diffuser at the end to produce small bubbles and guarantee proper mixing. The tube was extended all the way to the bottom of the reactor tank. The temperature of the reaction was kept constant by using a water jacket facility, which kept the reactor at a low temperature by circulating 15°C cold water around it. Hot water at 40°C was cycled through the reactor to maintain a comparably high temperature. A peristaltic pump was used to recycle the contents of the mixture once it had been improved.

Following the reaction, the solid contents were collected using a bottom-mounted tap.

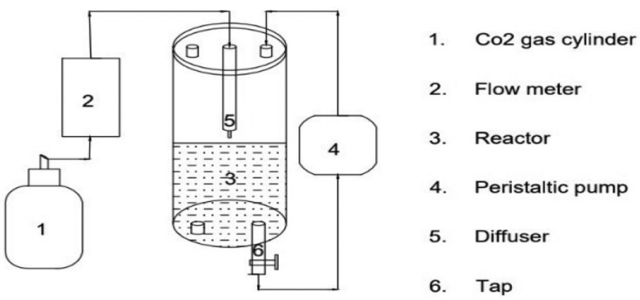

Figure 1: Carbon dioxide capture apparatus.

Analytical Techniques

At the conclusion of each experiment, the reactor's contents were emptied and filtered. The precipitate was collected, dried for 1 hour in a 105°C oven, and used for SEM studies. The SEM photos were taken with an SEM-JSM 6360 and an ion sputter method on a gold target. The sodium content of the filtrate and precipitate was determined using a flame spectrophotometer. The alkalinity titration technique is used. The precipitate was analysed to determine the concentrations of sodium carbonate and sodium bicarbonate. The phenate approach was used to measure the NH_4^+ 17 using a spectrophotometer calibrated to 640 nm.

Result and Discussion

The current work includes a series of batch tests to choose an amino acid addition that may improve the efficiency of the modified Solvay method for recovering Na+ in the form of bicarbonate while utilizing carbon dioxide.

Characteristics of the Textile Dye Bath Reject

The characteristics of textile dye bath's reject collected from the textile industry. The textile dye bath and reject are both somewhat alkaline, and the sodium content and high sodium content are both present in Table 1 textile dye bath reject. Table 1 shows the parameters of the effluent and the optimum values.

Table 1: Parameters of the effluent and the optimum values

Parameter	*Untreated textile effluent*
COD	1588
TDS	86701
Na^+	27070
Cl-	41635
PH	5.5
E C	102

* All values except pH and Electrical conductivity are in mg/L

Identification of Suitable Amino Additive

In this work, sodium bicarbonate recovery from the textile dye bath reject and CO_2 capture using the amino acids Glycine, L-Arginine, and L-Alanine was explored. The reactions were carried out under the following experimental conditions: 300 mL of textile dye bath reject; 27,070 mg/L of Na^+ in the textile dye bath reject; 2 L/min of CO_2 gas flow; 180 minutes of carbonation; and 27°C as the temperature. Figure 2 displays the sodium recovery for the three amino acids when the ammonia concentration was changed from 2 M to 3.3 M.

Both the sodium recovery and the amount of precipitate reclaimed were significantly high when arginine (an amino acid) was used as the addition. The sodium recovery was close to 56 percent when 2.5 moles of ammonia and 0.01 moles of arginine were used. The precipitate's subsequent characterisation indicated that it included a higher concentration of sodium and carbonate. The recovery was higher when 2.5 moles of NH_3 were used for each of the three amino acids under investigation. Figure 2 shows comparative analysis of sodium recovery at different NH_3 concentrations.

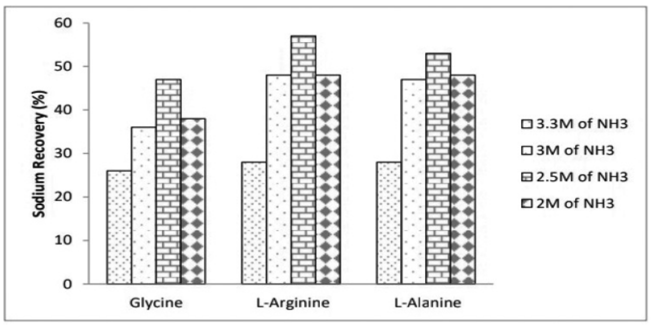

Figure 2: Comparative analysis of sodium recovery at different NH_3 concentrations.

This demonstrates that using the amino acid addition arginine has boosted the Solvay process' effectiveness with regard to salt recovery while reducing the amount of ammonia needed by 1 mole (from 3.3 to 2 moles). These results lead to the conclusion that arginine is the ideal amino acid additive for the Solvay process. As a result, thorough optimisation was done utilising arginine as the additive.

Optimisation of Process Parameter

Effect of Carbon Dioxide Gas Flowrate

For 180 minutes at room temperature, the reactor's mole ratio of [Na^+: NH_3: Amino Acid] was maintained at -1:2.3:0.01, and the flow rate of CO_2 gas was adjusted from 1.2 L/m to 2 L/m (27°C). Figure 1 displays a plot of the percentage of Na+ removed as a function of the gas flow rate. Figure 3(a) represents the flow rate of the CO_2 gas. The greatest recovery was attained at a flow rate of 1.6 L/m as the Na^+ recovery increased along with the CO_2 gas flow rate. Na^+ recovery and precipitate yield decreased with additional increases in flow rates. This demonstrates unequivocally that at greater flow rates, CO_2 gas diffusion in the solution decreased, which led to a decrease in conversion efficiency of 1.6 L/m. Na+ recovery and precipitate yield decreased with additional increases in flow rates. This demonstrates unequivocally that at greater flow rates, CO_2 gas diffusion in the solution decreased, which led to a decrease in conversion efficiency. As a result, 1.6 L/m was determined to be the ideal flow rate for CO_2 gas, with a Na^+ recovery of nearly 62%.

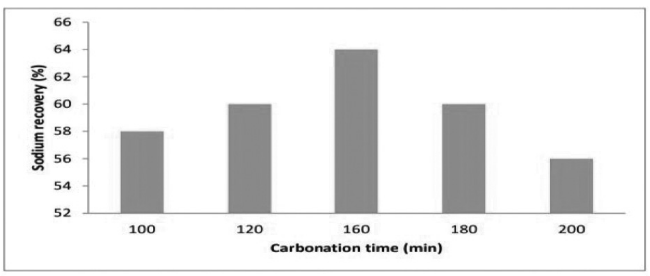

Figure 3a: Flow rate of CO_2 gas (L/min).

Effect of Carbonation Time

Later studies were conducted to examine the effect of carbonation duration on the recovery of Na+ from the textile dye bath reject brine. Under identical conditions, i.e., a mole ratio of [Na^+: NH_3: Amino acid] of -1:2.3:0.01 and a flow rate of CO_2 gas to 1.6 L/m at room temperature, the carbonation time of the process was changed from 80 to 180 minutes. Each case's Na^+ recovery was discovered, and the results are shown in Figure 3d. Initially, as carbonation time rose, so did the recovery percentage, reaching a peak of almost 67 percent at 150 minutes. As the ammonium carbonate equilibrium approached in equations (3,4), the efficiency of the Na^+ ion conversion decreased as the carbonation duration grew. Figure 3c shows the time taken for carbonation in minutes. Ammonium carbonate predominated at the beginning of the conversion, but as more carbonation occurred, ammonium bicarbonate and then sodium bicarbonate were produced. Therefore, 150 minutes was chosen as the ideal carbonation time.

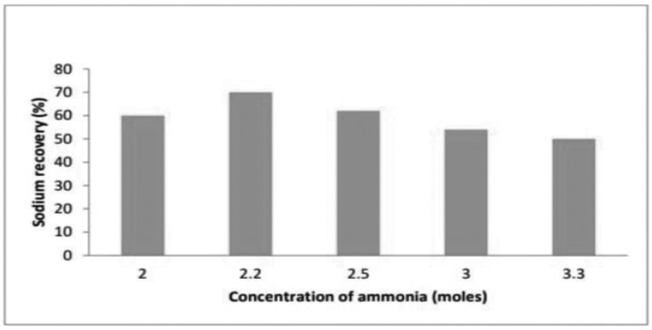

Figure 3b: Carbonation of ammonia (moles).

Effect of Ammonia Concentration

The Na^+ ion recovery was determined for each example while varying the ammonia concentration for the reaction from 1 to 3 moles while maintaining all other parameters constant. The findings are shown in Figure 3b. When the ammonia level was reduced, sodium recovery increased, reaching a maximum of roughly 67 percent for 2.2 moles of ammonia. Further reduction decreased the output because the acidic nature of the reactor content prevented sodium bicarbonate from precipitating due to a shortage of ammonium hydroxide. As a result, the ideal ammonia concentration was determined to be 2.2 moles. The addition of the amino acid additive alanine doubled the salt recovery rate and reduced the quantity of ammonia required for the Solvay process from 3.3 moles (using the classic Solvay technique without the addition of amino acids) to 2.2 moles (with arginine). This study shows that adding the amino acid supplement arginine increased salt recovery from 33 to 67 percent while decreasing the demand for ammonia.

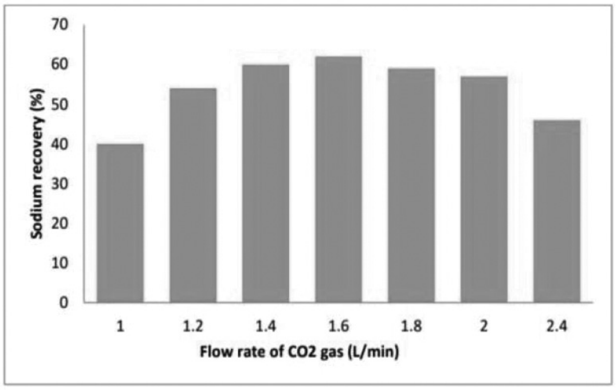

Figure 3c: Carbonation time (min).

Effect of Arginine Concentration

From 0.005 to 0.080 moles of arginine were used in the reaction, which took 150 minutes to complete under the same conditions (mole ratio [Na^+: NH_3] - 1:2.3, room temperature, and 1.6 L/m of CO_2 gas flow). Figure 3e depicts a plot of the proportion of Na+ eliminated vs arginine concentration. When 0.01 moles of arginine were utilised, and the highest recovery % was attained. This form is characterised as a zwitterion because the amino group is protonated and less reactive toward CO_2 at low pH when amino acids are dissolved in water. To create the alkaline medium, a certain quantity of ammonia was added to the aqueous solution, which was then utilised to activate the amino group. Because of the low pH (less than 7) conditions, the yield and salt recovery % fell as the arginine concentration increased. This is similar to the findings by previous researchers.

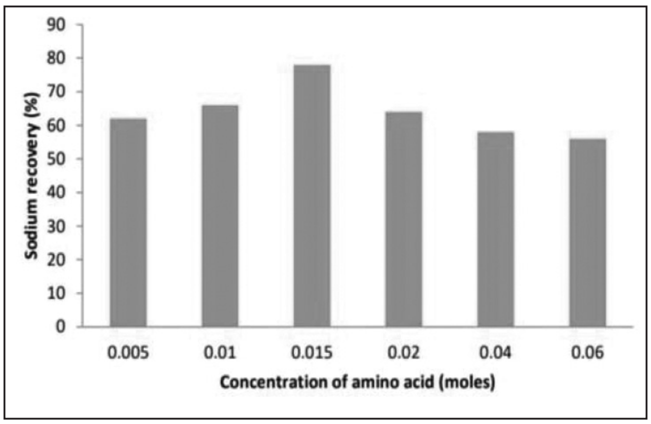

Figure 3d: Concentration of amino acid (moles).

Effect of reaction temperature

The amino acid additive has to be used at a lower dosage i.e. (0.01 moles). It impacts the reaction temperature at a mole ratio of [Na^+: NH_3: Amino acid] and carbon dioxide gas flow rate of 1.6 L/min for 150-minutes. Carbonation has been researched at various reaction temperatures ranging from 15 to 35°C. The temperature of the reaction was kept constant by using a water jacket facility. Figure 3c depicts a plot of the proportion of Na+ eliminated vs arginine concentration. Because CO_2 gas is more soluble at lower temperatures, the amount of CO_2 collected increased with decreasing temperature. The optimal temperature was discovered to be 20°C in this experiment, indicating that 19.3°C is the best temperature. The less ammonia removed from the process, the more stable the reaction within the reactor. As previously stated, the sodium recovery efficiency decreases with increasing temperature.

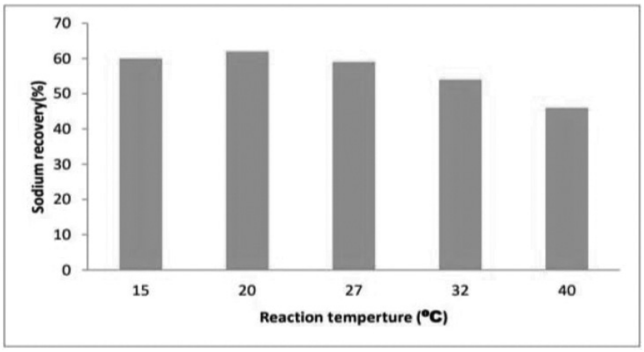

Figure 3e: Reaction temperature (°C).

Characterisation of the Recovered Precipitate

The optimal reaction conditions (mole ratio of [Na^+: NH_3: Amino acid] -1:2.3:0.01, reaction temperature of 20°C, CO_2 gas flow rate of 1.6 L/min, and carbonation time of 150 min) were employed to precipitate sodium bicarbonate. After drying at 105°C for an hour, the precipitate was collected for carbonate and bicarbonate determinations using the alkalinity titration technique. Under optimal conditions, the precipitate contained almost 40% bicarbonate and 60% carbonate. Figure 3e shows the reaction temperature needed for solvent recovery. It is self-evident that the precipitate that developed contained a combination of sodium carbonate and sodium bicarbonate. After the carbonation process was continued for 190 minutes, the precipitate's bicarbonate level increased to almost 90%. Figure 4 displays the precipitate's bicarbonate and carbonate content for various carbonation times, as well as the pH variation with reference to the carbonation duration. The pH of the solution decreased from 10.2 to 8.5 as the carbonation period extended from 120 to 190 minutes, as shown in Figure 4. The carbonate concentration of the precipitate was about 80 percent at 120 minutes, although it steadily increased in bicarbonate content from 120 to 190 minutes.

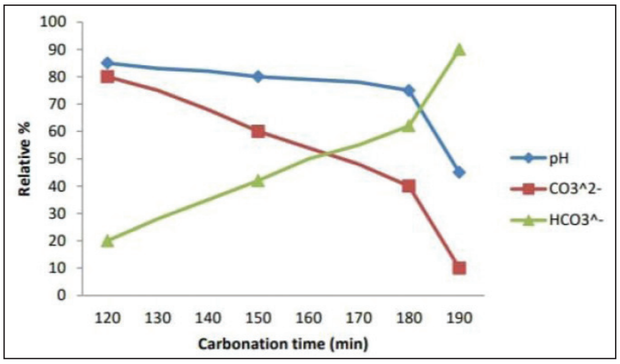

Figure 4: Speciation of carbonates and bicarbonates.

Conclusion

In this study, amino acid additions were used to improve the recovery of baking soda from textile dye bath rejection using the standard Solvay technique. After evaluating the additive amino acids, such as glycine, L-alanine, and L-arginine, alanine was identified as the correct amino acid. The modified Solvay technique for sodium recovery was 70% efficient at a flow rate of 1.6 (L/min) and carbonation time of 150 minutes using 2.5 moles of ammonia and 0.01 moles of arginine at a temperature of 20°C.

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