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Green Micellar Extraction of Phenol from Wastewater Using Eco-Friendly Hydrophilic Deep Eutectic Solvents

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ABSTRACT

Phenol, an extremely toxic wastewater pollutant, adversely affects animals, plants, and aquatic life. Its accumulation in aquatic and terrestrial ecosystems affects nutrient recycling and their long-term stability. Industrial wastewaters constitute a major source of phenol, and its removal is a major concern of researchers and governments across the world. In the current research work, green extraction of phenol from model industrial wastewater was studied by using Aquoline, a new type of eco-friendly deep eutectic solvent (DES), prepared from choline chloride as an H-bond receiver and water as an H-bond contributor. Separation of phenol from wastewater is facilitated by micelles formed in Aquoline due to the CTAB surfactant. The prepared DES was characterized by UV, FTIR, and NMR spectroscopy. Maximum phenol extraction efficiency of 93.27 % was obtained with Aquoline DES + CTAB under optimized conditions (initial phenol concentration = 5000 ppm, temperature = 301 K, pH =10, CTAB concentration = 0.9 mM.L^{-1}, volume ratio of wastewater to solvent = 10:1). The recovered solvent was reused four times after removal of phenol. FTIR studies on recovered solvent showed no change in its structure. This is the first research to use a hydrophilic deep eutectic solvent in the extraction of phenol from wastewater. The research will pave the way for the use of a new type of eco-friendly solvent while adhering to principles of green chemistry.

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1. INTRODUCTION

Phenol and phenolic pollutants are released into wastewater by numerous industries, for example, petroleum, pharmaceutical, coke oven plants, coal washing facilities, dyes, polymer production, and metal processing, are a serious concern due to their toxicity. While phenol concentrations in petroleum refinery wastewater can be as high as 135 ppm, coke oven plants discharge significantly greater amounts, with levels sometimes surpassing 1,000 ppm. The highest phenol concentration was reported for coal gasification plant wastewater at 5000 ppm (Yang et al. 2006).

The contaminated wastewater leaches into underground aquifers, water bodies, and lands, thus affecting entire ecosystems. Phenol is fatal to animal life as it is carcinogenic and causes mutagenesis. Its presence is detrimental to aquatic ecosystems as it hinders the growth and reproduction of marine organisms. For humans, 10-24 ppm of phenol concentration is considered hazardous, while a concentration of 1500 ppm in the bloodstream is considered lethal. At these concentrations, it can affect the liver, kidneys, skin, and respiratory system, and prolonged exposure may result in cancer. (Hammam et al. 2015, Pradeep et al. 2015, Hamad et al. 2021, Panigrahy et al. 2022).

Environmental Protection Agency (EPA), U.S.A., mandates that a maximum of 0.1 ppm of phenol be allowed in industrial effluents. For drinking water, the WHO allows only 0.001 ppm of phenol. In India, the prescribed limits for

phenolic compounds are: 0.001 ppm in drinking water, 1 ppm in inland surface waters, and 5 ppm in public sewers. These strict effluent disposal norms set up by government authorities have forced industries to seek new methods and implement effective protocols for phenol elimination from wastewater. (Mukherjee et al. 2007, Sadhu et al. 2014, Patel & Desai 2022).

For the removal of phenol, different methods such as adsorption, liquid-liquid extraction, membrane separation, foam fractionation, electrocoagulation, electro-Fenton methods, steam distillation, chemical oxidation, precipitation and biological treatments (enzymatic and microbial) were used by researchers. (Divate & Hinge 2014, Villegas et al. 2016, Othman et al. 2017).

The adsorption method has numerous difficulties, such as low adsorption capacities, pore clogging in the case of clays, surface saturation, longer times needed for reaching equilibrium, limited selectivity in mixed wastewater, high costs, difficulties in regeneration and reusability (Mandal et.al. 2020, Adnan et.al. 2021, Al-Ghouti et al. 2022, Ho 2022). Membrane separation techniques suffer from various problems, including membrane fouling, emulsion instability in the case of liquid membranes, high production costs, and potential degradation under harsh wastewater conditions in the case of polyimide ultrafiltration membranes (Reis et al. 2011, Feng et.al.2021, Ali et al. 2021). Despite obtaining high extraction efficiency, the application of enzymatic processes on an industrial scale may be impractical when handling large volumes of wastewater (Bevilaqua et al. 2002).

Among all methods, liquid-liquid extraction was the most preferred method. The choice of solvent was the major aspect affecting phenol removal in extraction. Alcohols, esters, ethers, and ketones are often the solvents of choice due to their favorable distribution coefficients as well as their comparatively low toxicity and good biodegradability. (Kieezyk & Mackay 1973). Combining these solvents with an inert diluent presents an appealing approach. The following solvents were mentioned in the literature: cumene (Liu et al. 2013), iso-kerosene (Abbassian et al. 2015), methyl iso-butyl ketone (Greminger et al.1982), di-isopropyl ether (Greminger et al.1982), hexanol (Rao et al. 2009), heptanol (Rao et al. 2009), octanol (Rao et al. 2009), castor oil (Rao et al. 2009), toluene (Mohmed et al. 2001), benzene (Abdelmonem et al. 2001), benzyl 2-ethylhexyl sulfoxide(Wang et al. 2015), mesityl oxide (Feng et al. 2017), ethanolamine (Gai et al. 2019), butyl acetate(Prasad et al.1991), biodiesel (Yu et al. 2012), tridodecylamine in benzene (Datta et al. 2014), kerosene (Juang et al. 2010), polyethylene glycol (Chasib 2013), dimethylsulfoxide (Chasib 2013), tetramethylene sulfolone (Chasib 2013), diethylene glycol(Chasib 2013), ethylene glycol (Chasib 2013), methanol (Klen et al. 2011), palm oil (Othman et al. 2017), etc.

Petroleum-derived solvents were preferred due to their strong affinity for phenol and high extraction efficiency. However, these solvents are carcinogenic and inflammable, thus requiring careful handling and disposal. Recovery of phenol and solvent from the extract was time-consuming and costly. Usage of expensive petroleum solvents to remove phenol from wastewater is uneconomical and unsustainable. (Nanda et al. 2021).

Novel solvents such as ionic liquids managed to extract phenol efficiently due to the presence of hydrogen bonding, formation of complexes between cationic species and phenolate ions, and their hydrophobic nature. The structure of ILs, temperature, and pH determined the efficiency. However, high viscosities and the non-biodegradable nature of ingredients may limit their uses. (Deng et al. 2011, Wang et al. 2015, Sas et al. 2020, Skoronski et al. 2020).

Eutectic mixtures of H-bond contributors and H-bond acceptors are termed deep eutectic solvents (DES). The depression in melting and freezing points, compared to that of its individual constituents, is characteristic of DESs. Hydrogen bonding is responsible for the depression in melting point. Due to ease of preparation, low cost, low volatility, high biodegradability, and a tunable nature, these mixtures are considered to be a new generation of green solvents. They are being studied as solvents in "green extraction" (Smith et al. 2014).

Recent work by, Cheng et al. (2022), Rodríguez-Llorente et al. (2020), Cañadas et al. (2021), Sas et al. (2019), Wazeer et al. (2023), and Tan et al. (2025) has demonstrated the effective use of various hydrophobic deep eutectic solvents (HDES), including: menthol + nonanoic acid, C_8OOH + menthol, methyl trioctylammonium chloride + menthol, dodecanoic acid +decanoic acid, and trioctylphosphine oxide + menthol, fenchol+ menthol HDES. (Sas et al. 2019, Rodríguez-Llorente et al. 2020, Cañadas et al. 2021, Cheng et al. 2022, Wazeer et al. 2023, Tan et al. 2025). The costly nature of ingredients negates the advantages offered by HDES and makes scale-up unfeasible.

Aquoline is a new generation of deep eutectic solvent that uses water as the hydrogen-bond acceptor. It shows Newtonian behavior, has a pH near neutral, and is polar. (Triolo et al. 2021, Hirpara et al. 2022)It could be used as a solvent, along with CTAB. Positively charged groups of CTAB (cetyltrimethylammonium bromide), a cationic surfactant, tend to attract negatively charged ions of phenol from the sample due to electrostatic attraction. (Shammala et al.1999, Malkoc et al. 2018). Huang et al. (2012)

postulated that the presence of surfactant micelles improved solubilization of phenol. (Huang et al. 2012). Hirpara et al. (2022) studied the micellization behavior of various surfactants in Aquoline. Critical micelle concentration and aggregation number of CTAB were lower in Aquoline compared to water. Thus, lower amounts of CTAB would be necessary for micelle formation. (Hirpara et al. 2022).

In the presence of excess CTAB beyond the critical micelle concentration (CMC), micellar aggregates are formed that incorporate Aquoline, phenol, and CTAB. On account of having lower densities, these aggregates tend to separate from the aqueous phase, thus facilitating faster isolation. This indicates that CTAB micelles in Aquoline can effectively solubilize phenol from wastewater, thereby demonstrating a novel application of hydrophilic deep eutectic solvents (DES). Moreover, for the extraction of acidic impurities, the use of neutral hydrophilic DES containing CTAB might provide better efficiency.

Only a limited number of studies have been reported about the applications of hydrophilic deep eutectic solvents (DESs) for extracting pollutants from wastewater, largely due to their high propensity for dissociation and difficulty in recovery from aqueous solutions. These DESs were extensively used for drug delivery and as a reaction medium. Hydrogen bonding between ChCl and water in Aquoline DES is much stronger compared with other hydrophilic DES (for example, choline chloride and urea). This property permits the Aquoline DES to retain its structure without dissociating when added to water (Dwamena et al. 2019).

To overcome the drawbacks of existing solvents, this research work focuses on the extraction of phenol from wastewater by using a combination of Aquoline (water + choline chloride) and a cationic surfactant (CTAB). We intend to extend the applications of a surfactant-DES system for the recovery of phenol beyond commonly studied wastewater systems (such as textile, refinery, and coke oven plant wastewater). This study aims to remove phenol from wastewater obtained from coal gasification plants (phenol concentration = 5000 ppm). The extraction of phenol from wastewater using hydrophilic DES aided by surfactant has never been tried before. This is the first research work that explores the application of a cheap DES and surfactant combination for the removal of phenol. Usage of a new solvent system might help in reducing waste and contribute to the sustainability of the process.

2. MATERIALS AND METHODS

2.1. Chemicals

Phenol, choline chloride (98 % pure),4-amino-antipyrine, potassium ferricyanide, ethanol (99 %), cetyltrimethylammonium bromide (> 98%), and dichloromethane (all analytical grade) were purchased from Merck Life Science, Mumbai, India.

2.2. Preparation of Aquoline DES

Choline chloride was vacuum-dried before use. The method described in published research papers was used for the preparation of Aquoline (Triolo et al. 2021, Mangiacapre et al. 2023). The mole ratios of HBC and HBR are mentioned in Table 1. All mixtures were stirred until the complete dissolution of choline chloride crystals, resulting in the formation of a homogeneous and slightly viscous liquid. Aquoline could not be prepared at mole ratios of 1:1 and 1:5.

2.3. Characterization of Aquoline

UV absorption spectra of the selected Aquoline DES were studied on a Shimadzu UV-1280 spectrophotometer. The chemical structure of fresh and recovered Aquoline solvent was studied by FTIR spectroscopy on a Perkin-Elmer spectrophotometer. ^{1}H and ^{13}C NMR analysis of fresh and recovered Aquoline was carried out using an Agilent 500 MHz NMR using DMSO /D_2O as solvent.

2.4. Extraction Procedure

Simulated wastewater solutions of phenol with concentrations ranging from 100 to 5000 ppm were used for experiments. A measured amount of sample solution was taken in a graduated and stoppered test tube, and a predetermined quantity of solid choline chloride was added so that Aquoline solvent formed within the system. The mixtures were stirred on a vortex mixer for 15 minutes. Subsequently, a predetermined quantity of the CTAB surfactant was introduced, and the resulting mixture was permitted to settle for 24 hours. Formation of two layers was observed, with the top layer containing phenol, CTAB, and Aquoline. The bottom aqueous layer was analyzed for the concentration of phenol.

Table 1: Formulation of Aquoline DES.

Name	H-bond receiver (HBR)	H-bond contributor (HBC)	Mole ratio of HBR: HBC
Aquoline- 1	Chloride salt of choline	Water	1:2
Aquoline- 2	Chloride salt of choline	Water	1:3
Aquoline-3	Chloride salt of choline	Water	1:4

Extraction efficiency is determined using the formula:

$$\text{Extraction Efficiency} = \frac{(C_{extract})}{(C_{Total})} \times 100$$

Where $C_{extract}$ represents the concentration of phenol in the extracted layer, and C_{total} reflects the concentration of phenol in the original sample.

This method was used for the selection of an appropriate molar ratio for Aquoline DES for extraction and optimization studies. Every experiment was carried out in triplicate.

2.5. Analysis

The concentration of phenol in the lower layer was found by UV spectrophotometer (Shimadzu - UV-1280) using 4-aminoantipyrine dye. A wavelength of 510 nm was used to measure the absorbance of phenol in the sample (Sas et al. 2020).

3. RESULTS AND ANALYSIS

3.1. Characterization of Solvent

3.1.1. UV Spectra of Aquoline-3 DES

The UV spectra of Aquoline-3 are presented in Fig. 1. The spectra show strong absorption in the 177–186 nm range. Maximum absorbance of 3.82 was obtained at λ_{max} = 185.67. As choline chloride lacks conjugated double bonds or aromatic rings, it does not go through $\pi \rightarrow \pi^*$ or $n \rightarrow \pi^*$ transitions. The maximum absorption below the UV range must arise due to strong $\sigma \rightarrow \sigma^*$ transitions of the O–H bonds in water and the ammonium groups of choline chloride. Absorbance steadily falls from 200 nm to 260 nm. Absorbance stabilizes at very low values (<0.1) beyond a wavelength of 400 nm.

3.1.2. NMR Spectra of Aquoline-3 DES

^1H and ^{13}C NMR spectra for Aquoline-3 are shown in Fig. 2 and Fig. 3. The sharp peak at 3.199 ppm belongs to the nine equivalent methyl protons attached to nitrogen in choline chloride. The triplet at 3.51 ppm belongs to the methylene protons adjacent to oxygen ($-CH_2OH$) in ChCl. It appears as a triplet due to coupling with $–CH_2N^+$. The peak at 3.865 ppm belongs to the methylene protons adjacent to nitrogen ($-CH_2N^+$). A broad singlet peak at 5.67 ppm belongs to the hydroxyl proton (-OH) of choline chloride. It's broad due to exchange with water and is shifted downfield. The signal at 2.573 ppm represents DMSO.

The ^{13}C spectrum shows three main signal groups. The peaks at 53.564, 53.586, and 53.617 ppm (centered at 53.59 ppm) are assigned to the three equivalent methyl carbons attached to nitrogen (CH3)3N+. The peak at 55.476 ppm is assigned to the $-CH_2$-OH carbon. Peaks at 67.221, 67.244, 67.267 ppm (centered at 67.24 ppm) are assigned to the $N-CH_2$- carbon. The cluster of peaks at 39.102–40.103 ppm represents the solvent DMSO.

1H NMR spectra confirm the presence of choline chloride in Aquoline-3. ^{13}C NMR confirms retention of the ChCl structure within the superstructure of the DES. The presence of hydrogen bonding and a chlorine atom may have caused downshifting of peaks in both ^1H and ^{13}C NMR to higher frequencies. (Delso et al. 2019, Shahhosseiniet al. 2024).

3.1.3. FTIR spectra of Aquoline -3

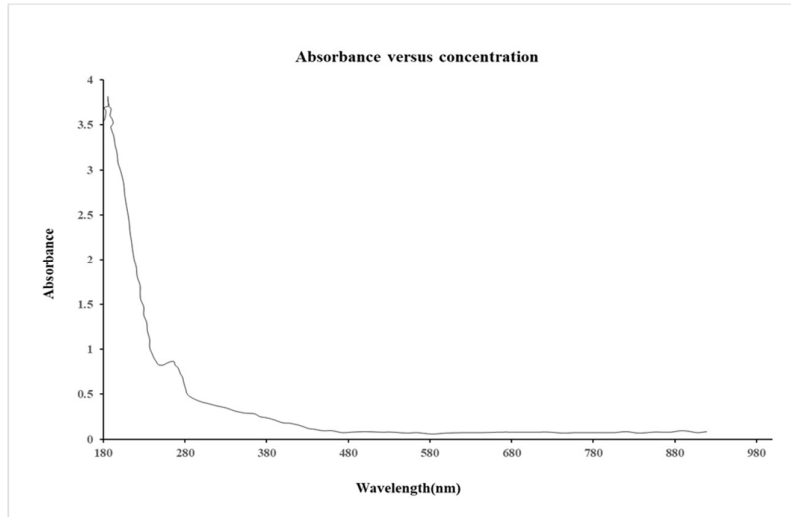


Fig. 1: UV spectrogram of Aquoline-3.

FTIR spectra of ChCl and Aquoline-3 are depicted in Fig. 4. Peaks at 3346, 3018, 2948, 1478, 1007, 952, and 865 cm^{-1} represent stretching of the hydroxyl group, N-H stretching vibration of the quaternary ammonium group, C-H stretching of the methyl and methylene groups, and C-N stretching of the quaternary ammonium group. Peaks at 1133, 1083, and 1050 cm^{-1} show C-O stretching, while peaks at 952 and 865 cm^{-1} show C-N stretching of the quaternary ammonium group. Due to the formation of DES between choline chloride and water, the peaks for ChCl were seen shifted in the spectra of Aquoline-3. (Shahhosseini et al. 2024).

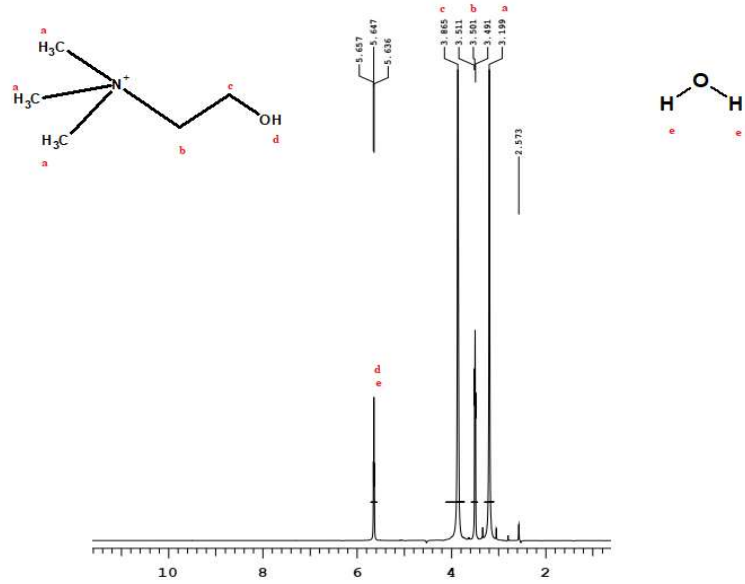


Fig. 2: ^{1}H NMR spectra of Aquoline -3.

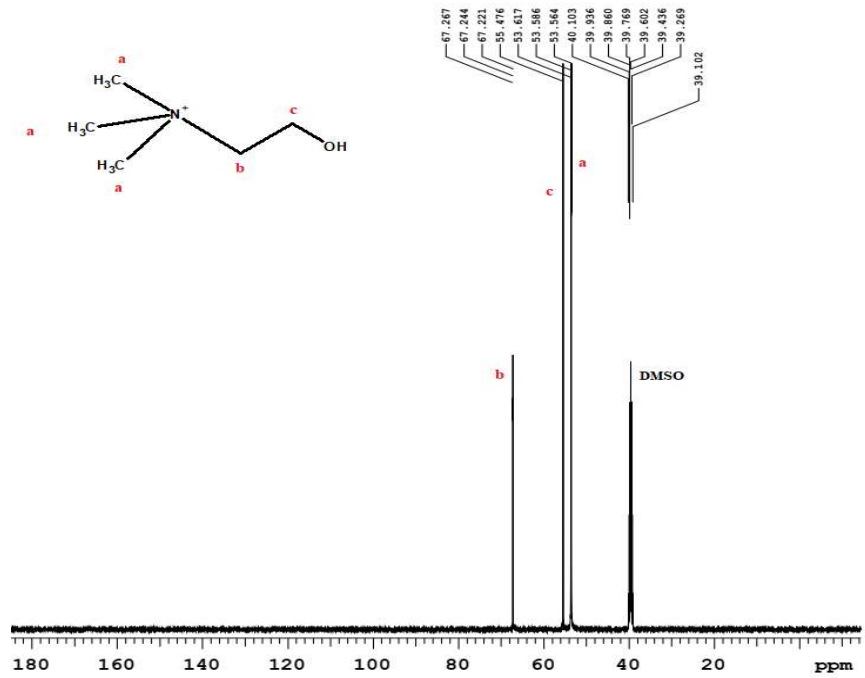
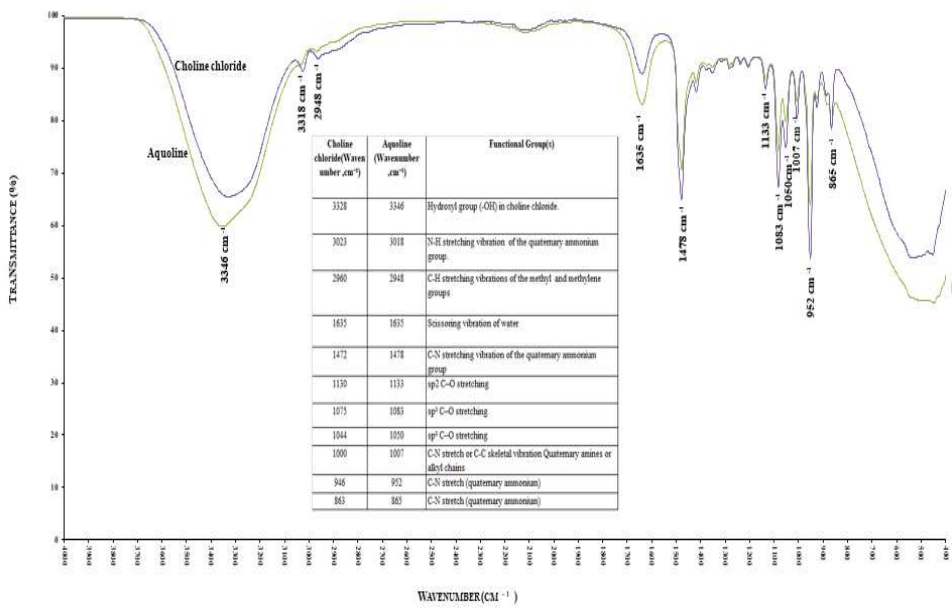


Fig. 3: ^{13}C NMR spectra of Aquoline -3.



Choline chloride(Wavenumber ,cm⁻¹)	Aquoline (Wavenumber ,cm⁻¹)	Functional Group(s)
3328	3346	Hydroxyl group (-OH) in choline chloride.
3023	3018	N-H stretching vibration of the quaternary ammonium group.
2960	2948	C-H stretching vibrations of the methyl and methylene groups
1635	1635	Scissoring vibration of water
1472	1478	C-N stretching vibration of the quaternary ammonium group
1130	1133	sp^2 C–O stretching
1075	1083	sp^2 C–O stretching
1044	1050	sp^2 C–O stretching
1000	1007	C-N stretch or C-C skeletal vibration Quaternary amines or alkyl chains
946	952	C-N stretch (quaternary ammonium)
863	865	C-N stretch (quaternary ammonium)

Fig. 4: FTIR spectra of choline chloride and Aquoline-3.

3.2. Selection of Aquoline with Proper Mole Ratio for Extraction of Phenol

The molar ratio of the HBC to the HBR is strongly correlated with the physical and chemical properties of DESs and affects their selectivity (Tan et al. 2025). The prepared Aquoline DESs (with varying mole ratios of HBC and HBR) were used in extraction experiments, and the results are shown in Fig. 5. Aquoline -3 (mole ratio of HBR to HBC = 1:4) gave the highest extraction efficiency. The positively charged hydrophilic headgroup of CTAB in Aquoline-3 interacts with phenol anions in sample water, causing phenol to concentrate in the upper layer while the lower layer remains free of phenol. Aquoline-3 will be used for the rest of the experiments.

Extraction parameters: Initial phenol concentration = 5000 ppm, temperature = 301 K, pH =10, waste-water solution to DES ratio = 10 :1, CTAB concentration = 0.9 $mM.L^{-1}$.

3.3. Influence of Changing Process Parameters on Extraction Efficiency

To maximize extraction efficiency (with Aquoline -3 as solvent), process parameters (pH, mole ratio of wastewater sample to Aquoline, initial phenol concentration, CTAB concentration) were varied, and their impact was studied.

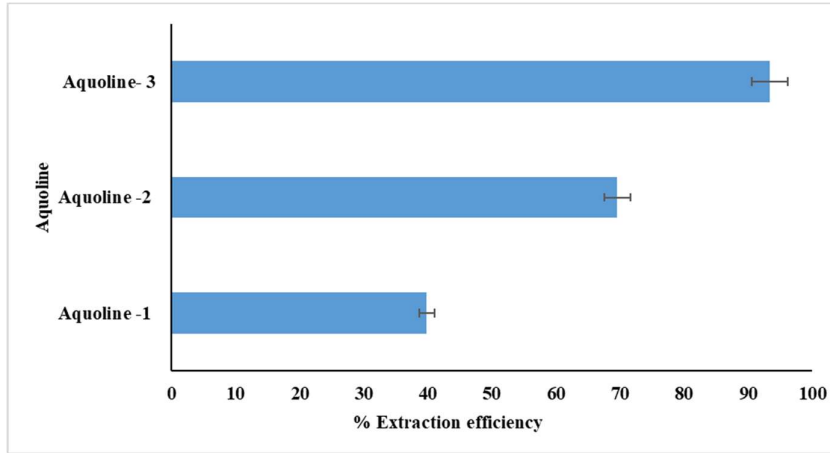


Fig. 5: Selection of Aquoline with the proper mole ratio for extraction of phenol.

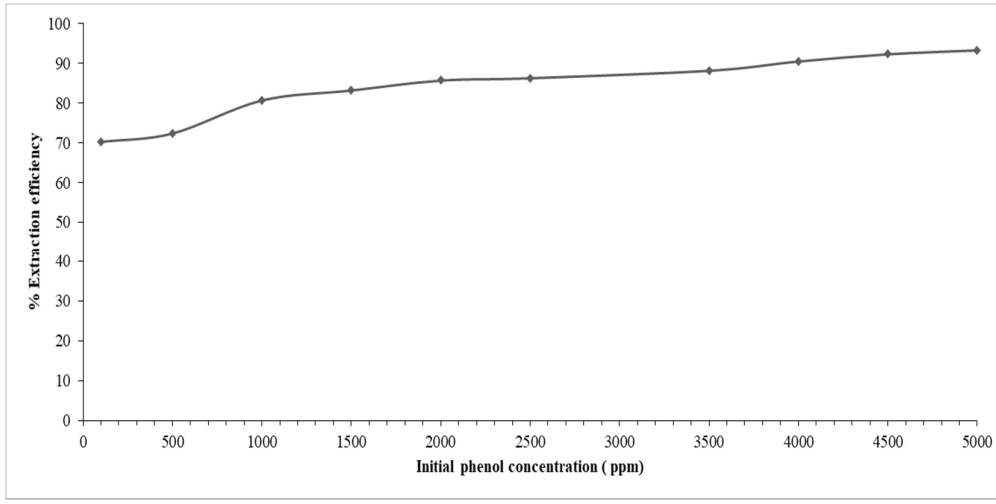


Fig. 6: Influence of changing initial phenol concentration on extraction efficiency.

3.3.1. Influence of Changing Initial Phenol Concentration on Extraction Efficiency

Effects of changing the concentration of phenol in the feed (100 to 5000 ppm) on phenol recovery are depicted in Fig. 6. The extraction efficiency showed a positive trend with subsequent increase in phenol concentration in the feed. At higher concentrations, phenolate ions interact with positively charged CTA^+ ions in micelles, thus increasing solubilization. The micellization of CTAB is further facilitated by alkaline pH and high polarity of Aquoline-3.

Lakshmi et al. (2013) noticed a significant drop in efficiency when phenol concentration was increased beyond 10 ppm with ionic liquid $[Bmim]^+[BF_4]^-$ + tributyl phosphate as solvent (Brinda Lakshmi et al. 2013). Sas et al. (2019) observed that efficiency increased with initial phenol concentration, but solvents with low hydrophobicity showed higher efficiencies (Sas et al. 2020). Tan et al. (2025) reported that initial phenol concentration did not affect extraction efficiency when fenchol-menthol HDES was used as an extractant. This was ascribed to the fact that the degree of

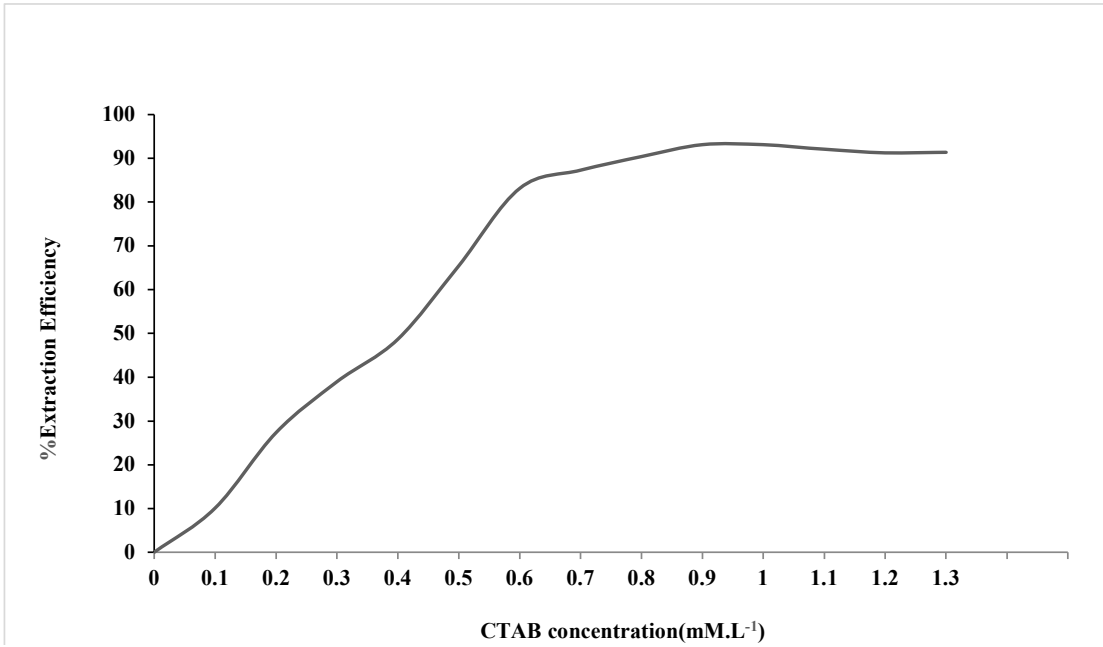


Fig. 7: Influence of changing CTAB concentration on extraction efficiency.

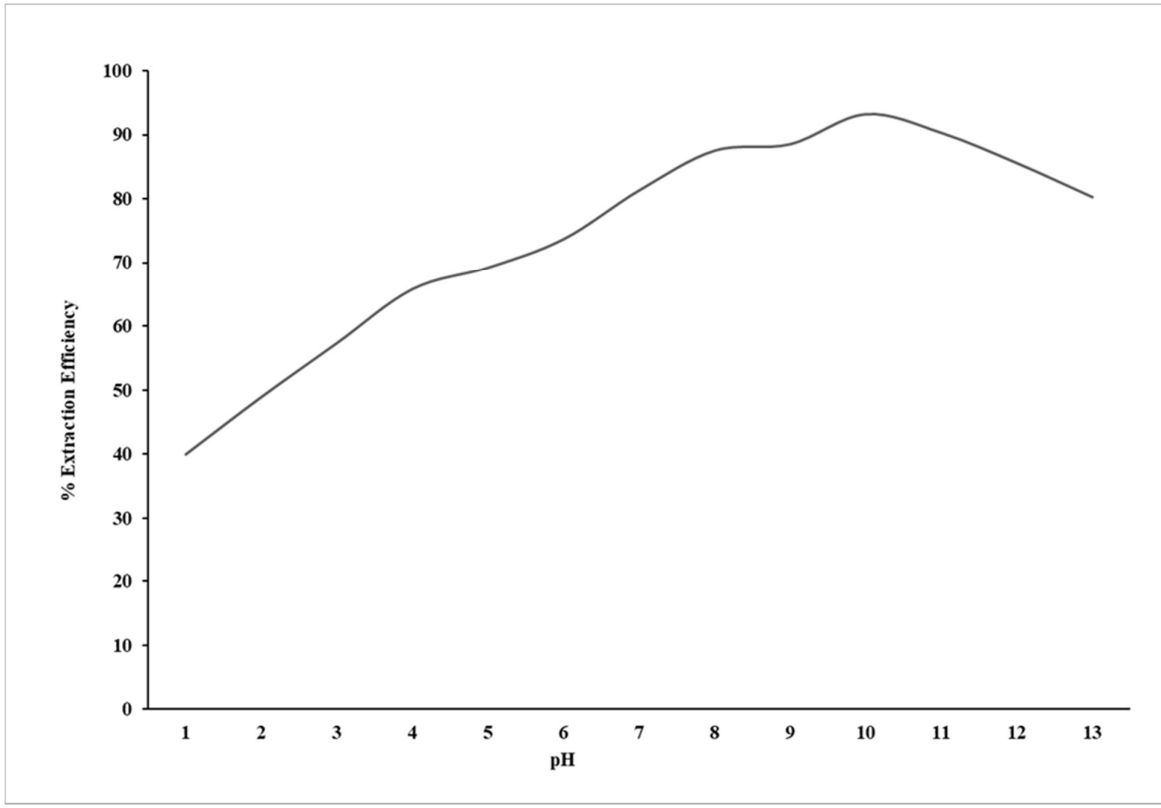


Fig. 8: Influence of changing pH on extraction efficiency.

solvation of phenol was the same at all concentrations (Tan et al. 2025).

Extraction parameters: temperature = 301 K, pH =10, volume ratio of waste-water solution to DES = 10 :1,CTAB concentration = 0.9 mM.L^{-1}.

3.3.2. Influence of Changing CTAB Concentration on Extraction Efficiency

CTAB forms micelles in Aquoline at a CMC value of 0.82 mM.L^{-1}. The micelles formation involves incorporation of the hydrophobic head of the surfactant into the micelle due to the high polarity of DES (Hirpara et al. 2022). When the amount of surfactant in water was higher than its CMC, aggregates of large micelles were formed. Initially, phenol molecules in water tend to attach to the outer layer of micelles. With increasing concentration, phenol molecules penetrate the center of micelles (Chaghi et al. 2008). Phenol serves as a micelle modifier in concentrated CTAB systems. By inserting itself into the surfactant structure, it drives a transformation of the micelles from a standard sphere to rod-like structures, leading to a measurable decrease in the overall solution viscosity (Mata et al. 2006).

CTAB concentration was varied from 0.1 mM.L^{-1} to 1.3 mM.L^{-1}, and results are shown in Fig. 7. At low CTAB concentration, extraction efficiency was low due to negligible solubilization of phenol into micelles. With increasing CTAB concentration, the solution viscosity decreased, and more phenol was solubilized, resulting in the formation of micellar aggregates. Maximum extraction efficiency was obtained at a CTAB concentration of 0.9 mM.L^{-1}.

Extraction parameters: Initial phenol concentration = 5000 ppm, temperature = 301 K, pH =10, volume ratio of wastewater solution to DES = 10 :1.

3.3.3. Influence of Changing pH on Extraction Efficiency

At alkaline pH, phenol dissociates into phenoxide ion, while it stays in molecular form in acidic media due to low pKa (Liu et al. 2013, Tan et al. 2025). Three interaction mechanisms between phenol and CTAB were reported. Hydrophobic interactions at low pH, electrostatic interactions between phenoxide ions and CTAB at high pH, and hydrogen bonding interactions between the phenolic -OH group and water (Shammala et al. 1999).

To facilitate easier extraction of phenol, the pH has to be kept higher to promote electrostatic interactions between CTAB and phenoxide ions. In the acidic pH range, electrostatic interactions between the phenolate ion

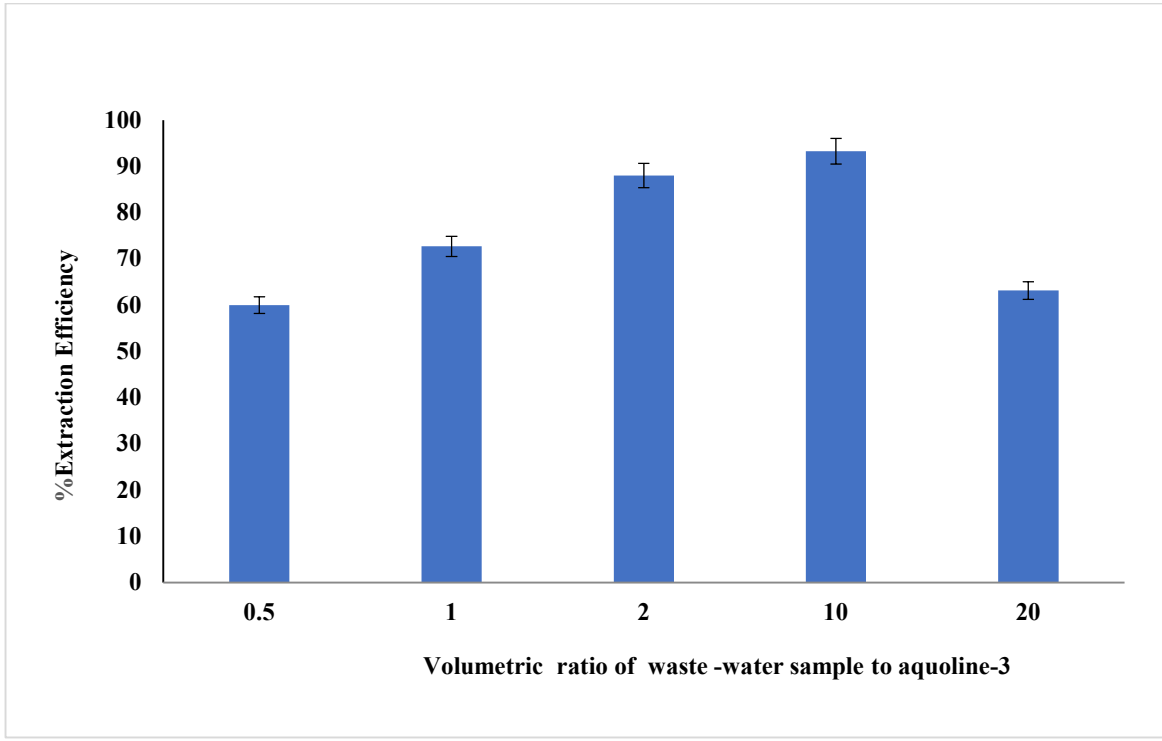


Fig. 9: Influence of changing the volume ratio of wastewater sample to Aquoline-3 on extraction efficiency.

and CTA^+ are negligible, and extraction proceeds due to hydrophobic interactions. At pH = 7, the phenol molecule gets attached to the outer layers of micelles. At alkaline pH, phenolate ions interact with CTA^+ ions, leading to increased solubilization of phenol (Sabatino et al. 2010).

Fig. 8 displays the extraction efficiency as a function of pH. The amount of phenol recovered increased steadily, and the highest efficiency was obtained at pH = 10, but dropped later. So, it is essential to keep the pH in the alkaline range to maximize the extraction of phenol. In published literature, Tan et al. (2025) and Wang et al. (2015) obtained the highest efficiency at neutral pH (Wang et al. 2015, Tan et al. 2025).

Extraction parameters: Initial phenol concentration = 5000 ppm, temperature = 301 K, volume ratio of wastewater solution to DES = 10:1, CTAB concentration = 0.9 $mM.L^{-1}$.

3.3.4. Influence of Changing Volume Ratio of Wastewater Sample to Aquoline on Extraction Efficiency

The volume ratio of wastewater sample to Aquoline-3 was adjusted from 1:1 to 20:1 (Fig. 9). As the treatment ratio increased, the extraction efficiency improved, reaching its maximum at a ratio of 10:1. Beyond this point, efficiency declined due to limited solvent availability. Therefore, a 10:1 volume ratio was chosen. Similar patterns were reported by Lakshmi et al. (2013), who selected a 5:1 treat ratio. Conversely, Tan et al. (2025) achieved maximum extraction at a 1:1 ratio when using HDES as the solvent.

Extraction parameters: Initial phenol concentration = 5000 ppm, temperature = 301 K, pH =10, CTAB concentration = 0.9 $mM.L^{-1}$.

Based on optimization studies, the following optimized parameters were obtained: Initial phenol concentration = 5000 ppm, temperature =301 K, pH =10, CTAB concentration = 0.9 $mM.L^{-1}$, volume ratio of wastewater solution to DES = 10:1.

3.4. Extraction Mechanism

A hypothetical extraction mechanism is proposed for the micelle-assisted extraction of phenol from wastewater using CTAB in Aquoline. The process is primarily governed by interactions between phenoxide ions and CTAB, interactions between the phenolic -OH group and water, and hydrophobic interactions between phenol and CTAB. At high pH, phenol was deprotonated into phenolate ions, which interacted with the positively charged quaternary ammonium head groups of CTAB. Concurrently, hydrophobic interactions between the phenyl ring of phenol and the alkyl chains of CTAB further stabilized this association. Under these conditions, micelles capture phenol molecules in the outer layer. Above the CMC, CTAB formed micelles that encapsulated phenol molecules

within their hydrophobic cores, while the charged head groups remained solvated in the polar Aquoline medium. At higher CTAB concentrations, mixed aggregates containing Aquoline, CTAB, and solubilized phenol were formed. Due to their lower density relative to the bulk aqueous phase, these aggregates separated rapidly, facilitating efficient phase disengagement and enhancing phenol recovery into the DES-rich phase. The presence of CTAB micelles therefore significantly improved phenol solubilization and transfer from the aqueous to the surfactant-rich phase (Shammala et al. 1999, Mata et al. 2006, Chaghi et al. 2008, Liu et al. 2013, Tan et al. 2025).

3.5. The Stripping and Reuse of Extraction Solvent

After extraction, the upper layer (comprising CTAB + phenol + ChCl) was separated by decantation. Phenol, present in this layer, was converted to its salt, i.e., sodium phenoxide, by using sodium hydroxide. The solution was subjected to solvent extraction using dichloromethane for the separation of CTAB. The top organic layer obtained after extraction consisted of CTAB, which was recovered after evaporation of the solvent. The bottom aqueous layer(consisting of phenol) was acidified with hydrochloric acid. Phenol could be separated from this layer by extraction with dichloromethane as solvent. The remaining aqueous portion consisted of choline chloride. It was subjected to evaporation to adjust the ratio of choline chloride and water.

Despite not being considered a green solvent, dichloromethane was used due to its strong affinity for phenol and CTAB, immiscibility with water, and ability to preserve the stability of choline chloride, while requiring only a small amount for DES purification. In future applications, environmentally friendly hydrophobic deep eutectic solvents, such as menthol–nonanoic acid or fenchol–menthol HDES, represent promising alternatives (Cheng et al. 2022, Tan et al. 2025).

The recovered solvent was replenished with fresh solvent and reused for 4 further extractions (Fig. 10). A gradual decline in extraction efficiency was noticed. It may be due to the gradual decomposition of choline chloride during repeated recovery operations. The recycling studies were carried out for only four cycles. Long-term reusability and stability of Aquoline were beyond the ambit of this study.

Extraction parameters: Initial phenol concentration = 5000 ppm, temperature = 301 K, pH =10, CTAB concentration = 0.9 $mM.L^{-1}$, volume ratio of wastewater solution to DES = 10.

Fig. 11 displays FTIR spectra for pure Aquoline, recycled Aquoline-3, and the Aquoline-CTAB upper micelle layer. The peaks at 3342 and 3348 cm-1 indicate hydroxyl groups associated with water. A shift toward higher wavenumbers suggests interaction between micelles and Aquoline. N-H stretching between 3018 and 3021 cm-1 reflects alterations in the CTAB carbon chain due to phenol presence, supporting previous observations of micelle DES interactions. Minor C-H shifts at 2950 cm-1 demonstrate CTAB's involvement in the DES structure. The chemical shifts in the fingerprint region, from 946 to 864 cm-1, indicate C-N stretching, implying that the quaternary ammonium in CTAB has interacted with phenol and DES, confirming the interdependence of CTAB and Aquoline-3 in phenol extraction. Additionally, shifts at 1075 cm-1 in the sp^3 C–O region suggest choline hydroxyl interactions. Peaks between 1004 and 1007 cm-1 show typical vibrations associated with

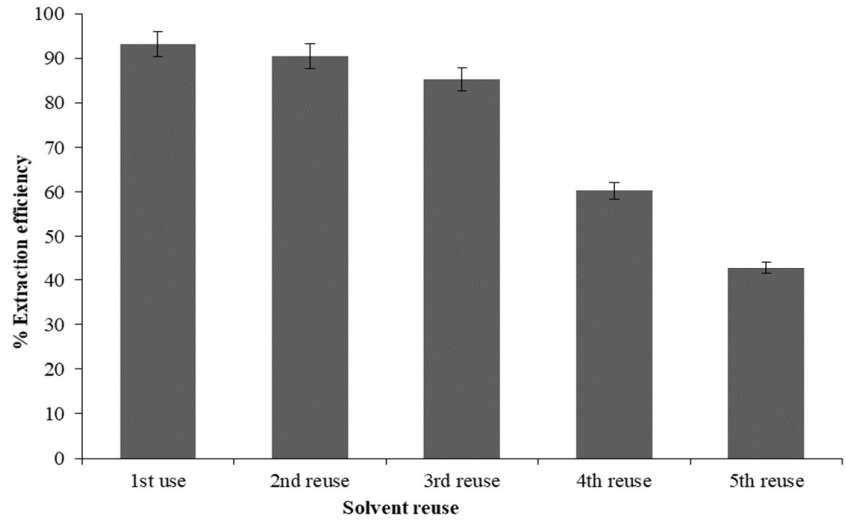
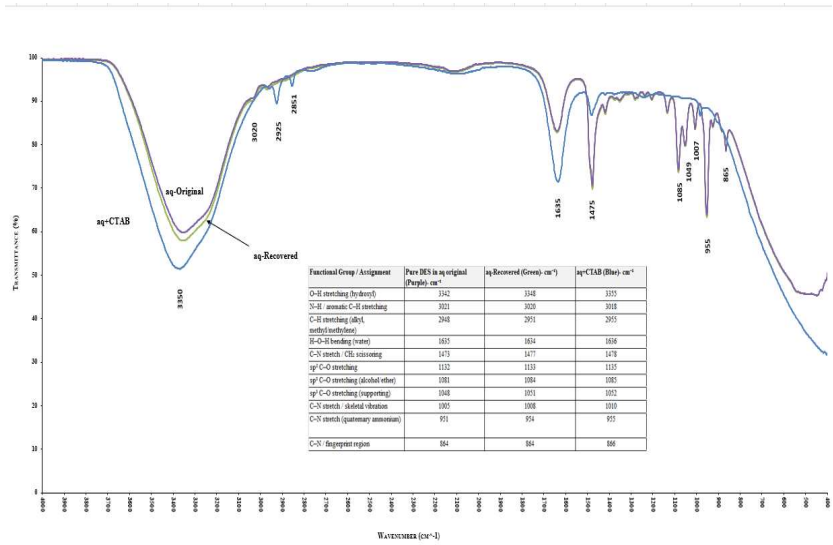


Fig. 10: Extraction efficiency as a function of solvent reuse.



Functional Group / Assignment	Pure DES in aq original (Purple)- cm^{-1}	aq-Recovered (Green)- cm^{-1}	aq+CTAB (Blue)- cm^{-1}
O–H stretching (hydroxyl)	3342	3348	3355
N–H / aromatic C–H stretching	3021	3020	3018
C–H stretching (alkyl, methyl/methylene)	2948	2951	2955
H–O–H bending (water)	1635	1634	1630
C–N stretch / CH$_2$ scissoring	1473	1477	1478
sp^2 C–O stretching	1132	1133	1135
sp^2 C–O stretching (alcohol/ether)	1081	1084	1085
sp^2 C–O stretching (supporting)	1048	1051	1052
C–N stretch / skeletal vibration	1005	1008	1010
C–N stretch (quaternary ammonium)	951	954	955
C–N / fingerprint region	864	864	866

Fig. 11: FTIR spectra of Aquoline -3, upper micellar layer and recycled Aquoline-3.

Table 2: List of solvents used in the extraction of phenol**.**

Sr. No.	Name of solvent	Feed concentration [ppm]	Volume ratio of solvent to feed	pH	Temp [K]	Time [h]	Extraction efficiency [%]	Ref.
1.	Iso-kerosene	100	1:1	6	323	2	70	(Abbassian et al. 2015)
2.	Methyl iso-butyl ketone	1000	1:1	2	303	0.25	97	(Abdelmonemet al. 2001)contact time, solvent/ feed ratio, shaking time and agitation speed on the removal of phenol by each solvent were investigated. The equilibrium condition was also determined and the rate of extraction was measured as a function of the above. The performance of each solvent is discussed with respect to the operating conditions. The results lead strongly towards the use of methyl iso-butyl keton (MIBK
3.	Di-isopropyl ether (DIPE)	1000	3:1	2	303	0.25	98	
4.	Toluene	1000	3:1	2	303	0.25	94	
5.	Benzene	1000	3:1	2	303	0.25	95.5	
6.	Ionic liquid $[Bmim]^+[BF_4]^-$ + tributyl phosphate	10	1:1	3	303	0.33	96	(Brinda Lakshmi et al. 2013)experiments have been carried out with a focus to reduce the volume requirement of solvent by mixing with imidazolium based ionic liquids (ILs
7.	Benzyl 2-ethylhexyl sulfoxide	10	1:1	2	298	0.08	98.7	(Wang et al. 2015)we synthesized a novel structure of sulfoxide, benzyl 2-ethylhexyl sulfoxide (BSO
8.	Aliquat – 336	2500	1:16.67	5	--	0.67	96.3	(Rao et al. 2009)
9.	Trioctylphosphine oxide (TOPO) -menthol HDES(mole ratio = 1:1)	7 wt %	1:1 mass ratio	--	298	26	96	(Wazeer et al. 2023)
10.	Fenchol- menthol HDES(mole ratio = 1:5)	1000	1:1	7	303	0.5	95.9	(Tan et al. 2025)
11.	Menthol and octanoic acid HDES (molar ratio of 1:1)	50	--	9.5	298	2.5	85	(Sas et al. 2019)
12.	Cumene	2167	--	7	298	0.5	80	(Liu et al. 2013)
13.	CTAB + Aquoline	5000	10:1	10	301	24.25	93.27	This work

surfactant skeletons during DES interactions (Su et al. 2015, Shettigar et al. 2018). In the upper micelle layer, the peak at 3355 cm-1 indicates hydroxyl groups in phenol and water. The range from 1417 to 1475 cm-1 corresponds to CH2 deformation in phenol. Phenolic OH in-plane deformation is observed at 1352 cm-1. A broad peak at 1248 cm-1 denotes C–O and OH bending modes in phenol. The FTIR spectra confirm that the structure of recovered Aquoline-3 closely matches that of the original Aquoline-3.3.

3.6. Comparison of Aquoline-3 with Other Solvents

Table 2 presents various solvents used as extractants for phenol removal from wastewater under significantly different operating conditions compared to those in our study. The Aquoline-3 + CTAB system effectively separated phenol from wastewater samples with high phenol concentrations, requiring only a small amount of Aquoline solvent relative to other solvents. While deep eutectic solvents (HDES) are efficient, they are costly and unsuitable for high phenol loads or larger wastewater volumes. Additionally, phase separation took 24 hours, which could limit industrial application due to the need for extra settling tanks. However, process intensification methods such as centrifugation and ultrasonication could accelerate micellization, making the process more scalable industrially. Alternatively, membrane filtration may facilitate faster separation of micelles. Despite these challenges, the combination of Aquoline-3 + CTAB offers a more environmentally friendly alternative.line-3 + CTAB would prove to be a more benign alternative.

4. CONCLUSIONS

A novel type of deep eutectic solvent, Aquoline (choline chloride to water molar ratio of 1:4), was employed to separate phenol from wastewater. The process was enhanced by CTAB surfactant, which forms micelles within Aquoline. Notably, this is the first study to utilize hydrophilic DES combined with CTAB for phenol removal from wastewater. Characterization of the solvent was conducted using UV-VIS, NMR, and FTIR spectroscopy. An impressive maximum extraction efficiency of 93.27% was achieved under optimized conditions: initial phenol concentration of 5000 ppm, temperature of 313 K, pH 10, CTAB concentration of 0.9 mM·L−1, and a wastewater-to-solvent volume ratio of 10. FTIR spectra of the recovered solvent confirmed that its original structure remained intact. The solvent demonstrated reusability for up to three cycles, with a gradual decrease in efficiency. Compared to other solvents, this system performs effectively with wastewater containing high phenol concentrations (up to 5000 ppm). Process intensification techniques such as centrifugation, ultrasonication, and membrane filtration could further improve settling time and expedite micelle separation. Future research should explore the extraction mechanism and conduct detailed structural analysis of the micelles.

5. ABBREVIATIONS

Aliquat – 336 - N-Methyl-N

N-dioctyloctan-1-aminium chloride

ChCl - Choline chloride

CTAB - Cetyl trimethyl ammonium bromide

DES - Deep eutectic solvents

DMSO - Dimethyl Sulfoxide

EPA - Environmental Protection Agency

FTIR - Fourier Transform Infrared spectroscopy

HBC - Hydrogen bond contributors

HBR - Hydrogen bond receivers

NMR - Nuclear Magnetic Resonance spectroscopy

pKa - Acid dissociation constant

TOPO - Trioctylphosphine oxide

UV - UV-Vis spectroscopy

GC - Gas chromatography

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