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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 solvents (DES), prepared from choline chloride as H-bond receiver and water as H-bond contributor. Separation of phenol from wastewater is facilitated by micelles formed in Aquoline due to 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, 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 hydrophilic deep eutectic solvent in the extraction of phenol from wastewater. The research will pave the way for the use of new type of eco-friendly solvents while adhering to principles of green chemistry.

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—and these substances 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 lives as it is carcinogenic and causes mutagenesis. Its presence is detrimental to aquatic ecosystems as it hinders 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 liver, kidneys, skin, 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 maximum of 0.1 ppm of phenol to be allowed in in industrial effluents. For drinking water, 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 into 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; Desai *et al.* 2022).

For removal of phenol, different methods such as, adsorption, liquid-liquid extraction, membrane separation, foam fractionation, electrocoagulation, electro-Fenton methods, steam distillation, chemical oxidation, precipitation, biological treatments (enzymatic and microbial) were used by researchers (Hinge *et al.*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, and limited selectivity in mixed wastewater, high costs , difficulties in regeneration and reusability (Das *et.al.* 2020; Adnan *et.al.* 2021;

Al-Ghouti *et al.* 2022; Ho *et.al*. 2022). Membrane separation techniques suffer from various problems, including membrane fouling, emulsion instability in case of liquid membranes , high production costs and potential degradation under harsh wastewater conditions in case of polyimide ultrafiltration membranes (Reis *et al.* 2011; Feng *et.al.*2021; Ali *et al.* 2021).Despite obtaining high extraction efficiency, application of enzymatic processes on 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. 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 (Mackay *et al.* 1973). Combining these solvents with an inert diluent presents an appealing approach. Following solvents were mentioned in 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 *et al.* 2013), dimethylsulfoxide (Chasib *et al.* 2013) , tetramethylene sulfolone (Chasib *et al.* 2013), diethylene glycol(Chasib *et al.* 2013), ethylene glycol (Chasib *et al.* 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 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 presence of hydrogen-bonding, formation of complex between cationic species and phenolate ions, and their hydrophobic nature. Structure of ILs, temperature and pH determined the efficiency. However, high viscosities and 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 receivers are termed as 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 depression in melting point. Due to ease in 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*. (2025), Rodríguez-Llorente *et al*. (2022), 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). Costly nature of ingredients negates the advantages offered by HDES and makes scale up unfeasible.

Aquoline is a new generation of deep eutectic solvent which uses water as HBC. It shows Newtonian behavior, has pH near to neutral, and is polar (Triolo *et al.*2021; Hirpara *et al.*2022).It could be used as a solvent, alongwith CTAB. Positively charged groups of CTAB (cetyltrimethylammonium bromide), a cationic surfactant , tend to attract negatively charged ions of phenol from sample due to electrostatic attraction (Shammala *et al.*1999; Malkoc *et al.* 2018). Huang *et al*. (2012) postulated that presence of surfactant micelles improved solubilization of phenol (Huang *et al.*2012). Hirpara *et al*. (2022) studied micellization behavior of various surfactants in Aquoline. Critical micelle concentration and aggregation number of CTAB was 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 effecting 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 DES were extensively used for drug delivery and as a reaction media. 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 into water (Dwamena et al. 2019).

To overcome drawbacks of existing solvents, this research work focuses on extraction of phenol from wastewater by using a combination of Aquoline (water + choline chloride) and a cationic surfactant (CTAB). It is our intention 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 first research work which explores application of cheap DES and surfactant combination for removal of phenol. Usage of new solvent system might help in reducing waste and contribute to sustainability of process.

2. MATERIALS AND METHODS

2.1 Chemicals

Phenol, choline chloride (98 % pure), 4-aminoantipyrine, potassium ferricyanide, ethanol (99 %), cetyl trimethyl ammonium bromide (> 98%) , 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. Method described in published research papers was used for 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 ratio of 1:1 and 1:5.

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

2.3 Characterization of Aquoline

UV absorption spectra of the selected Aquoline DES was studied on Shimadzu UV-1280 spectrophotometer. 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 by using 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 forms 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 concentration of phenol. Extraction efficiency is determined using the formula:

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

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

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

2.5 Analysis

Concentration of phenol in lower layer was found by UV spectrophotometer (Shimadzu - UV-1280) using 4-aminoantipyrine dye. Wavelength of 510 nm was used to measure absorbance of phenol in 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 is presented in figure 1. The spectra show strong absorption in 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 UV range must arise due to strong $\sigma \rightarrow \sigma^*$ transitions of the O–H bonds in water and ammonium groups of choline chloride. Absorbance steadily falls from 200 nm to 260 nm. Absorbance stabilizes at very low values (<0.1) beyond wavelength of 400 nm.

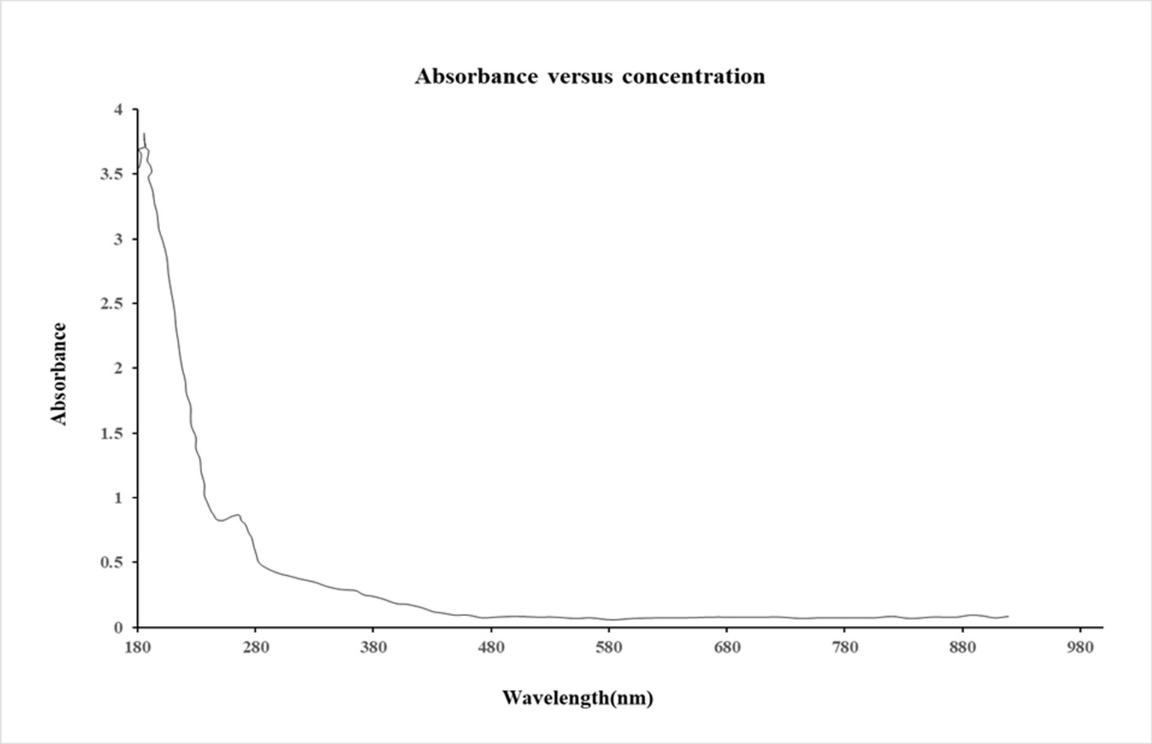


Figure 1: UV spectrogram of Aquoline- 3.

3.1.2 NMR spectra of Aquoline-3 DES

^{1}H and ^{13}C NMR spectra for Aquoline-3 is shown in figure 2 and figure 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 shifted downfield. Signal at 2.573 ppm represents DMSO.

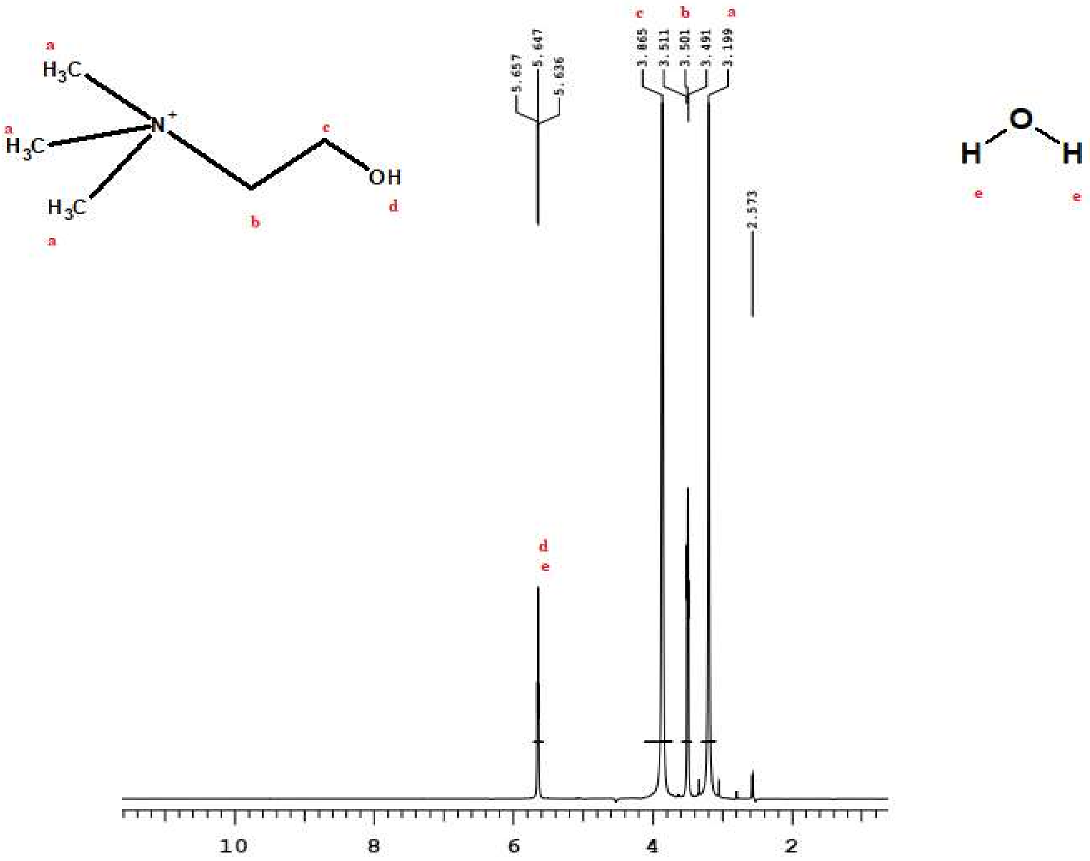


Figure 2: ^{1}H NMR spectra of Aquoline -3.

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($(CH_3)_3N^+$). 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.

Presence of choline chloride in Aquoline-3 is confirmed by ^{1}H NMR spectra. ^{13}C NMR confirms retainment of ChCl structure within superstructure of DES. Presence of hydrogen bonding and chlorine atom may have caused downshifting of peaks in both ^{1}H and ^{13}C NMR to higher frequencies (Delso *et al.*2019; Shahhosseini*et al.*2024).

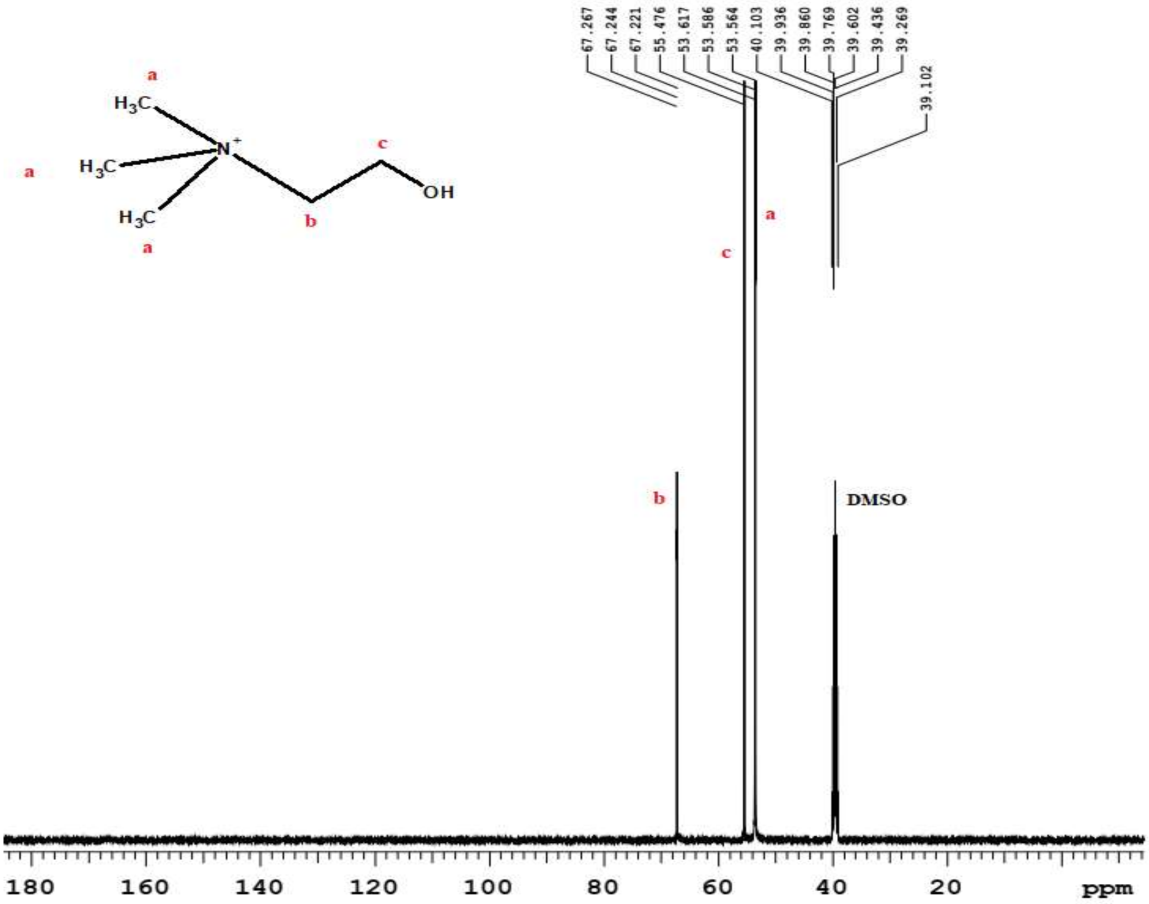
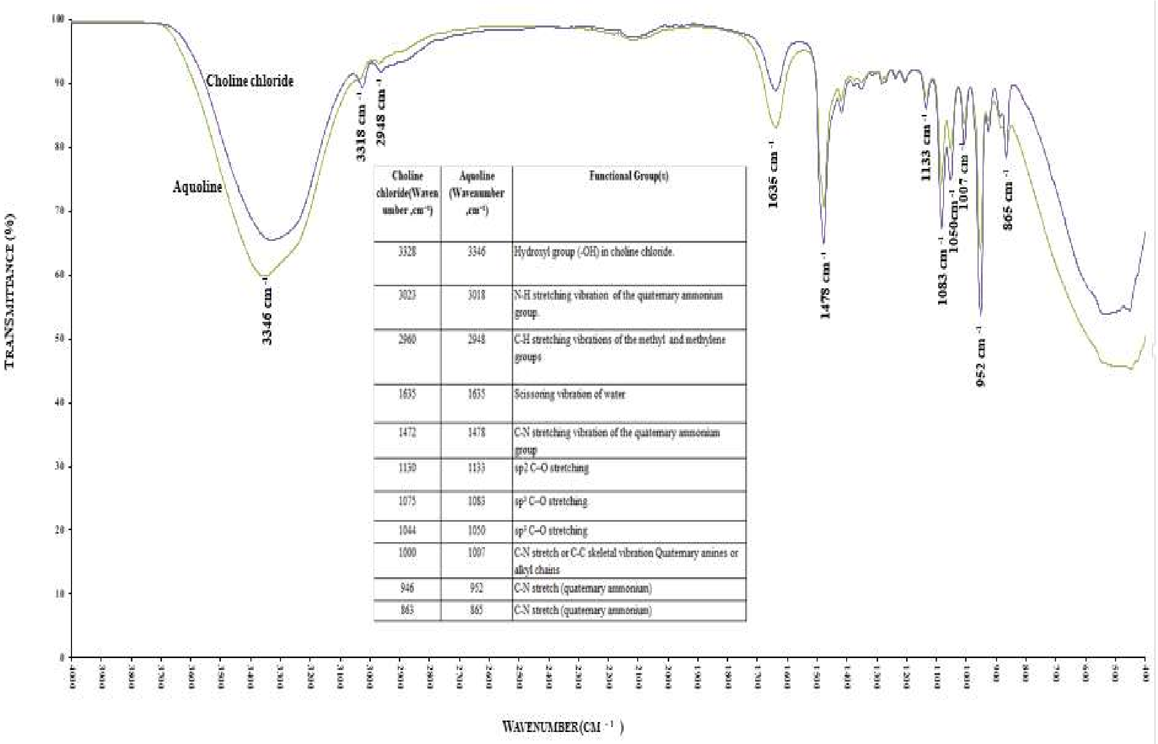


Figure 3: ^{13}C NMR spectra of Aquoline -3.

3.1.3 FTIR spectra of Aquoline -3

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



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	sp2 C–O stretching
1075	1083	sp^3 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)

Figure 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 physical and chemical properties DESs and affects its selectivity (Tan *et al.*2025). The prepared Aquoline DESs (with varying mole ratio of HBC and HBR) were used in extraction experiments and results shown in figure 5. Aquoline -3 (mole ratio of HBR to HBC = 1:4) gave 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 rest of the experiments.

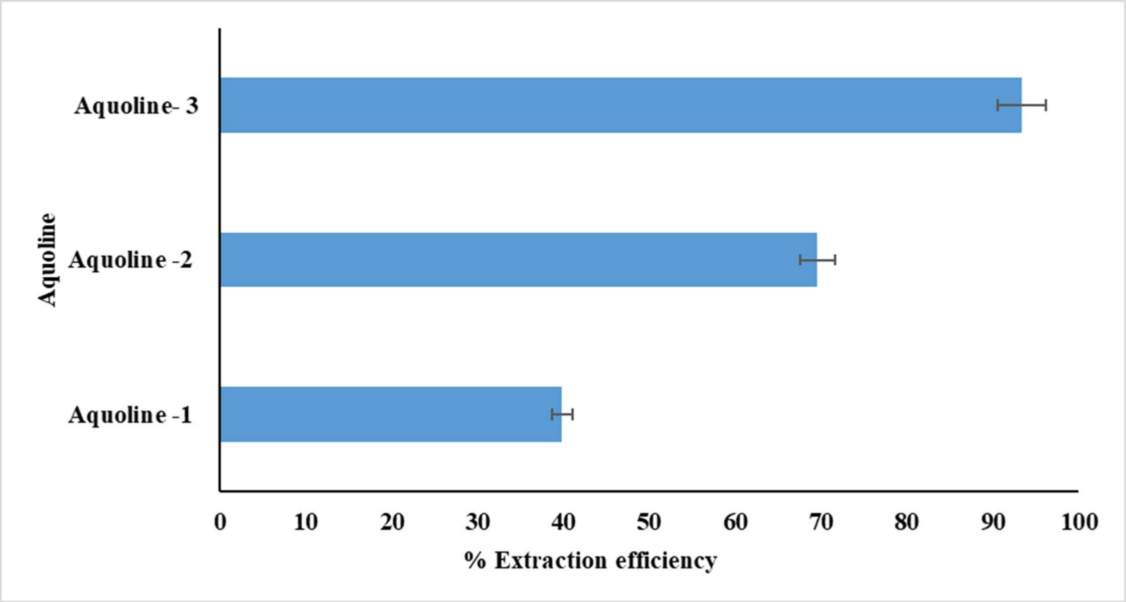


Figure 5: Selection of Aquoline with proper mole ratio for extraction of phenol.

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.

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.

3.3.1 Influence of changing initial phenol concentration on extraction efficiency

Effects of changing concentration of phenol in feed (100 to 5000 ppm) on phenol recovery is depicted in figure 6.The extraction efficiency showed positive trend with subsequent increase in phenol concentration in feed . At higher concentration, 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 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 had no effect on extraction efficiency when fenchol-menthol HDES was used as an extractant. This was ascribed to the fact that degree of solvation of phenol was same at all concentrations(Tan *et al.*2025).

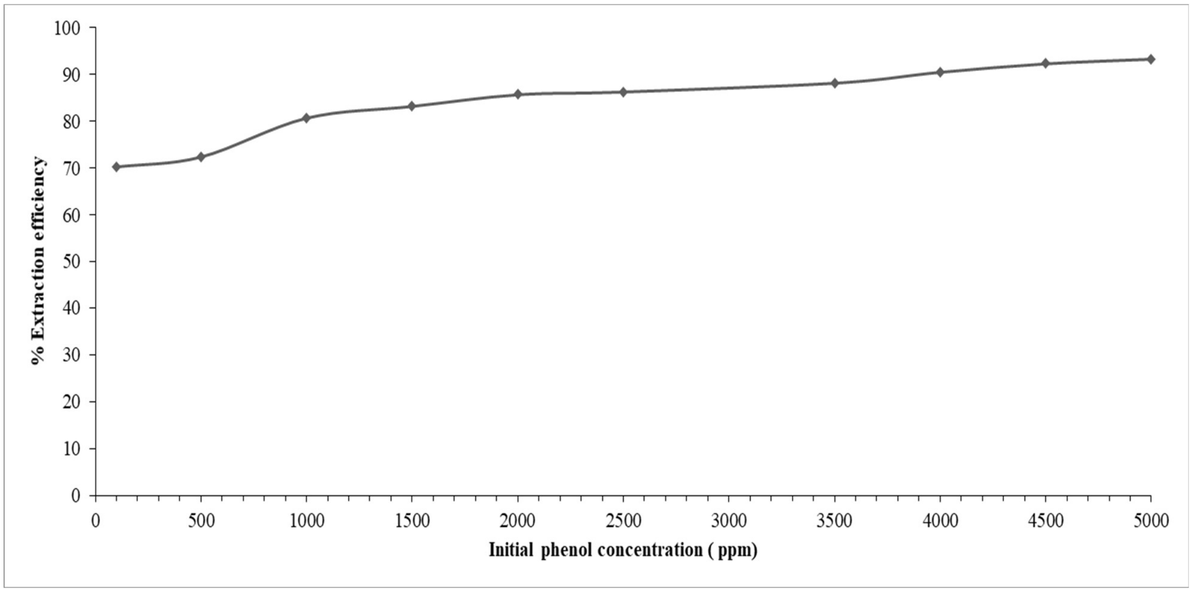


Figure 6: Influence of changing initial phenol concentration on extraction efficiency.

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

3.3.2 Influence of changing CTAB concentration on extraction efficiency

CTAB forms micelles in Aquoline at CMC value of 0.82 mM/L. The micelles formation involves incorporation of hydrophobic head of surfactant into micelle due to high polarity of DES (Hirpara *et al.*2022). When amount of surfactant in water was higher than its CMC, aggregates of large size micelles were formed. Initially, phenol molecules in water tend to attach to outer layer of micelles. With increasing concentration, phenol molecules penetrate into 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 to 1.3 mM/L and results are shown in figure 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 solubilized, resulting in formation of micellar aggregates. Maximum extraction efficiency was obtained at CTAB concentration of 0.9 mM/L.

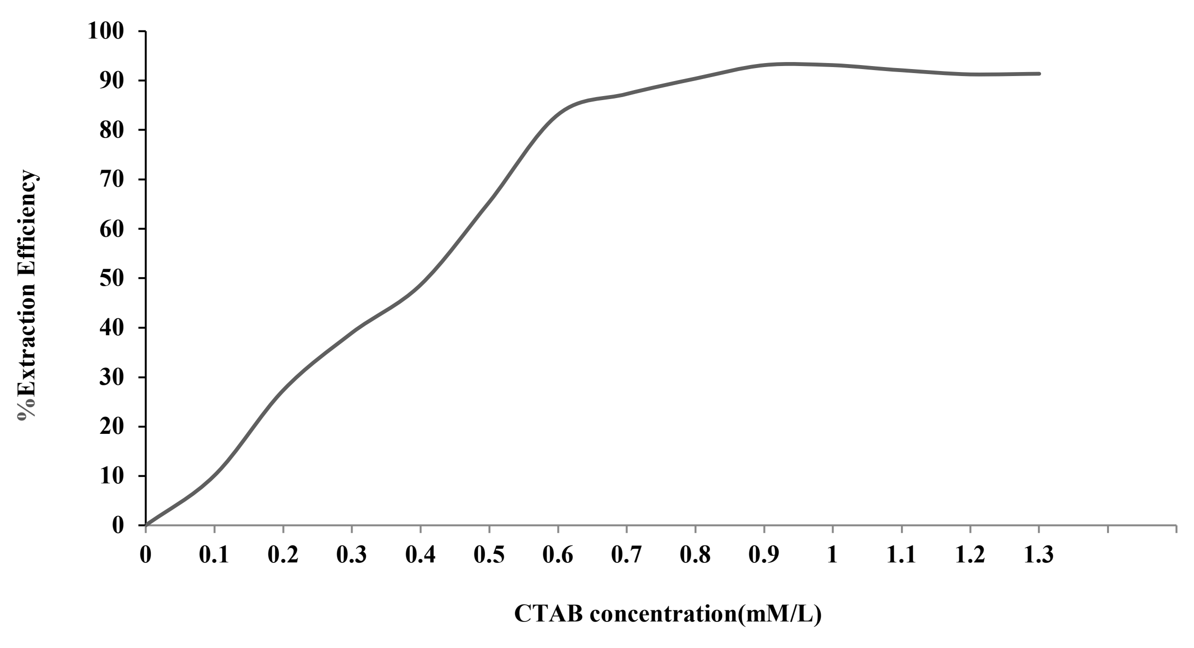


Figure 7: Influence of changing CTAB concentration on extraction efficiency.

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 phenolic -OH group and water (Shammala *et al.*1999).

To facilitate easier extraction of phenol, pH has to be kept higher to promote electrostatic interactions between CTAB and phenoxide ions. In acidic pH range, electrostatic interactions between phenolate ion and CTA^+ are negligible and extraction proceeds due to hydrophobic interactions. At pH = 7, phenol molecule gets attached on outer layers of micelles. At alkaline pH ,phenolate ions interact with CTA^+ ions, leading to increased solubilization of phenol (Sabatino *et al.*2010).

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

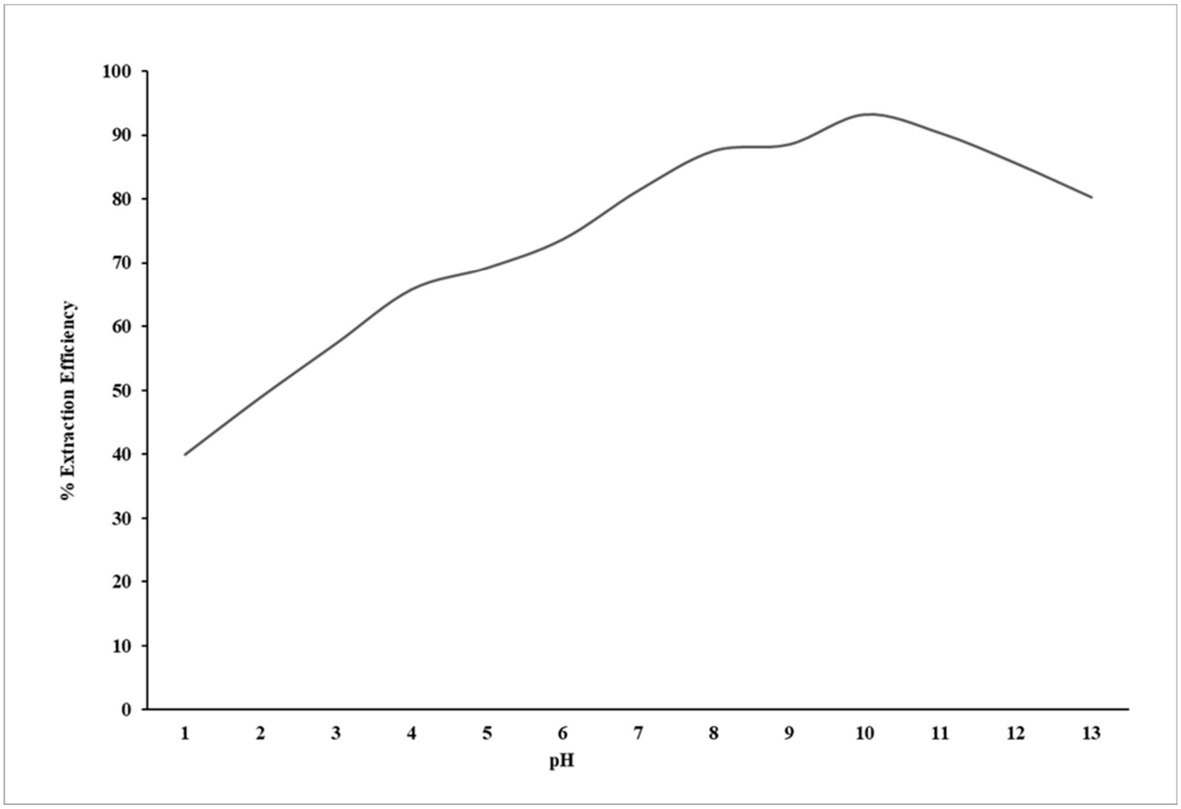


Figure 8: Influence of changing pH on extraction efficiency.

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.

3.3.4 Influence of changing volume ratio of wastewater sample to Aquoline on extraction efficiency

Volume ratio of waste water sample to Aquoline-3 was changed from 1 to 20 (Figure 9).With increase in treat ratio, extraction efficiency increased. Maximum efficiency was obtained at treat ratio of 10. Beyond this ratio, efficiency decreased due less amount of solvent available. Volume ratio of 10:1 was selected. Similar behavior was observed by Lakshmi *et al*.(2013) and treat ratio of 5:1 was selected (Brinda Lakshmi *et al*. 2013).Tan *et al*.(2013) obtained maximum extraction at treat ratio of 1 when HDES was used as solvent (Tan *et al*. 2025).

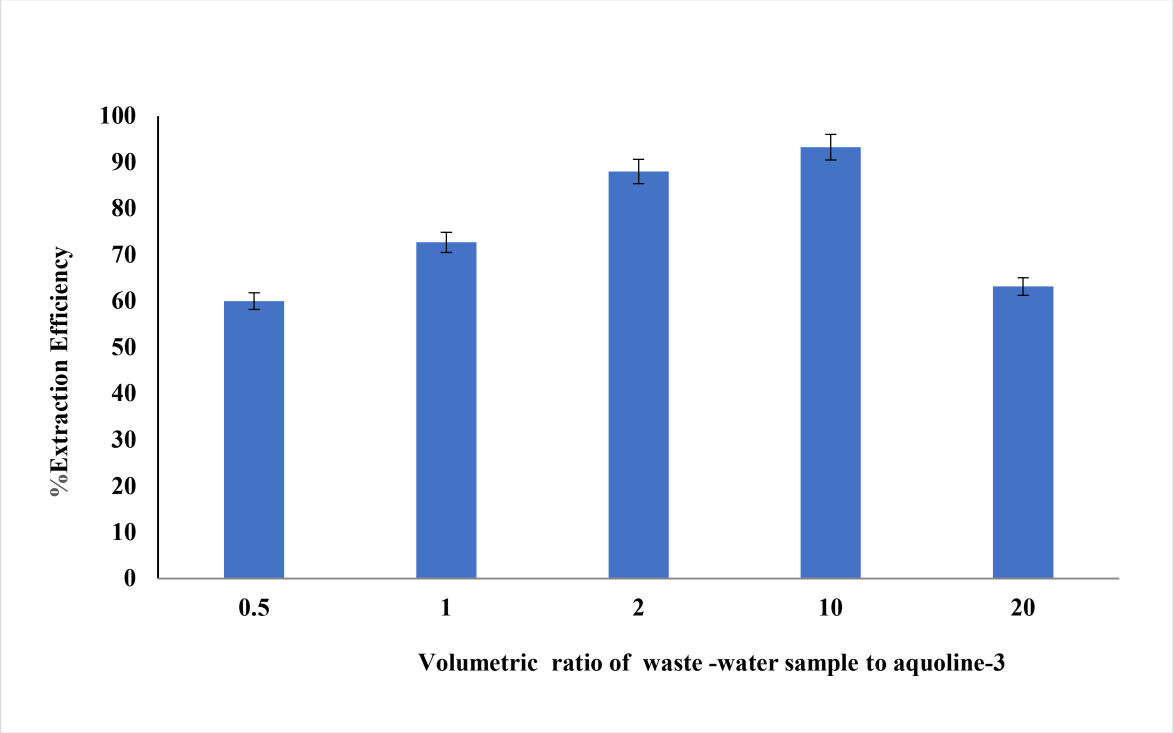


Figure 9: Influence of changing volume ratio of wastewater sample to Aquoline-3 on extraction efficiency.

Extraction parameters: Initial phenol concentration = 5000 ppm, temperature = 301 K, pH =10, CTAB concentration = 0.9 mM/L.

Based on optimization studies, following optimized parameters were obtained :Initial phenol concentration = 5000 ppm, temperature =301 K, pH =10, CTAB concentration = 0.9 mM/L, 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 phenolic -OH group and water, 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 outer layer. Above the CMC, CTAB formed micelles that encapsulated phenol molecules within their hydrophobic cores, while the charged head groups remain 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 enhanced 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 of 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 separation of CTAB. The top organic layer obtained after extraction consists of CTAB which was recovered after evaporation of 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 comprised of choline chloride. It was subjected to evaporation to adjust ratio of choline chloride and water.

Despite not being considered as 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 (Figure 10). A gradual decline in extraction efficiency was noticed. It may be due to gradual decomposition of choline chloride during repeated recovery operations. The recycling studies were carried for only four cycles. Long term reusability and stability of Aquoline was beyond ambit of this study.

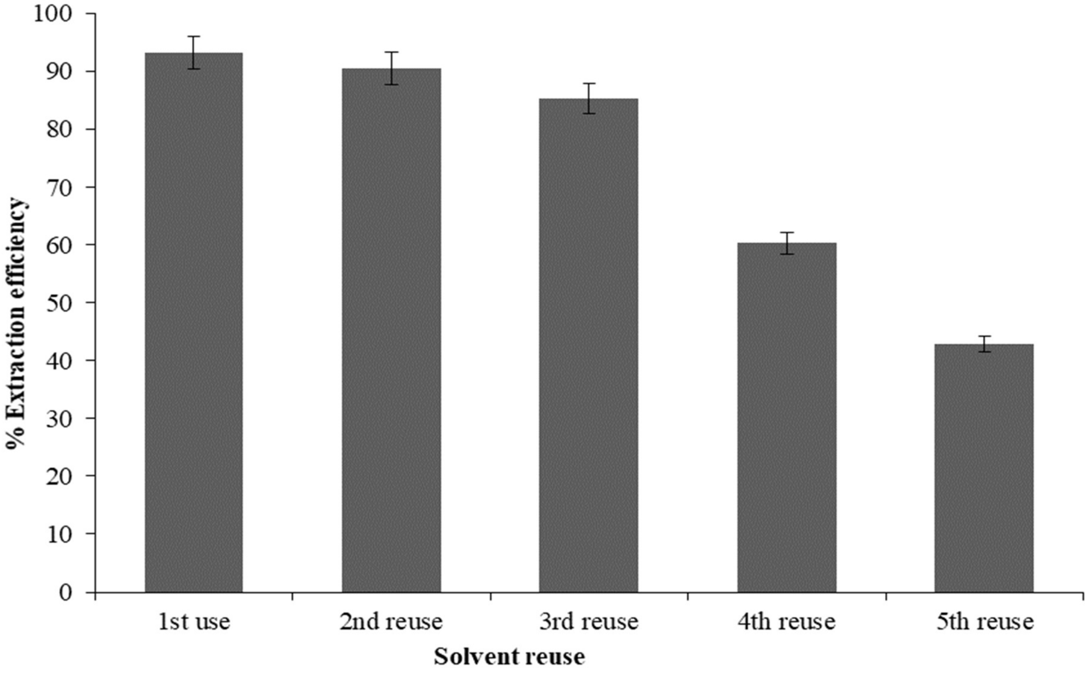
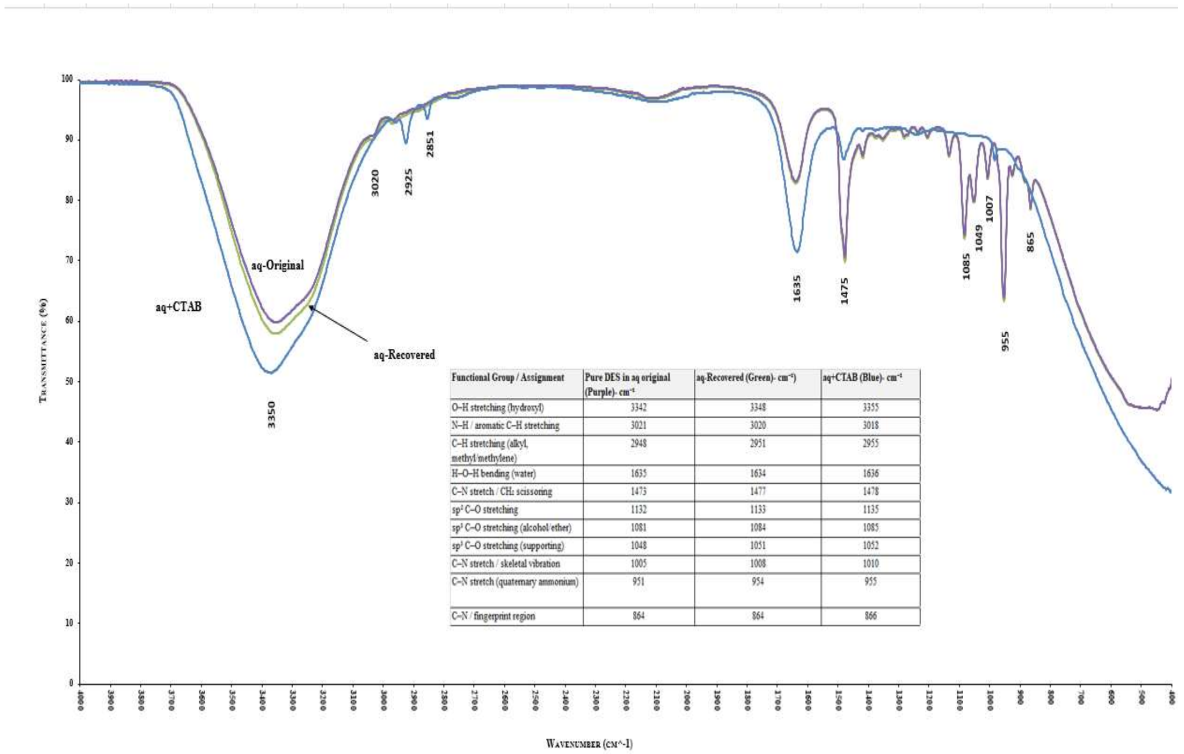


Figure 10: Extraction efficiency as function of solvent reuse.

Extraction parameters: Initial phenol concentration = 5000 ppm, temperature = 301 K, pH =10, CTAB concentration = 0.9 mM/L, volume ratio of wastewater solution to DES = 10.

Figure 11 shows FTIR spectra for pure Aquoline, recycled Aquoline-3 and Aquoline-CTAB upper micelle layer. Peaks at 3342 and 3348 cm-1 indicate the presence of hydroxyl groups in water. Shift to a higher wave number indicates interaction of micelles with Aquoline. N-H stretching ranging from 3018 to 3021 cm^{-1} reflects changes in CTAB carbon chain due to presence of phenol thus substantiating the previous observation of micelle DES interaction. Small C-H shifts at 2950 cm^{-1} demonstrates CTAB's participation in the DES structure. Chemical shifts observed in the fingerprint region of 946 cm^{-1} to 864 cm^{-1} show C-N stretching from which it can be deduced that quaternary ammonium present in CTAB has interacted with phenol and DES thus exhibiting interdependency of CTAB and Aquoline-3 to extract phenol. This is further seen in sp^3 C–O based shifts at 1075 cm^{-1} which show that choline hydroxyl interactions have taken place . Peaks ranging from 1004 to 1007 cm^{-1} depict typical vibrations experienced by the skeleton of a surfactant while interacting with a DES (Zhu *et al.*2015; Shettigar *et al.*2018).

For upper micelle layer, peak at 3355 cm^{-1} showed presence of hydroxyl groups in phenol and water. Peaks ranging from 1417 to 1475 cm^{-1} show CH_2 deformation in phenol. Phenolic OH in plane deformation by depicted at peak at 1352 cm^{-1}.Broad peak at 1248 cm^{-1} denotes C–O and OH bending modes in phenol (Altuntaş *et al.*2012).The FTIR spectra proved that structure of recovered Aquoline-3 clearly matches with original Aquoline-3.



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	1636
C–N stretch / CH_2 scissoring	1473	1477	1478
sp^2 C–O stretching	1132	1133	1135
sp^3 C–O stretching (alcohol/ether)	1081	1084	1085
sp^3 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

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

3.6 Comparison of Aquoline-3 with other solvents

Table 2 lists various solvents utilized as extractants for removal of phenol from wastewater under vastly different operating conditions when compared with our research. Aquoline -3 + CTAB system was able to separate phenol from wastewater samples with high phenol concentration. Low amount of Aquoline solvent was required, compared to other solvents. HDES ,although efficient, are very costly and can not be used for high phenol loading and higher volumes of wastewater. Long time (24 hours) was required for phase separation may limit industrial use due to requirement of extra settling tanks. However, process intensification strategies such as centrifugation and ultrasonication could speed up the micellization process and make the process industrially scalable (Banjare *et al.* 2020; Merouani *et.al.* 2024).Alternatively, membrane filtration might help in separation of micelles at faster rates (Mei *et al.*2020). Despite these limitations, the combination of Aquoline-3 + CTAB would prove to be more benign alternative.

Table 2: List of solvents used in 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	(Abdelmonem , *et al.* 2001)
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)
7	Benzyl 2-ethylhexyl sulfoxide	10	1:1	2	298	0.08	98.7	(Wang *et al.*2015)
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

4. Conclusion

A new type of deep eutectic solvent ,Aquoline (mole ratio of choline chloride to water = 1:4) was utilized for separation of phenol from wastewater. The separation was facilitated by CTAB surfactant which forms micelles in Aquoline. This is first research work to use hydrophilic DES and CTAB for separation of phenol from wastewater. The characterization of solvent was carried out by UV-VIS, NMR and FTIR spectroscopy. Highest extraction efficiency of 93.27% was obtained under optimized conditions (Initial phenol concentration = 5000 ppm, temperature = 313 K , pH =10, CTAB concentration = 0.9 mM/L, volume ratio of wastewater to solvent = 10). FTIR analysis of the recovered solvent indicated that original structure of DES was retained. The solvent could be reused thrice with gradual drop in efficiency. Compared with other solvents, this system can be used on wastewater samples with high phenol loading (5000 ppm). Process intensification strategies(such as centrifugation , ultrasonication ,membrane filtration) could help in optimizing settling time and fast separation of micelles.Future studies need to focus on mechanism of extraction and structural characterization of micelles .

Abbreviations used:

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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