

Handling heat-stable salts in post-combustion CO₂ capture: A detailed survey

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Abstract: The generation of heat-stable salts (HSSs) in alkanolamine solutions for CO₂ capture processes, which is adapted for power plant technologies, exists irrespective of the class of amine solution used for the capture process. Their presence do not only trigger decrements in the CO₂ absorption capacities of the solvents and contribute to further alkanolamine degradation, but also result in foaming and loss of solvents, which impacts system economics and threatens the environment. HSSs also promote the corrosiveness of the metallic structures of capture systems by lowering the pH and increasing the conductivity of the absorbent solutions. Overall, these effects substantially subvert the reliability and integrity of CO₂ capture units. This survey affords sufficient background on the existence of HSSs by unraveling the flow process in a typical alkanolamine-based CO₂ capture unit with respect to their formation points and potential threats. Furthermore, the major HSSs removal and alkanolamine reclamation methodologies (electrodialysis, distillation, ion exchange, electromagnetic separation, and solvent extraction) were comprehensively explored. We believe that this review paper will benefit researchers across disciplines as we continue to explore new and complex solvent formulations to minimize the cost of CO₂ capture while maximizing efficiency. © 2023 Society of Chemical Industry and John Wiley & Sons, Ltd.

Keywords: Distillation; electrodialysis; heat-stable salt; ion exchange; post-combustion capture; reclamation

Introduction

Fossil fuels have become the largest sources of energy for electricity generation worldwide and their global share is on the increase.^{1,2} Despite many nations diversifying their energy sources, the dependence on fossil fuels for global energy demand is still expected to last for several more decades.³ This means that the threats of CO₂ emission and the greenhouse effect will continue to attract interesting discussions. Consequently, global leaders, industries, and research have shown increasing interest in abating CO₂ emissions while still optimizing the demand and consumption of fossil fuels. One of such interventions is the invention of carbon capture systems (CCS) and their successful improvements in recent years.^{4–6} Among the different CCS technologies, post-combustion CO₂ capture (PCC), especially the chemical absorption process which employs solvents as absorbents, has been distinguished as a very promising technology for CO₂ removal from industrial flue gases.^{7–9}

The potential of several solvents for CO₂ removal in PCC units has been evaluated, and the primary factor for consideration is that the absorbed CO₂ gas satisfies the requisite purity specification while the secondary factor is that the solvent optimizes equipment size, and it is cost effective in terms of plant operations. Generally, alkanolamines are the most widely applied solvents and they have been accepted as their reliabilities are tested over different operating conditions.¹⁰ They possess two polar functional groups (amino and hydroxyl groups) on either side of a chain of methylene groups which are fundamental to their performances in CO₂ removal. While the amino group reacts with the CO₂ molecule, the hydroxyl group maintains low saturated vapor pressure and strong intermolecular bonds.¹¹ They are classed as primary, secondary, or tertiary alkanolamines, and each class has an accepted range of process conditions associated with it based on its merits and demerits.

The generation of heat-stable salts (HSSs) in absorbent solutions exists irrespective of the type of alkanolamine employed for the CO₂ capture process. HSSs are the reaction products of alkanolamines with acids considerably superior in strength than CO₂.¹² The sources of the acids can either be by direct introduction from the flue gas constituents or the products of alkanolamine degradation. HSSs are

Table 1. Characteristic flue gas constituents¹⁷.

Constituent	Amount (% mole)
CO ₂	7–15
H ₂ O	5–15
O ₂	2–12
N ₂	65–75
NO _x	1–400 ppm
SO ₂	2–400 ppm
SO ₃	1–10 ppm

formed from soluble acidic flue gases such as SO_x and NO_x which are important flue gas components (see Table 1). Alkanolamine solvents degrade either through oxidative or thermal processes to form HSSs.¹³ By their very nature, HSSs are nonregenerable during the solvent regeneration phase of CO₂ capture processes. Consequently, they stay put and accumulate in the absorbent solution during plant operations. Their presence not only triggers a decrease in the absorption capacities of the solvents and contribute to further alkanolamine degradation, but also results in the loss of solvents, which impacts system economics and threats to the environment. HSSs also promote the corrosiveness of the capture system by lowering the pH and increasing the conductivity of the absorbing solution.^{14,15} Aside from impacting the strength and safety of the construction materials for capture units and installations, the increments in corrosion rate also increase the amount of corrosion product scales in the bulk solutions, thereby triggering other operational challenges, like solution foaming and fouling.^{11,16} Overall, these effects substantially subvert the reliability and integrity of CO₂ capture units.

To fully understand the influence of HSSs in CO₂ capture units, it is very important to conduct an in-depth assessment of research studies completed so far. While the earlier studies will be useful in providing reliable background and history of the existence of HSSs in capture units as well as the challenges they have posed over the years, the more recent studies will offer updated information on the successes of alkanolamine reclamations and HSSs removal procedures. Being comprehensive in its approach, the review paper will benefit both beginners and experienced researchers across different disciplines.

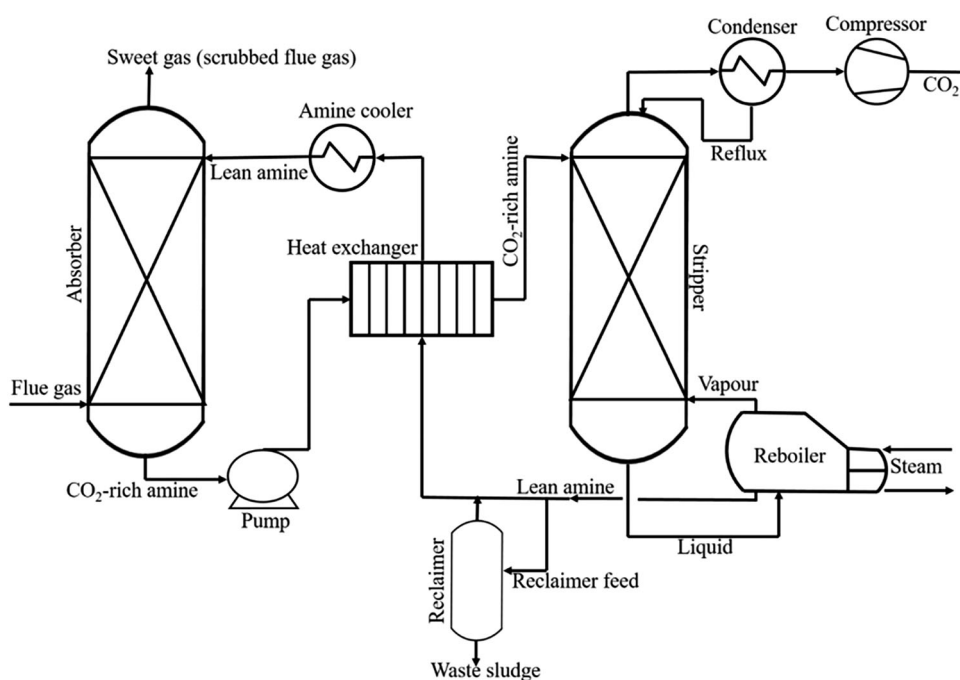


Figure 1. Flow sketch of alkanolamine-based PCC process.

Overview of a typical alkanolamine-based PCC unit adapted for power plant technology

In PCC technology, the fossil fuels are treated like typical energy generation systems, and the CO₂ is secured from the discharged gas at the exhaust after combustion. This PCC approach is widely used because it can be easily reconstructed to fit already existing CO₂ sources like power plants.¹⁸

Figure 1 shows a typical alkanolamine-based CO₂ capture unit adapted for power plants. The unit consists of the following features: absorber column, stripper column, heat exchangers for rich/lean alkanolamine solvents, reboiler, booster pumps, partial condenser, reclaimer unit, and cooler. The working temperature of the absorber column fluctuates in the range of 40–70°C and the pressure ranges from ambience to 20,000 Kpa, and is hugely reliant on the pressure of the flue gas.^{19,20} The absorber works in counter-current flow mode²¹; herein, the flue gas is first introduced into the absorber, then the absorbent (aqueous alkanolamine solution) enters the absorber from the top leading to a counter-current interaction. While flowing down the column, the aqueous alkanolamine solution absorbs acidic species from the flue gas and becomes saturated. It is good to note that HSSs are formed in the absorber column following the reaction of alkanolamines with

the constituent acidic species besides CO₂ and H₂S in the inlet process stream to the absorber.²² The saturated aqueous amine solution, otherwise known as CO₂-rich alkanolamine solution, exits the absorber column to a lean/rich heat exchanger unit where it is preheated by the hot regenerated lean amine solution coming from the reboiler before entering the stripper column at ~100°C temperature.¹⁹

In the stripper column, the temperature is operated in the range of 105–120°C and the pressure varies between 150 kPa and 200 kPa. The rich amine solution is heated by the stripping vapor from the reboiler resulting in the release of CO₂ from the solution and the regeneration of amine.²³ The stripping vapor is generated from the reboiler where the supplied steam is between 200 and 250°C and the pressure lies around 400 and 500 kPa.¹⁹ The stripping vapor and the released gases leave the stripper column via the top to a partial condenser where the stripping vapor condenses and is sent back to the stripper column as a reflux flow. The CO₂ is compressed for transport and sequestration while the acid gases are either discharged into the atmosphere or transported to a desulfurization plant. Furthermore, the regenerated alkanolamine exits the lean/rich amine heat exchanger at 60°C and goes through additional cooling in the cooler to decrease its temperature to ~40°C, which is the ideal inlet

Table 2. Composition of lean amine solution.

Species	MDEA from South pars gas complex ²⁵	MDEA from Ilam gas refinery ²⁶	DEA from Kharg liquid gas complex ²⁷
DEA	<0.1 wt%		
MDEA	39.8 ± 1.1 wt%		
MEG	2.5 ± 0.2 wt%		
H ₂ O	56 ± 1.2 wt%		
Succinate	242 ppm		
Formate	146 ppm	2354 mg/L	121 ppm
Acetate	633 ppm	2335 mg/L	86 ppm
Glycolate	<1 ppm		
Nitrate	4 ppm		
Nitrite	<1 ppm		
Propionate	99 ppm		
Butyrate	108 ppm		
Chloride	2080 ppm	49 mg/L	350 ppm
Thiosulfate	680 ppm	1843 mg/L	232 ppm
Sulfate	131 ppm	544 mg/L	243 ppm
Sulfite	124 ppm	176 mg/L	
Phosphate	7 ppm		
Thiocyanate	6 ppm		
Oxalate	24 ppm	574 mg/L	153 ppm

temperature for CO₂ capture processes. Meanwhile, slipstream of lean amine from the stripper is sent to the reclaiming process in order to eliminate HSSs and high molecular weight degradation products while the reclaimed solvent is returned to the capture unit.²⁰ In different HSS recovery studies, the composition of the investigated lean amine solutions revealed significant presence of different HSSs in varying compositions (Table 2).

CO₂ absorption reactions of alkanolamines

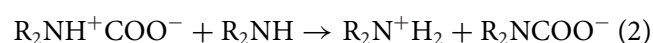
The chemical solvent method of PCC frequently employs amines as absorbents. For this process, monoethanolamine (MEA), diethanolamine (DEA), and N-methyldiethanolamine (MDEA), representing primary, secondary, and tertiary alkanolamine, respectively, are all industrially significant amines.²⁴ The reactions between CO₂ and amine solution, on the other hand, proceed at a finite rate that varies depending on the amine.

Earlier, it was thought that all alkanolamines had a straightforward mechanism and kinetics for this reaction. MEA, DEA, and TEA, have all had significant data discrepancies reported; however, Danckwerts suggested a thorough reaction mechanism that basically covers all the kinetic details involving primary alkanolamines and secondary alkanolamines, as shown in the following²⁸:

Zwitterion formation:



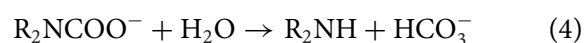
Deprotonation of zwitterion or formation of carbamate:



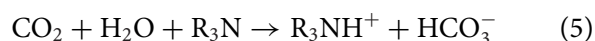
Protonated amine formation:



Bicarbonate formation:



The zwitterion mechanism describes the reaction involving primary and secondary amines with CO₂, which was first proposed using a number of amines. As shown above, the absorption of CO₂ by an aqueous alkanolamine solution two involves two steps: formation of zwitterion and deprotonation of zwitterion (also known as the formation of carbamate). A similar research reached the conclusion that tertiary alkanolamines serve as base catalysts for CO₂ hydration.²⁹



The following equations depict the comprehensive reaction mechanism:



Moreover, recent studies have involved the utilization of sterically hindered amines as more appealing absorbent options compared with the conventional amines, from a commercial standpoint. To boost the cyclic absorption capacity for CO₂, Sartori et al. proposed the utilization of branched amines like 2-amino-2-methyl-1-propanol (AMP),³⁰ noting that steric effects impact the stability of formed carbamates. Despite reports of the thermodynamic and physicochemical characteristics of this system in an

earlier investigation,³¹ studies on the reaction kinetics of CO₂-AMP are relatively scarce.

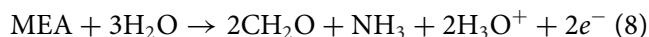
Formation of HSSs

There is an existent formation of HSSs in absorbent solutions irrespective of the type of amine employed for the CO₂ capture process. As previously mentioned, HSSs result from reactions of alkanolamines with acids that are substantially greater in strength than CO₂. The sources of the acids are either by direct introduction from flue gas constituents or as products of amine degradation. Generally, HSSs develop from soluble acidic flue gases like SO_x and NO_x which are critical flue gas constituents. Oxidation and thermal degradation are the two known routes for amine solvents degradation.

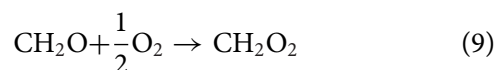
Oxidative degradation

Oxidation degradation of alkanolamines occurs during the CO₂ absorption processes at minimal temperatures of about 55°C under oxidizing conditions. It is a major cause of solvent loss in flue gas containing about 3%–5% oxygen content. In addition to oxygen, other oxidizing species such as NO_x, SO_x, free radicals, and metal ions can also contribute to the oxidative degradation process.³² This processes result in the loss of alkanolamines by the degradation of their molecules and the generation of HSSs.³³ The main mechanisms are the direct oxidation of the alkanolamines to organic acids and the indirect interaction of oxygen and other oxidizing species to form intermediate products, which then combine with the amines to produce other degradation compounds.³⁴ The degradation process is initiated by the radical loss of electrons from the nitrogen in the alkanolamine or the loss of hydrogen from the carbon atom α or β to the nitrogen. The oxidative degradation products of alkanolamine are usually grouped into primary and secondary species. While the primary degradation products represent oxidized species generated directly from alkanolamines such as aldehydes, ammonia, amines, amides, carboxylic acids and organic acids,³⁵ secondary degradation products are generated from the primary products, and they comprise formates, oxalates, acetates, succinates and glycolates such as N-(2-hydroxyethyl)glycine, N,N'-bis(2-hydroxyethyl)oxalamide, N-(2-hydroxyethyl)acetamide, 1-(2-hydroxyethyl)piperazine-2-one, 2-hydroxy- γ -N-(2-hydroxyethyl)acetamide, and

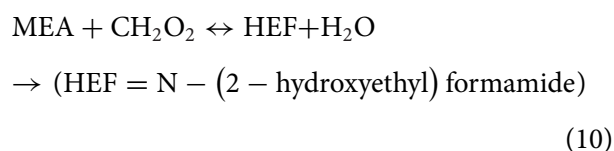
1-(2-hydroxyethyl)piperazine-2-one.^{36,37} Acetate and formate from MEA, as well as N-(2-hydroxyethyl)acetamide and N-(2-hydroxyethyl)formamide, are formed from acetic and formic acids. The formation of HSSs with MEA is also favored by glycolic acid. However, its amide is unstable and mostly hydrolyzed back into glycolates and MEA. Moreover, oxalic acid mainly acts as intermediary specie that decomposes into formic acid, and then generates a stable N-(2-hydroxyethyl)formamide, while succinic acid is converted through an intermediary specie of N-(2-hydroxyethyl)succinamide into a stable N-(2-hydroxyethyl)succinimide.³³ For example, in their work on the modeling of MEA oxidative degradation, Pinto and colleagues presented the following reaction pathways:



This depicts formaldehyde formation. The reaction is assumed to proceed via a hydrogen abstraction mechanism with the aid of a radical initiator. In this case, oxygen creates the required radical for the reaction.



Equation (9) illustrates formic acid formation, which is a simple oxidation reaction of the formaldehyde produced in Eqn (8). Aldehydes undergo quick oxidation reactions to acids even when exposed to air



The pathway reveals the formation of glyoxal via a two-step reaction. First, MEA was reduced to acetaldehyde and ammonia, followed by the oxidation of acetaldehyde to afford glyoxal and water. Assuming a pseudo steady state for the acetaldehyde, Eqns (8) and (9) can be combined, as presented in Eqn (10).

Thermal degradation

Over the past decades, the thermal degradation of alkanolamines during PCC has been comprehensively investigated.^{35,38–40} This form of degradation sometimes referred to as carbamate polymerization takes place in the stripper section at higher temperatures. However, the degradation process can also take place in the heat exchanger and reclaimer

sections.⁴¹ Although, the exact reaction temperature has not yet been established, most studies report temperatures between 100 and 200°C which basically increases with increasing CO₂ loading.^{36,42,43} Higher molecular weight organic compounds like ethylene oxide (EO), methanol, trimethylamine (TMA), methylaminoethanol, MEA, DEA, triethanolamine, dimethylethanolamine, N,N,N-tris (hydroxyethyl) ethylenediamine, N,N-dimethyl-acetamide, N,N-bis (hydroxyethyl) piperazine, N,N-bis(2-hydroxyethyl) glycine (bicine), and HSSs have been identified by various researchers as the main products of the thermal degradation of MDEA.^{38,44} Ahmad et al. also identified low molecular weight compounds formed from organic acids such as acetates, nitrates, sulfates, thiosulfates, glycolates, and formate while investigating the formation of HSSs during thermal degradation of MDEA solvent.³⁹ Moreover, it is documented that the degradation products obtained from fresh alkanolamine solvents differ slightly from those gotten from industrial lean alkanolamines.⁴⁵ Additionally, the degradation products of MDEA degradation vary from those obtained from MEA. The principal products for the thermal degradation of MEA are recognized as N-(2-hydroxyethyl)imidazole (HEI), 1-(2-hydroxyethyl) imidazolidone (HEIA), N-(2-amino-ethyl)-N'-(2-hydroxyethyl) imidazolidin-2-one (AHEIA), N-(2-hydroxyethyl) ethylenediamine (HEEDA), and 2-oxazolidinone (OZD).^{46,47} Kim and Sartori first proposed the formation mechanisms of these compounds,⁴⁸ before Davis et al. further expanded on their findings.⁴⁶ The carbamate species is believed to undergo cyclization leading to the formation of OZD. Then, the species undergo ring-opening reactions with another molecule of MEA to form HEEDA. Once transformed, HEEDA is converted to HEIA or AHEIA.⁴⁹

Several factors influence the oxidation and thermal degradation of alkanolamines, some of which include the type and characteristics of oxidants, type and characteristics of alkanolamines, oxygen concentration, temperature, CO₂ loading, oxidizing conditions, pH of the system, and concentration of the amine species, etc.^{41,50–53} The existence of O₂ and CO₂ influences the stability of alkanolamine solvent against thermal and oxidative degradation. Bougie and Illiuta found that the sterically hindered alkanolamines (such as AMP) were more resistant to thermal degradation compared to their unhindered counterparts (MEA, MDEA).⁵³ However, their oxygen degradation is significant in the

absence of CO₂. Dissolved oxygen increases oxidative degradation with the generation of organic acids. A higher acid content increases the corrosion potential resulting in more HSS formation.

Operational problems caused by HSSs

The presence of HSSs in absorbent solutions is associated with reductions in the removal capacities of CO₂, lowering of amine pH, increments in conductivity, dissolution of protective films, and initiation of corrosion. Moreover, the solid particulates in amine-based solutions accompanying the presence of HSSs cause velocity-accelerated corrosion leading to erosion, hence encouraging the removal of protective layers. We shall therefore enumerate the operational challenges caused by HSSs in this subsection.

Corrosion/erosion

As highlighted in the red segments of Fig. 2 (an adaptation of Fig. 1), the locations in absorption units that are prone to corrosion attacks are usually those involving high levels of acid gas loading and elevated temperatures, example are the absorber (lower section), condenser, regenerator (whole section), heat exchangers, pumps, and valves. Experts have identified the following as the leading factors influencing metal corrosion in alkanolamine units^{54–56}: temperature, solution velocity, amine loading, CO₂ and O₂ loading, concentration and type of alkanolamine solution, contaminants in alkanolamine solutions including HSS. Also, several other researchers have confirmed the influence of temperature in power plant-related corrosion problems in addition to material type and flue gas composition (leading to HSS generation).^{57–60} In addition to reducing the CO₂ absorption capacity of alkanolamines, HSSs accumulation has also been proven to significantly increase the corrosiveness of the absorbing solutions.^{61–63} Beside compromising the material safety of absorption units, and substantially affecting the economics of the system,⁶⁴ increased solution corrosiveness also leads to increased amounts of iron carbonate particles within the solution, which triggers other operation complications like foaming and fouling.^{65,66} This ultimately threatens the efficiency of absorption units, especially with regards to operation and output.

Xiang and colleagues investigated the influence of various operational parameters (such as concentration

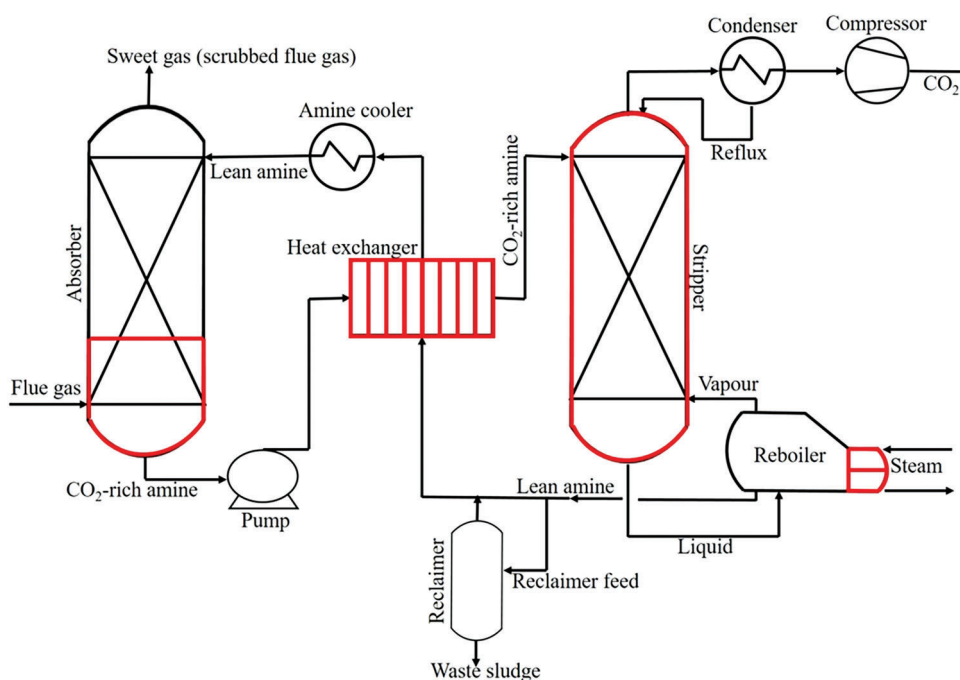


Figure 2. Corrosion-susceptible areas in a typical alkanolamine absorption unit.

of oxygen, presence of HSSs, temperature, and solution velocity) on the corrosion behavior of carbon steel in MEA solution,⁶⁷ and they showed that the presence of HSSs caused elevated corrosion phenomenon at early exposures, but their effects were weakened on prolonged exposure. The study inspired investigations by Emori and coworkers,^{12,68–70} where they investigated the deterioration behavior of steel in HSS-containing environments for CO₂ capture systems. Of particular interest was time and temperature considerations on the effect of sodium sulfide for carbon steel corrosion. They noted that while sulfide impressively inhibited the corrosion of steel at low concentrations, the corrosion resistance automatically decreased as sulfide concentration increased. Moreover, temperature increment also declined the inhibitive effect of sulfide. Similarly, the corrosivity of six kinds of HSSs (namely, formate, malonate, succinate, oxalate, glycolate, and acetate) were investigated on carbon steel material by Tanthapanichakoon and coworkers, and they found that while all the tested salts increased the corrosion potential of the absorbing solution to different extents, oxalate exhibited the most corrosiveness.¹⁵

Moreover, erosion–corrosion is another operational problem in alkanolamine units initiated by high solution speeds, solution turbulence, and the impinging gas streams and alkanolamine solutions on

the surfaces of metal structures. These detach protective oxide layers originally shielding pipes and other structures from corrosion.^{71–74} When HSS anions are in excess in alkanolamine solutions they may crystallize and precipitate into several salts.⁷⁵ Hence, the consequence of erosion–corrosion is further intensified by the existence of suspended particles like HSSs in alkanolamine solutions.

Foaming of absorbing solvent

Foaming has been studied recently as one of the operational issues brought on by the production of HSSs. Foam formation contributes to decreased effectiveness and triggering financial losses since it causes the direct loss of solvent and the indirect modification of the solution's composition. After the formation of HSSs, both the changes in the solution's surface-active properties and its higher viscosity contribute to the enhancement of foaming. Foaming of absorbing solvents have negative effects, including increased absorbent loss due to drop entrainment, pressure drop, increased column flooding probability, and decreased gas/liquid contact surface, which also lowers mass transfer efficiency and reduce the quantity of CO₂ capture.^{76,77} Previous studies identified the following factors to be responsible for foaming of

alkanolamine solutions^{78,79}: naturally-occurring organic acids as constituents of inlet gases, suspended corrosion inhibiting agents, liquid hydrocarbons, surface active agents in lubricants for valves and oil for rotary equipment, injection of surplus antifoam agents, and HSSs. The most common approach to arresting foaming is the use of antifoaming agents, such as high boiling alcohols and silicone oils.^{80–82}

Mansooria et al. investigated the foaming ability of lean MDEA solutions to determine the specific design parameters of an ion exchange resin procedure,²⁵ and they reported that the presence of HSS was associated with increased foaming and reduced CO₂ loading of the MDEA solutions. In their study on influence of HSSs on the absorption–desorption capacity of aqueous MEA solvent for PCC, Ling and coworkers found that foaming properties play more superior roles in the absorption rate of CO₂ in contrast to the decline of initial pH values or the declining levels of mobile amine molecules.⁸³

Effect on operational conditions (physicochemical properties) of the absorbing solvent

Process equipment are designed to work with absorption liquids that fall within a certain range of physicochemical properties. Absorbing solvents become more dense and viscous when HSSs and other degradation products are present in high concentrations,^{83–85} decreasing the efficiency of heat and mass transmission in the existing equipment and increasing the power required to circulate solutions in the system. Changes in the surface tension of alkanolamine may cause the foaming in the solution, and the influence of foaming has been previously described.⁸⁶ It is noteworthy that the presence of excess of HSS anions in solution can trigger the precipitation and crystallization of different salts.⁷⁷ As solid particles become excess in the solvent, the equipment's components become fouled and plugged, producing precipitates in stagnant zones throughout the PCC system. The accumulation of precipitates in the heat exchanger and reboiler sections decreases heat transfer efficiency, thereby increasing the overall power required for the process. So as not to impair system performance, the content of HSSs and other degradation products should be maintained at specific levels. This is because high HSS concentrations cause the buildup of carbonate and bicarbonate as well as

more dissolved iron in the solution. Because HSS anions have chelating properties, an increase in HSS concentration can make amine absorbing solvents more corrosive. This, in turn, causes the pH of the solution to drop and its electrical conductivity to rise, which damages the protective coating on metal surfaces within the PCC system, as previously mentioned in this article.^{15,87–88} Additionally, iron and chromium ions generated from equipment corrosion can reduce the thicknesses of pipes and equipment, increasing current densities on the anodic sites, and necessitating frequent filter replacements.^{89–91} According to Rooney and Bacon,⁹² the overall HSS amount should be maintained around 0.5% of the total solution. Experts from MPR Services Inc. advise keeping the maximum HSS concentration ≤ 5000 ppm.

It is also important to note that with increasing temperature, the amount of HSS and other degradation products also increases. Temperature is one of the most extensively researched parameters in the reaction kinetics of CO₂ with chemical absorbents because it affects a variety of reaction equilibria, such as those for CO₂ absorption, carbamate formation, carbamate hydrolysis, solution pH, and solubility of chemical species, and because it aids in resolving problems with amine solvent performances. Consequently, corrosion reactions are sped up when solution temperature is raised.^{15,93,94} One study stated that since the CO₂ solubility of AMP is lower than that of MEA, there was typically less CO₂ loading while using AMP at higher temperatures compared with MEA.⁹⁴ Ju et al. compared some physicochemical properties (e.g. density, viscosity, refractive index, and electrical conductivity) of lab-based and industry-based degraded MEA solutions at standard temperature conditions to evaluate the influence of HSSs on the physical properties of the absorbing solvent.⁹⁵ They reported that the physicochemical properties displayed massive dependence on the mass fraction of MEA, mass fraction of HSSs, and CO₂ loading. They conducted empirical correlations for the lab-based and industry-based degraded solutions and concluded that the correlation of the industry-based degraded solution offered more reliability for density estimation compared with the lab-based solution.

Effect on CO₂ absorption capacity

According to an analysis of the reactions shown in Fig. 3, at least 1 mol of the alkanolamine must be

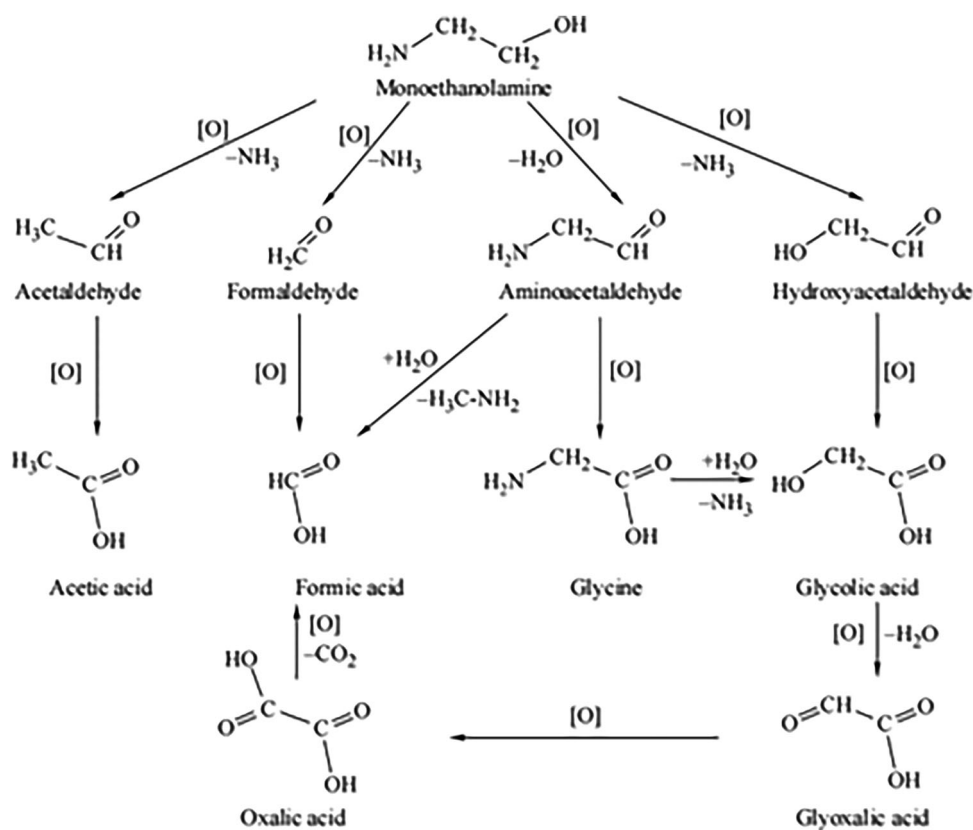


Figure 3. Scheme of thermochemical degradation of monoethanolamine with the formation of carboxylic acids.⁹⁶

consumed for 1 mol of an organic acid anion to form. With a ratio of at least 1:1, the newly produced anions are bound to the active alkanolamine. As a result, the absorbent's ability to bind CO₂ decreases, which affects the performance of the system as a whole. In this instance, PCC system experts are recommended to increase the circulation rates of the absorbing solvents and the steam supply to the desorption column.¹¹

Wibowo et al. studied the effect of absorption pressure and temperature on the CO₂ absorption capacity and rate of the deep eutectic solvent composed of choline chloride and monoethanolamine (ChCl-MEA DES). The researchers also investigated how the addition of water into ChCl-MEA DES affected the solvent's ability to absorb CO₂.⁹⁸ The batch absorption experiment run was determined by the Box–Behnken experiment design, and the data were analyzed using response surface methods (RSM). According to their findings, the initial operating conditions had significant impact on the ability of ChCl-MEA DES to absorb CO₂, with pressure

appearing to have a positive influence and temperature to have a negative one. The greatest CO₂ absorption capacity measured was 0.379 mol CO₂/mol DES, which indicates that physical adsorption was the predominant method of CO₂ absorption by ChCl-MEA DES at 40 °C and 10 bar (Fig. 4A). For absorption processes at the same initial pressure and temperature, it was discovered that water addition up to 50% V approximately doubled the CO₂ absorption capacity of ChCl-MEA DES (with range value of 0.206–0.379 mol CO₂/mol DES capacity compared to neat DES. Their research into gas analysis revealed that ChCl-MEA DES performed better at lower temperatures (Fig. 4C) and greater absorption pressure, with CO₂ concentration reducing from 85.14% to 77.13% as pressure climbed from 5 to 10 bar (Fig. 4B), primarily due to increased amount H₂ absorbed. These findings shed light on how to develop a ChCl-MEA DES that effectively absorbs CO₂ while consuming little energy and doing so under ideal operating conditions.

Aronu et al. investigated the vapor liquid equilibrium of three different MEA solutions at low and high

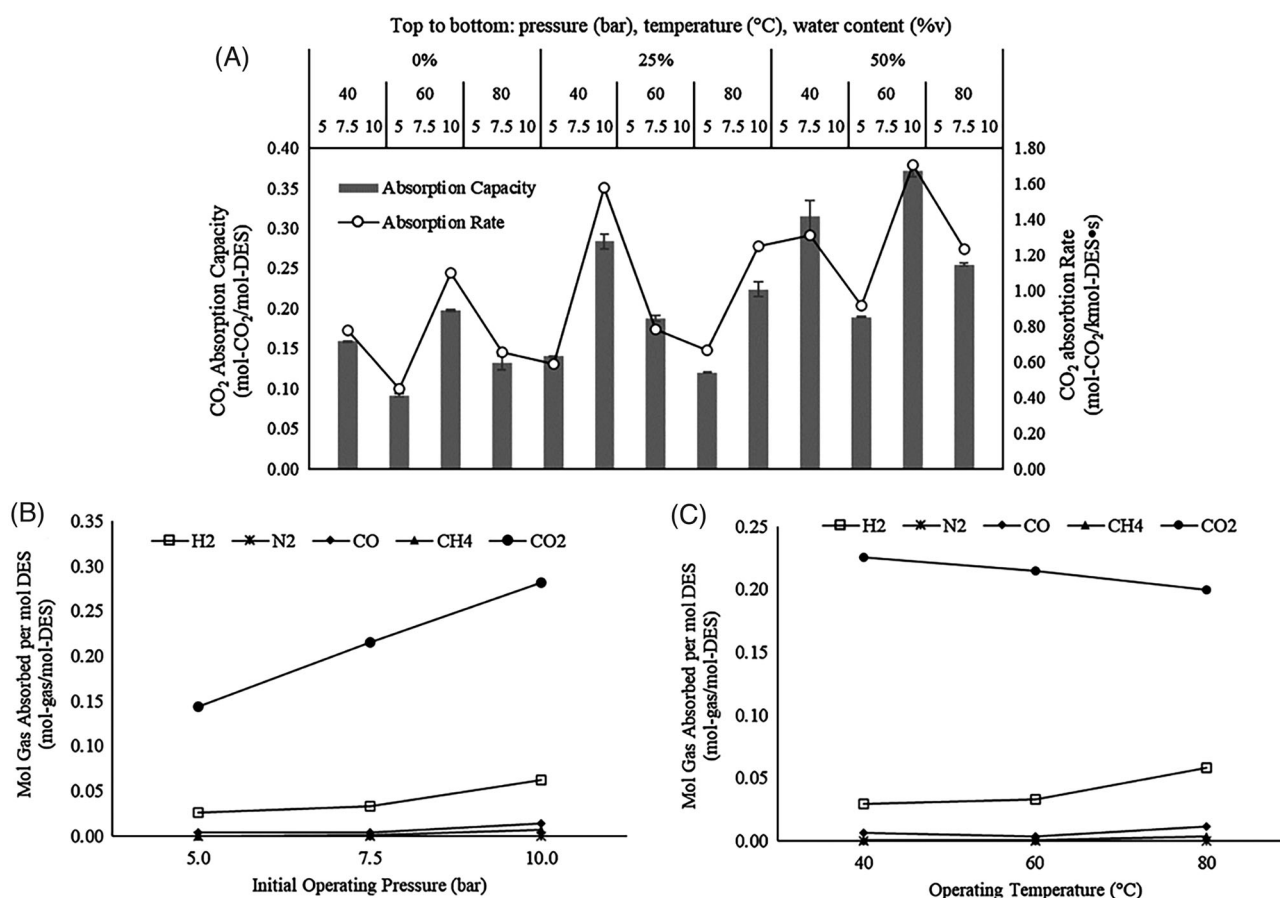


Figure 4. CO₂ absorption performance of choline chloride-monoethanol amine deep eutectic solvent (ChCl-MEA DES) in moderate operating conditions: (A) graphical illustration of the experimental data. Influence of initial pressure (B) and initial temperature (C) on gas absorption by 1 mol of DES.⁹⁷

temperature regimes, namely 30% MEA with artificial HSSs; 30% MEA with acetate, sulfate, and formate; and 30% MEA with real HSSs (obtained from a pilot plant).⁹⁸ At specific temperatures and CO₂ loadings, the CO₂ partial pressures of all the solutions were quite similar. From their investigation, the solution from the pilot plant was comparatively slower in achieving equilibrium, and this was attributed to its advanced degradation especially because the solution with artificial HSS represented the ideal solution with representative conditions and processes. Independent of the influence of other constituents in degraded alkanolamine solutions, Isokai et al. examined the impact of HSS on solution properties and process performances of PCC. As opposed to the general notion of the damages caused by HSSs, they sought to unravel the benefits of the potential presence of HSSs on CO₂ absorption and desorption.⁹⁹ They proved that beside their direct influence on solution properties,

HSSs modify operational ranges of CO₂ loading, which in turn modifies the properties of the absorbing solutions. Generally, these direct and indirect influences resultantly neutralize each other. Accordingly, the properties of the absorbing solutions are bound to maintain stability even in the event of producing large quantities of HSSs. Overall, their study illustrated that there may be some extent of acceptable accumulation levels of HSSs. This supports the earlier reports by Nakagaki et al. on the CO₂ absorption rate of an MEA solution containing carboxylic acids where they suggested that the detrimental effect of the presence of HSSs can be partly impeded if the CO₂ absorption rate of an absorbing solution is maintained at a high value within richer CO₂ loadings.¹⁰⁰ In their work, Weiland & Sivasubramanian also highlighted the beneficial presence of HSS to the operation of the stripper section alongside their damaging effects to the absorber operation.¹⁰¹

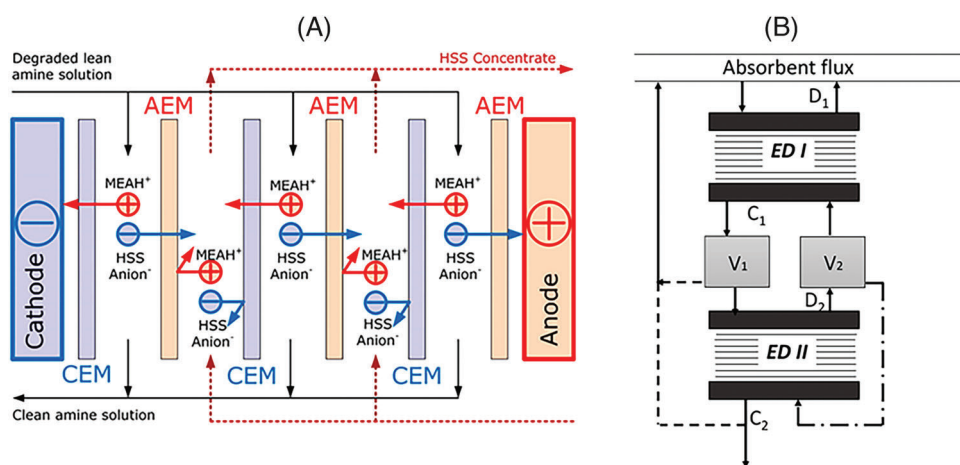


Figure 5. A simple illustration for HSS removal from MEA using ED (where AEM and CEM represent anion-exchange membrane and cation-exchange membrane, respectively).¹⁰⁵ (b) Flow pattern for two-step ED system. First-step dilute (D_1), first-step concentrate (C_1), D_2 —second stage dilute (D_2), second-step concentrate (C_2), buffer tanks (V_1 and V_2).¹⁰⁵

Reclamation procedures of alkanolamines and the removal of HSSs

The utilization of alkanolamines for CO₂ removal leads to the contamination/accumulation of various HSSs which are not easily regenerated by the application of heat within the normal temperature range of the stripping process. These salts can dramatically undermine the efficiency of the amine scrubbing process. Hence, the need for HSS removal and alkanolamine reclamation.

Electrodialysis

Electrodialysis (ED) is one of the methods for the removal of HSSs from spent alkanolamine solution. ED being a separation process employs charged membranes and electrical potential difference in the separation of ionic species from aqueous solutions and other uncharged components.^{102–104} Driven by electricity, they involve alternate arrangements of cationic and anionic ion-exchange membranes in direct current (DC) fields, and ionic salts are pushed through the membranes from less concentrated solution regions employing the influence of electrical potentials as illustrated in Fig. 5(A) and described by Grushevenko et al.¹⁰⁵ Utilizing this technology, Chen et al. developed a pilot-scale ED stack and monitored the elimination of HSSs from a spent MDEA solvent.¹⁰⁶ Achieved in the presence of NaOH, the HSSs removal

efficiency was 77% with 5.73% loss of MDEA with a total process cost of about 748.68 \$/(T HSS).

Grushevenko et al. investigated the reclamation of MEA solution with a laboratory-scale two-staged ED unit.¹⁰⁵ The results revealed that a prereduction of CO₂ (using an initial ED treatment) led to significant reductions of both loss of MEA and specific power consumption. According to Grag,¹⁰² compared with thermal treatments, membrane-based systems are cheaper and lower in energy consumption/demand (See Table 3).

Bazhenov and coworkers studied an electrodialysis-aided reclamation of HSSs and MEA in PCC. The pilot ED-testing exhibited HSS anions removal of up to 65% within 1 h of operation.¹⁰⁷ In 2014, Pal and Banat described the use of polymeric hydrogels for the removal of metal ions and HSSs from industrial lean amine solvents in a gas sweetening unit.¹⁰⁸ The study achieved the removal of 74.0% and 24.3% of the total metal ions and organic acid anions, respectively. The adsorption kinetics followed the pseudo-first-order model, and amongst the fitted adsorption models, the Langmuir adsorption isotherm model best predicted the adsorption behavior with an optimum intake capacity of 1.88 $\mu\text{equiv./gm}$ and 7.69 mg/gm for organic acid anions at 23°C.

Aguiar et al. reported the reclamation of MEA using electrodialysis.¹⁰⁹ The study revealed that substantial amounts of HSSs are removed with high rate of MEA recovery. Although the method is very effective, lower current densities in particularly lower salt

Table 3. Removal of HSS using membrane technology.

S/N	Title	Results	Removal rate	Energy consumption	Ref.
1	Removal of heat stable salts from aqueous solutions of N-methyldiethanolamine using a specially designed three-compartment configuration electro dialyzer	With optimized applied voltage and flow rate, HSSs were successfully removed by adjusting the arrangements of cation- and anion-exchange membranes	90% HSS	0.31 W h/L	111
2	Nanofiltration for the concentration of heat stable salts prior to MEA reclamation	Employed nanofiltration to concentrate HSSs within MEA solution prior to solvent reclamation	80% HSS		112
3	Effect of carbon dioxide loading on removal of heat stable salts from amine solvent by electrodialysis	Decreased CO ₂ content resulting in substantial decline of both solvent loss and energy requirement	Dependent on CO ₂ loading and treatment time, but the rates were generally above 85%	37 kJ/g HSS	105
4	Removal of HSSs from lean amine of a gas refinery via electrodialysis	Enhanced HSS elimination and reduced energy requirement obtained from a homogeneous membrane in comparison to a heterogeneous membrane	42% HSS	8.14 kWh/g HSS	113
5	Removal of heat stable salts from N-methyldiethanolamine wastewater by anion exchange resin coupled three-compartment electrodialysis	Decreased CO ₂ content resulting in substantial decline of both solvent loss and energy requirement	94% HSS	0.0717 kW·h/L	114
6	Two-step electrodialysis treatment of monoethanolamine to remove heat stable salts	Compared with a one-step electrodialysis treatment, the two-step treatment afforded 50% MEA recovery at the same efficiency of the removal of HSSs.	76%–97% for the range of HSSs studied		110

concentrations should be applied to reduce water splitting. The investigation of the membrane stability in a highly alkaline solvent showed that the studied membrane maintained stability after being exposed to 30 wt % MEA for about 4.5 months.

Grushevenko et al. adopted a two-step ED treatment to remove HSS from MEA (Fig. 5B).^{105,110} This two-step method by ED permitted reduced MEA loss and decreased the CO₂ residual concentration in the absorbent solution. Compared with a one-step ED treatment, this two-step method afforded MEA

recovery of about 50%, including heightened efficiency of the removal of HSSs.

Distillation

Distillation procedure is an important option for removing HSSs.^{11,115} Hence, this segment will highlight the adoption of distillation process for the reclamation of alkanolamines and the removal of HSSs. The distillation procedure involves a solution which is alkalized to give way to an enclosed amine, filtered to

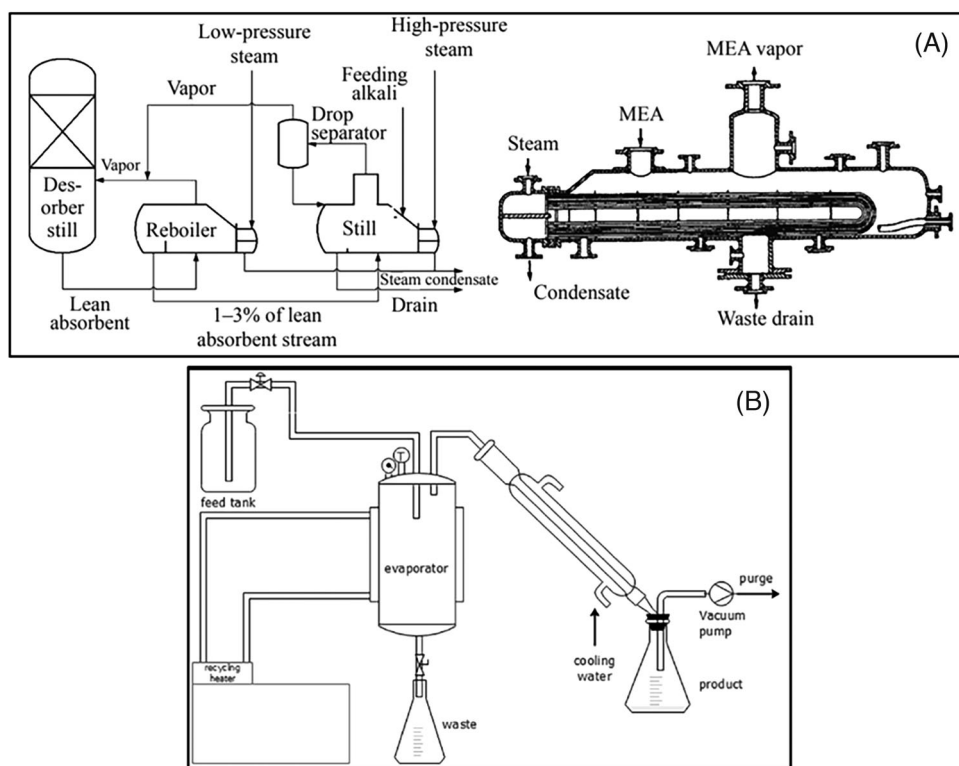


Figure 6. (A) A typical distillation process and the schematic illustration of a distillation still.¹¹ (B) Thermal distillation flow process.¹¹⁷

distinguish the solid and crystallized components, then added to a unique reclaiming chamber for the distillation process to take place. The produced amine with water is removed as vapors, while the less-volatile remaining products and HSSs are often settled within the bottoms.¹¹⁶

In most cases, the distillation media relies on the alkanolamine type as they influence the resistance to thermal degradation. Unlike MDEA that is unlikely to degrade at temperatures around 182 °C, MEA and DEA are often respectively stable under heating around 150 and 175 °C. Notwithstanding, under atmospheric pressure, the boiling points of most alkanolamines are higher than the temperatures at which they thermally degrade. Thus, distillation is expected to be carried out within a vacuum as shown in Fig. 6(A).

For vacuum distillation, the secondary reclamation of MDEA and DEA alkanolamines is a complex procedure with strict implementation for specific kinds of alkanolamines; however, this remains central to HSS separation processes. A related study by Golubeva et al. also presented the vacuum distillation process of leftover amine within a vacuum, as required due to the increase in MEA and DEA boiling points.¹¹⁸ In view of

this, alkanolamine solvent loss and decrease in heat steam are drastically reduced to an extent. With vacuum distillation, there is high possibility of removing the HSSs, volatile impurities and other molecular-weight compounds, that differs from the derived amine compound adopted.

Liu et al. carried out research on a reactive distillation mechanism pertaining to the production of some ethanolamine from ethylene oxide and ammonia via water.¹¹⁹ The theoretical method adopting the Aspen Plus software showed energy consumption and selectivity function of the distillation process. It was concluded that the use of distillation process was effective in the reclamation process of ethanolamine.

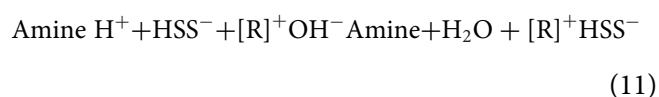
A study by Asad et al. showed a freshwater production mechanism adopting a mathematical model to monitor the membrane distillation process.¹²⁰ The research presented that the membrane distillation process is a non-isothermal separation method. This implies that the mass transfer and heat mechanism happen concurrently within a membrane distillation setup.

Furthermore, the impact of thermosiphon via vacuum distillation of amine media was examined for the amine solutions reclamation operation.¹¹⁷ The

analysis of the feed material, the reclaimed solvents, and the settled streams indicated that the novel reclamation operation incorporated with thermosiphon removed many of the unwanted materials. Thus, the thermosiphon was noted to effectively increase the recovery potential of degenerated alkanolamine reclamation via a steady-state. For higher recovery efficiency, the vacuum distillation temperatures were elevated to a higher value within the amine boiling point, through the feeding mode of operation periodically in equal time to elevate the evaporation process of amine at unstable state. The unit for the entire process was added in a recycling cooling component, with the capability of condensing all the available vapors as presented in Fig. 6(B).

Ion exchange

Unlike newer methods such as distillation and electrodialysis, traditional methods of contaminants treatments like the ion-exchange techniques, are economical approaches for HSS reclamation from amine solutions with lower energy requirements, and they offer operational competence with regards to cost. An ion-exchange methodology involves reversible ionic exchanges between solid resins (ion exchange materials) and liquid phases (in this case, the amine absorbing solutions) without causing significant changes in the resins structures.¹²¹ This method has been in practice for contaminant treatments since the 1950s. Adopting the same principle with strong acid-strong base neutralization reactions, this methodology involves exchanging ions between solids and liquids, such as the ion exchanges between HSS anions (found in amine absorbing solutions) with strong base solutions like KOH and NaOH (from the solid resins). The process basically involves two steps as illustrated in Eqns 11 and 12.^{11,122}



First step: Eqn (11) describes the reaction that occurs when an alkanolamine solution is streamed over an anionic resin bed ($[\text{R}]^+ \text{OH}^-$) to afford the adsorption of HSSs on the resin bed.

Step 2: Eqn (12) describes the regeneration process of the anionic resin bed using NaOH.

The biodegradability of the produced Na salt characterizes the advantages of the ion exchange procedure.¹²² Several studies have been successful in removing different anions from waste water solutions using ion exchange procedures, e.g. chloride,^{123–126} nitrate,^{127–132} sulfate,^{133–135} phosphate,^{136–138} and acetate.^{123,139} Ion exchange procedures present a chemically facile approach for alkanolamine reclamation, and its performance is deemed satisfactory for HSS removal with regards to its less chemical involvement, low energy requirement, and significantly suppressed solution foaming.^{122,140} That notwithstanding, many drawbacks are still associated with the procedure. Earlier procedures were reported either ineffectual or were characterized by the production of enormous amounts of diluted waste which necessitated secondary neutralization prior to disposal.

Despite being unsusceptible to chemical and physical wear, Simister et al. reported that ion exchange resins are prone to thermal degradation at elevated temperature conditions which impacts their reclamation effectiveness.¹⁴¹ Hence, ion exchange procedures are ideal at low temperature conditions (specifically below 45°C). This informs their strategic location around the amine cooler unit. Ion exchange procedures are driven in semicontinuous, continuous or batch modes, but scientists have favored their operations under the continuous mode because it ensures the possibilities of anion treatments at their points of formation, hence instantaneously averting operational challenges. Bayati et al. described a typical continuous ion exchange system (as illustrated in Fig. 7) where lean alkanolamine solutions are sent into the system from the bottom section of the resin column before going through a packed bed and exiting from the top.¹⁰³

In their reclamation testing applied in a PCC pilot plant operation, Moser et al. investigated the potency of ion exchange separation for the reclamation of a degrading absorbing solution composed of an aqueous solution of 3 M AMP and 1.5 M PZ at the lignite-fired power plant in Niederaussem after 40 months' continuous operation.¹⁴² They reported that although ion exchange was effective in removing both anions and cations from the absorbing solution, but the effects on the degradation rate of the solution require careful examination. For enhanced prediction of the degradation rate, the authors suggest that the solution matrix and accumulated contaminants should be taken

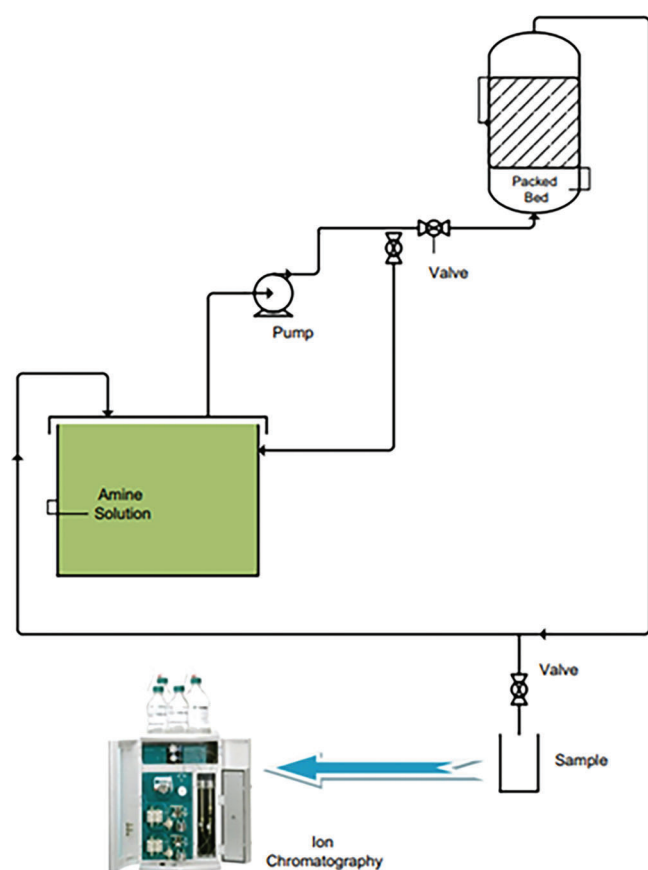


Figure 7. A typical continuous ion exchange system.¹⁰³

into account. Hence, results from lab-based investigations, investigations without consideration for representative industrial flue gas compositions, and short-term tests of MEA solution should not be adopted for other absorbing solutions and industrial application as the ideal expectation. This work is an extension of their previous study.^{143,144}

Electromagnetic separation

The electromagnetic mechanical salt removal (EMSR) procedure is established on the use of electrical and magnetic forces between HSS particles within alkanolamine systems. A unique electromagnetic (EM) setup for HSS elimination from industrial lean alkanolamine was developed by Zain et al.¹⁴⁵ Figure 8 presents a schematic diagram of the rectangular EMSR setup and a picture of the rectangle flow cell with aluminum electrodes shown. The system separates materials using electrical and magnetic forces. The set up helps remove HSS from MDEA without degrading the utilized alkanolamine or additional waste production. To simulate HSS ions, total organic acid

(TOA) was used as a model contaminant. By using EM forces via the Lorentz principle and a rectangular configuration with parallel electrodes, TOA was successfully eradicated. The direction of the electric field was perpendicular to the path of flow down the channel to improve the separation of ions at the outlet. The authors considered the effects of flow rate, voltage, and magnet position on the abstraction efficiency, and discovered that the best operating conditions occurred at 2 mL/min flow rate, 3 V applied potential, and parallel-positioned 1T magnets to fluid flow. According to the investigation, polarization took place when only an electrical field was applied, whereas electrosorption of ions onto the aluminum electrode happened when EM forces were introduced. Moreover, in the parallel flow cell, the removal rates of HSS without and with magnets were 3.2 % and 7.8 %, respectively.

Moreover, The EMSR process is operated under various operating situations, and its effectiveness has been investigated numerically.¹⁴⁶ The Navier-Stokes equations are solved using an Eulerian approach, and the HSS movement is solved using a Lagrangian approach, according to the mathematical methodology utilized for EMSR modeling. Using the developed model, the significant factors that influence the separation process are explored. The study's findings indicate that raising the temperature of the MDEA solution improves the efficiency of the EMSR process. It was discovered that the removal of the HSS is improved by the drop in MDEA concentration. The study discovered that by employing the EMSR, almost 70% of the HSS could be removed.

Hai and coworkers designed an easy-to-use EMSR procedure for the extraction of HSS from industrial lean amine solution.¹⁴⁸ Instead of using electrosorption, HSS were separated in this procedure using an electro-polarization mechanism. Aluminum conduit and rod were constructed into a lab-scale concentric flow configuration for this purpose, serving as the anode and cathode, respectively. Investigations were made into the impacts of the process' operating parameters, such as flow rate, applied voltage, residence time, and pH. The highest HSS removal percentage of 15 % was attained under ideal working circumstances in a single pass with a power consumption of 0.003 Wh/L.

Moreover, two types of electrochemical cells, namely indirect and direct contact cells, were devised and put to the test to investigate the separation of HSS from lean amine.¹⁴⁷ Figure 7(C) gives a schematic

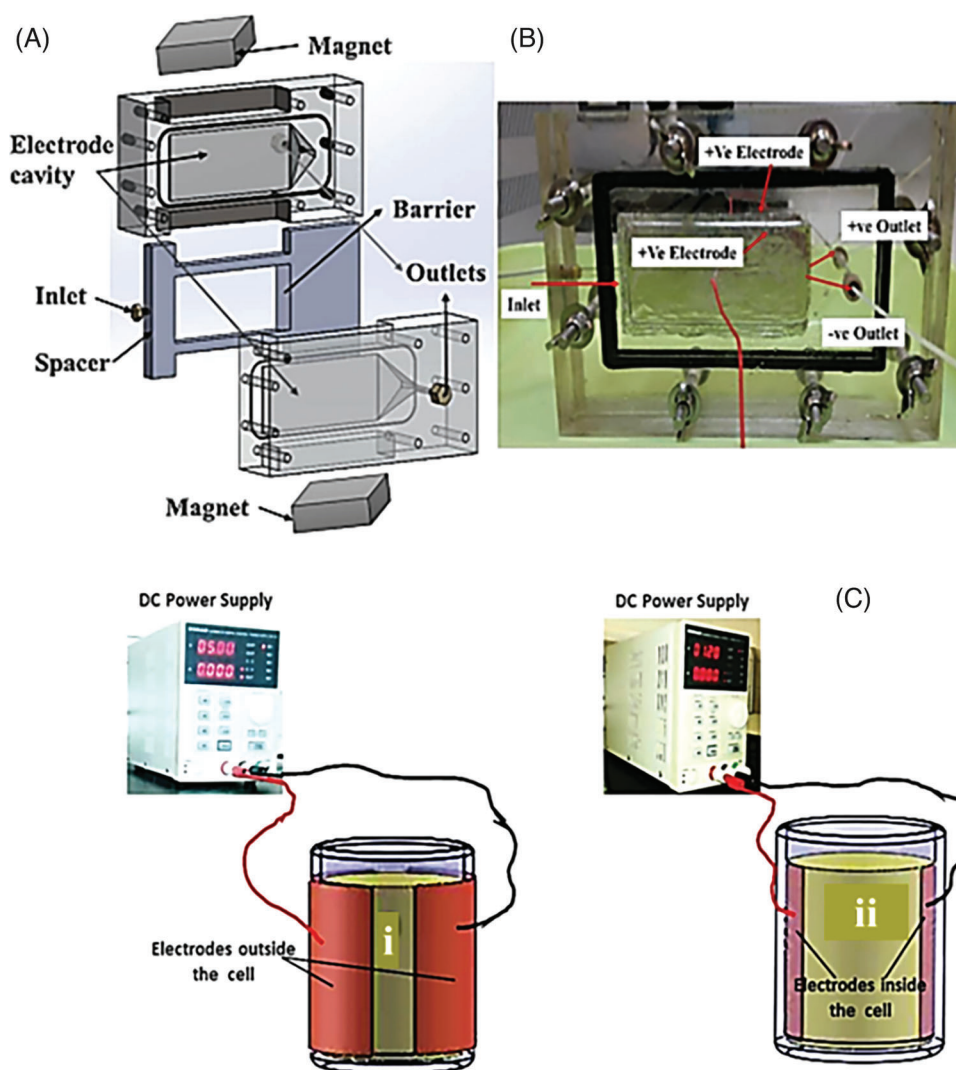


Figure 8. (A) Schematic illustration of the rectangular setup for EMSR; (B) aluminum electrodes are visible in the rectangular flow cell's image¹⁴⁵; and (C) Schematic diagrams for electrochemical cells: (i) indirect contact and (ii) direct contact.¹⁴⁷

representation of the electrochemical cells. The indirect electrochemical cell is made of a 50 mL acrylic pipe with an internal diameter of 1 inch and a thickness of 2 mm. A local gas treatment company in Abu Dhabi provided the HSS concentrated lean amine, which was deposited in the cell's outer layer between parallel copper sheets that were fastened. Copper wires were used to link the electrodes to a programmable DC power source. For two hours, the cell was run at room temperature with various applied voltages. In the second case (direct contact), a 50 mL PYREX Griffin glass beaker was utilized as the electrochemical cell for the direct contact design, allowing for direct contact between the electrodes and the amine solution. In the

cell, two electrode plates with a combined surface area of 4 cm^2 were positioned parallel to one another. Wire connections were used to connect both plates to the programmable DC power supply. The plate that is connected to the power supply's positive terminal serves as an anode, while the cathode plate is attached to the negative terminal. According to the findings, the configuration of an indirect electrochemical cell has no effect on the removal of HSS, thus all the electrodes were built inside a cell that was in direct contact with the MDEA solution. Surprisingly, the HSS's removal efficiency was significantly influenced by the applied voltage. Nickel foam (Ni) and platinized (Pt) coated titanium (Ti) electrodes were the evaluated materials'

most effective at removing HSS, with removal rates of 6.5% and 9.6% at voltages of 0.8 and 1.2 V, respectively. Additionally disclosed were the nickel and Pt coated Ti electrode's electrochemical properties, such as area capacitance and cyclic voltammetry. The removal effectiveness of the HSS in the batch mode of operation was unaffected by the magnetic forces.

The notable merits of this method include¹⁴⁵: (1) cost-effectiveness since its operation is maintained at lower voltages than electrodialysis in addition to aluminum being an inexpensive electrode material; (2) nongeneration of secondary byproducts compared with ion exchange method; (3) nondegradation of absorbing solvent compared with distillation process (4) nondisruption in separation processes resulting from membrane fouling as experienced with ion exchange and electrodialysis separations.

Solvent extraction

A more recent methodology involving the use of hydrophobic organic solvents for the extraction of HSS and alkanolamine reclamation is gaining traction in CCS study.^{149,150} This methodology is known as solvent extraction or liquid-liquid extraction. It allows the extraction of HSSs even at very low concentrations and they are achieved with reduced energy utilization.¹⁵¹ This is an improvement to the excessive energy demand and coevaporation problems associated with distillation, as well as the specificity for only the separation of ionic species associated with ion exchange technique. The principle of solvent extraction has been popularized in several industrial applications like wastewater treatment, biochemistry and biotechnology, biofuel treatment, pharmaceutical purification, and environment remediation.^{152–154} Using quaternary amine (tri-*n*-octylamine and aliquat 336) in 1-octanol diluent for the separation of HSSs resulting from the degradation of MEA, Akkarachalanont and coworkers are credited with the pioneer work of solvent extraction in CCS application.¹⁵⁵ They reported decreased extraction efficiency at elevated CO₂ loading and the process was not influenced by temperature. As an extension of the study, Karnwiboon et al. substituted the straight-chained 1-octanol dilutant with a branched-chain 2-ethyl-1-hexanol and this sufficiently increased the extraction potency of the solvent.¹⁴⁹ The solvent was also applied in the reclamation of AMP and MDEA, and the extraction efficiency was comparatively better with MDEA with nearly 100%

oxalate removal. Kostyanayaa et al. designed a new method of combining liquid-liquid membranes with organic solvents for HSS removal.¹⁵⁶ The method achieved selective HSS anion extraction by avoiding the direct mixture of liquid phases. Polypropylene, polysulfone, and polyvinylidene fluoride were the tested membranes, while the extractant contained 1-octanol and aliquat 336. Of all the membranes, polyvinylidene fluoride displayed the best extraction capacity with the removal of about 50% formic acid from a lean MEA solution.

Other methods

In a study by Edathil et al.,¹⁵⁷ magnetic calcium alginate hydrogel composites were employed to establish a method for removing HSS from amine solution. Magnetic calcium alginate hydrogel composites were fabricated by impregnating freshly made maghemite nanoparticles (γ -Fe₂O₃) using the traditional precipitation approach in calcium alginate beads. Using X-ray diffraction (XRD), scanning electron microscopy (SEM), and Fourier transform infrared spectroscopy (FTIR) to characterize the fabricated magnetic hydrogel composites (CA-Fe₂O₃), it was revealed that γ -Fe₂O₃ is kept uniformly in the matrix of the composites. Optimum HSS removal of 29.2% was attained under 240 min with 3 g of CA-Fe₂O₃ composites at 25°C, and the effectiveness of adsorption increased with increasing temperature, according to kinetic and equilibrium batch adsorption studies. Three kinetic models were employed for adsorption kinetics investigations, and the experimental data and the pseudo-second order kinetics showed that adsorption exhibiting both chemisorption and endothermic nature. Even after eight cycles, the reusability experiments revealed no appreciable decline in absorption capacity. Additionally, the magnetic composites' superparamagnetic properties made it simple to separate them from lean MDEA by utilizing an outside magnet. As a result, these magnetic alginate-Fe₂O₃ hydrogel composites can function as a simple and affordable adsorbent for the recovery of industrial lean MDEA, with the added benefits of high effectiveness, environmental friendliness, and ease of fabrication and recovery.

Diglycolamine (DGA), an alkanolamine used in natural gas sweetening unit, degrades into TOA anions, which negatively affects the operational units. In an investigation, TOA anions from industrial lean DGA

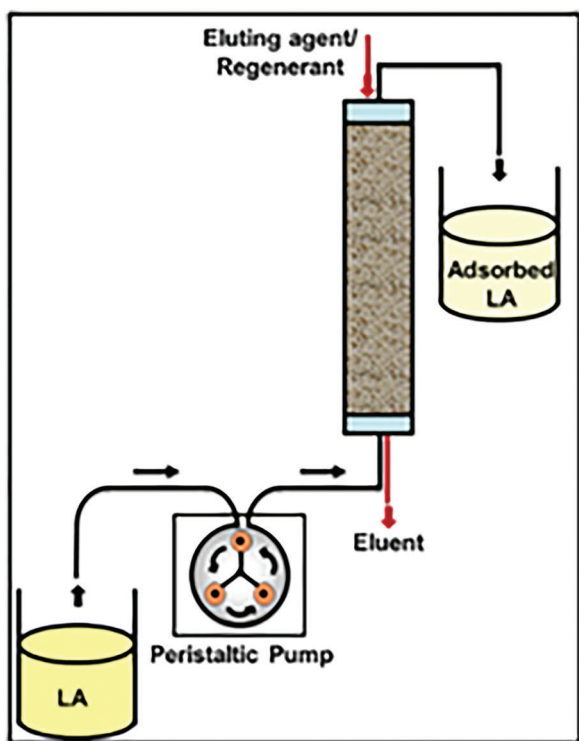


Figure 9. Diagrammatic representation of the up-flow action of the fixed-bed adsorption system.¹⁶¹

solvent were eliminated using calcium alginate carbon (CAC) adsorbents.¹⁵⁸ HSSs can hinder MEA solution's ability to absorb.²² Therefore, in a work by Ju and coworkers, a facile thermal reclaimer method was employed, and the amount of HSSs in the reclaimed MEA product was decreased to roughly 0.61 wt %.¹⁵⁹

The technological viability of employing a calcium alginate/clay hybrid (CAH) composite adsorbent in a continuous fixed bed adsorber to remove HSS like TOA from industrial lean MDEA solutions has been explored.¹⁶⁰ In Fig. 9, the fixed-bed adsorption arrangement is shown schematically in the up-flow operation mode. The goal of the adsorption system was to treat the industrial lean MDEA solution and deliver clean MDEA (without TOA) to the column's exit. The system was built such that the same column could be used for both adsorption and regeneration. A known quantity of adsorbent was used to fill the column, and a peristaltic pump was used to pump MDEA solution with a known concentration of TOA through the column at the specified flow rate in either an upflow or down-flow mode. The column was manually loaded with lean MDEA solution and then operated in down-flow mode to prevent channeling or problems

with viscous fingering. At predetermined intervals, treated MDEA effluent samples were taken from the column outlet, and the quantities of TOA ions in the effluent were determined using a UV-vis spectrophotometer. Once the concentration of TOA ions in the effluent samples equaled the influent concentration, the column's operation was terminated. Analysis was done on the impact of process variables, such as flow direction, feed flow rate, bed height, and bed diameter, on the fixed-bed sorption system's adsorption capacity, breakthrough time, and exhaustion time. The uptake capacity and breakthrough time were shown to increase with bigger bed diameter and height, lower flow rate, and up-flow column mode, according to experimental data. The adsorbent's viability was validated by a gradual decline in adsorption capacity after three cycles of adsorption and desorption. Additionally, foaming investigations supported the crucial function of CAH composite in lowering the foaming ability of lean MDEA solvents due to the synergy of consecutive biosorption and the durability of the saturated composites to many in-situ regeneration using CaCl_2 .

Table 4 compares the most applied reclamation methods with an aim to provide quick and informative details of their principles of operation, energy requirements, etc.

Reclaimer waste management

The choice of reclamation methodology is typically dependent on both process cost and available waste management procedures. This means that, just like every other environmental waste, the waste management procedure for effluents from reclamation units is governed by waste classifications against standard regulations,²⁰ and according to the European Union directive, waste classification is entirely the responsibility of the waste producer.¹⁶² In addition to illustrating the merits and demerits of some existent commercial reclamation methodologies, Table 4 also classifies the type of waste generated from these procedures.¹²²

As displayed in Fig. 10, Sexton et al. presented different available options for reclaimer waste disposal based on their classifications as hazardous or non-hazardous wastes.²⁰ Some of the highlighted disposal methods like landfilling^{163–165} and cement kiln incineration,^{166–170} have been previously popularized for other applications. Moreover, some studies have

Table 4. Comparative assessment of the commercialized alkanolamine reclamation methodologies.

Parameters	Ion exchange	Distillation	Electrodialysis
Applicability	Elimination of ionic contaminations	Elimination of nonvolatile constituents and solids	Elimination of ionic contaminations
Principle of operation	Ionic capture using ion exchange resins	Volatilization of constituents	Elimination of ions by electrodialysis
HSS removal capacity	90%	100%	92%
Metal/Nonionic product removal	0%	100%	0%
Total alkanolamine recovery	About 99%	About 85–95%	About 98%
Equipment required	Neutralization and filtration (one micron prefilter) required upstream of package ion exchange unit	Gas-fired heater, cross exchanger, inlet separator, vapor scrubber, overhead accumulator, vacuum pump, reflux condenser, cooler and carbon filter	Filtration, feed pump, membrane unit
Reagents used	Sodium hydroxide and sulfuric acid for regeneration of resin	Stoichiometric sodium hydroxide	Stoichiometric sodium hydroxide
Type of waste	Aqueous	Nonaqueous and hazardous	Aqueous and nonhazardous
Waste volume	High (about 40%–50%)	Low (5%–15%)	Moderate
Energy required	Low	High	Moderate
Capital cost per kg/h of HSS	\$101,000	\$76,000	\$70,000
Merits	Suitable for low salt concentrations; low energy demand	High waste concentration	Proficient for charged constituents; not influenced by concentration in feed; comparatively low chemical usage
Demerits	Not suitable for non-ionic constituents; elevated salt concentrations initiate recurrent bed exhaustion	Expensive; comparatively lower efficiency; energy demanding; need for vacuum	Not suitable for nonionic constituents; membrane choice and durability

reported beneficial applications of reclaimer waste.¹⁷¹ Landfills constitute remodeled semi-natural terrestrial ecosystems on lands degraded by waste dumping. Landfill locations may be accumulated with waste materials up to around 30 m thickness. The materials are exposed to natural decomposition processes after disposal, and when maintained efficiently, the location is revived for new usage via proper capping and revegetation. Depending on the type of distillation reclaimer used, generated wastes from distillation procedures may or may not involve water dilution for sludge removal. It is also important to note that

solidification affects disposal cost. Also of note is the need to follow strict regulations in the choice of landfilling hazardous or non-hazardous wastes.

Based upon the renewal nature of most waste materials, especially municipal solid waste, and with energy considerations, reclaimer wastes are applied in power plants to significantly drive down fuel costs. However, strict regulations are adhered to before considering the co-firing of hazardous waste in power plant furnaces. As indicated in the report by the International Agency Greenhouse Gas R&D Programme (IEAGHG),¹⁷² the suitability of wastes for

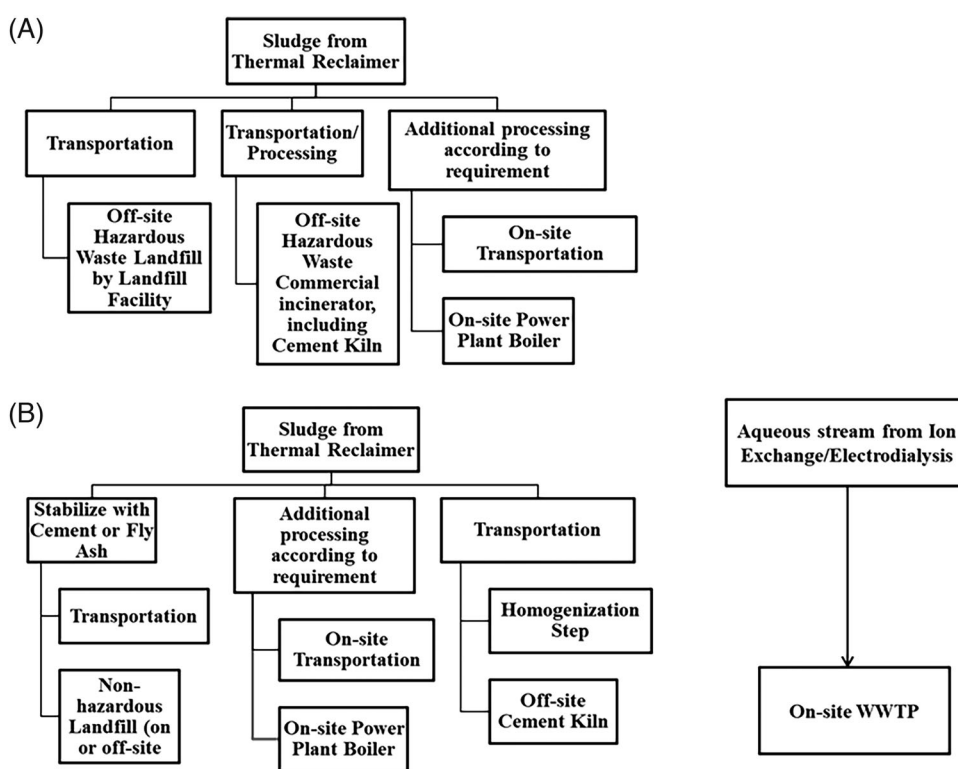


Figure 10. Illustrative demonstration of the disposal options for (A) hazardous and (B) nonhazardous waste from reclamation units.²⁰

firing in a supercritical pulverized-coal power plant is based on factors like moisture content, heating value, and contamination level. In addition to increasing the chloride contents in fuels resulting in the susceptibility of furnaces to corrosion, it is also important to take the composition of power plant flue gases into consideration when co-firing reclaimer wastes as there are possibilities of causing increased metal emissions.¹⁷³

Another application of reclaimer waste is in the cement manufacturing process where a calcium carbonate source, majorly limestone, is pre-calcined at high temperatures (around 900°C) to afford calcium oxide (CaO) and CO₂, and the CaO further clinkerized in a kiln with other raw materials at temperatures around 1400–1500°C to produce the cement clinker. Cement is eventually produced when clinker is ground in cement mills in the presence of other additives like gypsum and grinding aids. From the above description, cement production expends large quantities of energy, especially the precalcination and clinkerization processes. This gives room for the exploration of alternative fuels to supplement the use of the primary fuel source. Hence, reclaimer wastes have

found application in cement manufacturing, and their suitability for use should follow the recommendations of Khan et al.¹⁷⁴ Kilns have the advantage of handling reclaimer waste irrespective of their classification as hazardous and non-hazardous materials because kiln fuels are exposed to long gas residence times and extremely high temperatures in combustion chambers, hence all organic components are completely broken down during combustion.

Conclusion

The existence of HSSs in CO₂ capture processes using alkanolamines has been a trending topic for some time now. The potential threats of their presence have been exhaustively investigated by many researchers; hence their formation mechanisms and impacts on capture processes and equipment have been well understood. The following are the main deductions and perspectives from this review: Despite being the sole commercialized method for CO₂ capture, postcombustion capture process using alkanolamine is characterized by the formation of HSSs either through direct introduction with the flue gas contents or as a

result oxidative and/or thermal degradation of the employed alkanolamine. Hence, the presence of HSSs has been proven to be almost inevitable. Their manifestation in absorbing solvents are linked with several operational challenges like reductions in the removal capacities of CO₂, decreasing amine pH, solvent conductivity increments, solvent foaming, dissolution of protective layers and initiation of corrosion and erosion. Based on the aforesaid, numerous procedures have been developed to remove HSSs from alkanolamines, and these are aimed towards enhancing the efficacy of post-combustion absorption processes. These include traditional methods for contaminants removal like ion-exchange techniques, and the more contemporary methods like distillation, electrodialysis, electromagnetic separation, and solvent extraction.

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Conflicts of Interest

The authors declare no conflict of interest.

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