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Research Article

Room Temperature Ionic Liquid Based Extraction And Recovery Of Dyes From Their Aqueous Solutions

Chander Prakash[1] and Shafila Bansal[2]

[1]M.L.S.M. College, Sunder Nagar, Himachal Pradesh, India.
[2]Mehr Chand Mahajan DAV College for Women, Sector 36-A, Chandigarh.

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ABSTRACT

An attempt has been made for the removal of cationic/anionic dyes from water using room temperature ionic liquids (RTILs). RTILs *viz.* 1-butyl-3-methylimidazolium hexafluorophosphate ([BMIM][PF_6]), 1-ethyl-3-methylimidazolium bis(trifluoromethylsulfonyl)imide ([EMIM][NTf_2]), 1-butyl-3-methylimidazolium tetrafluoroborate ([BMIM][BF_4]) and 2-Hydroxyethyl-trimethylammonium L-(+)-lactate ([(C_2H_4OH)-$(CH_3)_3N$][Lactate])/CL have been applied for the removal of dyes like Rhodamine 6G (Rh6G), congo red (CR) and coomassie brilliant blue R-250 (CBB). The factors affecting the removal of these contaminants from their aqueous solution such as effect of hydrophilicity/hydrophobicity of RTILs, concentration of used RTILs and effect of pH of the dye solution have been investigated in order to study the mechanism of the extraction process. Partition coefficient, $P_{RTIL/W}$ of the dyes between RTILs and aqueous phases as well as their extraction efficiencies E% have been calculated and analyzed. The most remarkable feature of our work is the recovery of used RTIL. Also, we have reused the recovered RTIL for the further extraction processes to compensate the cost factor.

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Corresponding Author: *Shafila Bansal, Mehr Chand Mahajan DAV College for Women, Sector 36-A, Chandigarh*

INTRODUCTION

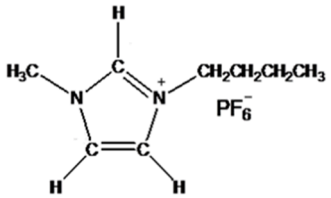
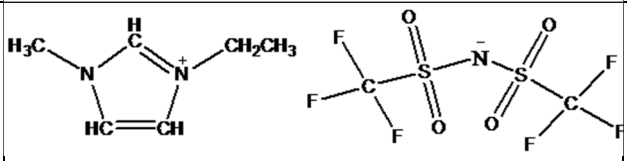
Wastewater from industries like textile, dyeing, printing, cosmetics, food coloring, papermaking, etc. is a major contributor of colored effluents.[1] Textile industry consumes large amount of water and different types of dyes impart color to effluents. The dyes/colors are toxic and are major pollutants, causing environmental problems as well as health hazards to human beings and aquatic animals.[2,3] Hence, in order to avoid issues due to the discharge of dyes into the ecosystem, there is a need to eradicate the residual dyes from the large volume of aqueous effluent.

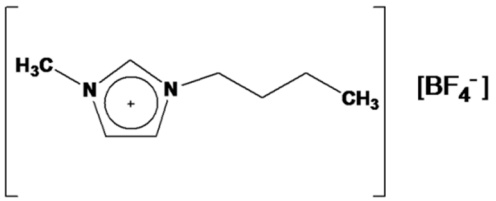
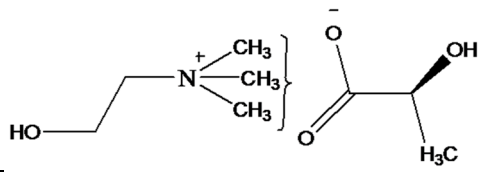
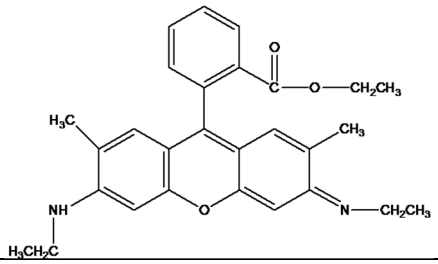
There are number of conventional ways[4,5] for the removal of such contaminants from water. Different methods including adsorption[6-9], biosorption[10], biological methods[11], reverse micelles[12,13], reverse osmosis[14], micellar enhanced ultrafiltration[15], nanofiltration[16], colloidal gas aphrons[17], electrochemical treatments[18] and UV-ozone or UVH_2O_2[19], etc have been tested for removal of dyes. Each method has its own advantages and disadvantages, which have been extensively reviewed.[20,21] But there is a need to develop environment friendly and economically effective method for the extraction process.

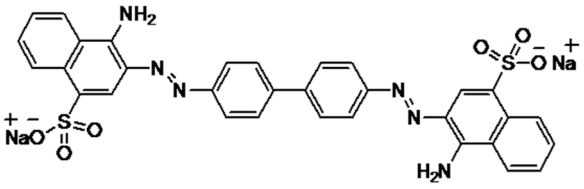
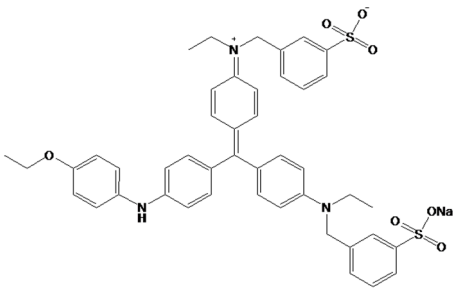
Adsorption methods are doing well[6-9] however, these are associated with problems of high production cost, unsatisfied adsorption capacity, separation and regeneration problems. Also, recycling of used adsorbents makes adsorption processes time-consuming as well as expensive. Low biodegradability of dyes makes biological methods less effective.[11] Micellar enhanced ultrafiltration and colloidal gas aphrons are not very efficient as compared to other known methods.[15,17] The degradation of dyes can be achieved by enzymatic reduction/oxidation reactions using bacterial species[22]. In recent years, nanomaterials have been attracting more attention in degradation of dyes[23-25]. However, in some cases, the degraded product has been found to be more harmful than the original effluents e.g. degraded products of azo dye are more carcinogenic and toxic[26-28]. The combination of biological methods with physical or chemical treatment processes have also been used but these methods are generally less efficient, costly and of limited applicability. "Large investment in equipment of recently used electrochemical processing[29,30] makes this method less effective". Hence, liquid–liquid extraction method is best choice for the separation of dyes[31]. However the use of traditional organic solvents for extraction processes leads to production of volatile organic compounds in atmosphere, which effects the environment and human health[32]. Recently, room temperature ionic liquids (RTILs) have been found to be best extractant for removal of water contaminants[33,34] due to their almost negligible volatility, less toxicity, non-flammability and ability to dissolve in wide range of solvents. Use of RTILs in separation processes and their mechanisms have been investigated by several research groups during the past few years[35-37].

To acquire the desired goal, we have used RTILs; 1-butyl-3-methylimidazolium hexafluorophosphate ([BMIM][PF_6]), 1-ethyl-3-methylimidazolium bis(trifluoromethylsulfonyl)imide ([EMIM][NTf_2]), 1-butyl-3-methylimidazolium tetrafluoroborate ([BMIM][BF_4]) and 2-Hydroxyethyl-trimethylammonium L-(+)-lactate ([(C_2H_4OH)-$(CH_3)_3$N][Lactate])/CL RTILs for the removal of dyes : Rhodamine 6G (Rh6G), congo red (CR) and coomassie **brilliant blue R**-250 (CBB) from their aqueous solutions. Structures and nature of used components are depicted in table 1. The factors like hydrophilicity/hydrophobicity of RTILs, concentration of used RTILs and pH of the dye solution affecting the extraction of cationic as well as anionic dyes have been investigated in order to study the extraction mechanism. The method has further been optimized for the recovery and recyclability of used RTILs.

Table 1 *Structure and nature of components used*

Components	Structure	Hydrophilicity/ Hydrophobicity	Nature
RTILs			
1-butyl-3-methylimidazolium hexafluorophosphate ([BMIM][PF_6])		Hydrophobic	Ionic
1-ethyl-3-methylimidazolium bis(trifluoromethylsulfonyl)imide ([EMIM][NTf_2])		Hydrophobic	Ionic

Components	Structure	Hydrophilicity/Hydrophobicity	Nature
RTILs			
1-butyl-3-methylimidazolium tetrafluoroborate ([BMIM][BF_4])		Hydrophilic	Ionic
2-Hydroxyethyl-trimethylammonium L-(+)-lactate (CL)		Hydrophilic	Ionic
Dyes			
Rhodamine 6G (Rh6G)		Lipophilic	Cationic

Congo red (CR)		Lipophilic	Anionic
Coomassie brilliant blue R-250 (CBB)		Lipophilic	Cationic

MATERIALS AND METHODS

Chemicals used

[BMIM][PF_6] (purum ≥97% HPLC), [EMIM][NTf_2] (≥98% ^{1}H-NMR), CL (≥95.0% (T)) and [BMIM][BF_4] (purum ≥97% HPLC) were purchased from SIGMA-ALDRICH. 1-butanol was purchased from FLUKA. Dyes; CBB (SISCO), Rh6G (SD FINE CHEMICALS LTD.) and CR (SD FINE CHEMICALS LTD.) were used in the present work. Samples were prepared in doubly distilled water. All the experiments were performed in triplicate to check reproducibility of data.

Methods

The stock solutions of 3×10^{-5} M of Rh6G, CR and 6×10^{-5} M of CBB were prepared by directly dissolving them into distilled water. UV-vis spectrum of each solution was obtained using Thermo Fisher Scientific Evolution 160 UV-visible spectrophotometer at their characteristic wavelengths *viz.* Rh6G; 525 nm, CBB; 554 nm and CR; 494 nm. 10 ml of each solution was taken in glass vials. To each of the solution, known concentrations of all the four used RTILs were directly added. Then vortex shaking of these mixtures for 1 minute was done and was left undisturbed for another 1 minute. Precipitation of dyes was visibly evident in each case as the colored solutions became nearly clear in some cases leaving behind the contaminants at the bottom. UV-vis spectrum of almost clear supernatant was observed in order to calculate the extraction efficiency by using the formula:

$$E\% = \frac{C_i - C_f}{C_i} \times 100\% \qquad (1)$$

where, C_i and C_f represents initial and final concentrations of used dye in an aqueous phase, respectively. Partition coefficient ($P_{RTIL/W}$) of the dyes between the RTILs and aqueous phases was estimated using equation

$$P_{RTIL/W} = \left\{\frac{C_i - C_f}{C_i}\right\} \times \frac{V_{aq}}{V_{RTIL}} \qquad (2)$$

where, V_{aq} and V_{RTIL} are the volumes of aqueous phase and RTILs, respectively. In order to study the effect of pH on the extraction efficiencies, the pH of the aqueous solutions was adjusted with HCl (0.1 mol L^{-1}) and NaOH (0.1 mol L^{-1}) solutions using pH meter (Cyberscan 510). To confirm the identity of recovered RTIL, FTIR spectra of pure as well as recovered RTIL were recorded using Perkin-Elmer (RX1) FT-IR spectrometer, in the frequency range of (4400–350) cm^{-1}. The uncertainty in the measurement of wave number ν was within ±0.01 cm^{-1}.

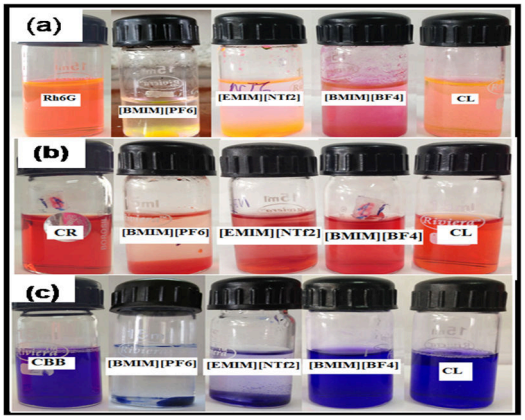
RESULTS AND DISCUSSION

Effect of hydrophilicity/hydrophobicity of RTIL

To understand the effect of hydrophilicity/hydrophobicity of RTILs on the

extraction efficiency of dyes, 2, 3 and 4 % of each RTIL was added in 10 ml of prepared solutions of Rh6G, CBB and CR respectively. A vortex shaking for 1 minute of this sample lead to precipitation of the dyes in RTILs (figure 1). UV-vis spectra of the supernatants left over were obtained in order to confirm the removal of all the three dyes in each RTIL (figure 2). The values of $P_{RTIL/W}$ of each dye against the used RTILs are given in table 2 and graphically shown in figure 3. Figure 2 confirms that hydrophobic RTILs show the best removal efficiency for all the dyes. Figure 3 shows that $P_{RTIL/W}$ of dyes in RTIL/aqueous solution follow the trend $P_{[BMIM][PF_6]/W} > P_{[EMIM][NTf_2]/W} > P_{[BMIM][BF_4]/W} > P_{CL/W}$.

Fig. 1. Removal of (a) Rh6G (b) CR (c) CBB using each RTIL



$P_{RTIL/W}$ of dyes is more in hydrophobic RTILs and it confirms that hydrophobicity of RTILs plays an important role in the removal of dyes. The values of $P_{RTIL/W}$ show that [BMIM][PF_6] has maximum removal efficiency of dyes. In the case of CBB, the values of $P_{RTIL/W}$ are negative (table 2) for both hydrophilic RTILs, suggesting the interaction of dye with RTILs instead of precipitation. Gharehbaghi et al.[38] have also extracted CR using hydrophobic 1-hexyl-3-methyl-imidazolium bis(trifluorosulfonyl)imide up to 95 % in 4-5 minutes, however, they also have used a dispersant along with RTIL. On the contrary, we have used pure RTILs and minimized the time to 1 minute only.

Fig. 2. UV-vis spectra showing (a) Rh6G precipitation using 2 % of each RTIL (b) CR precipitation using 4 % of each RTIL (b) CBB precipitation using 3 % of each RTIL

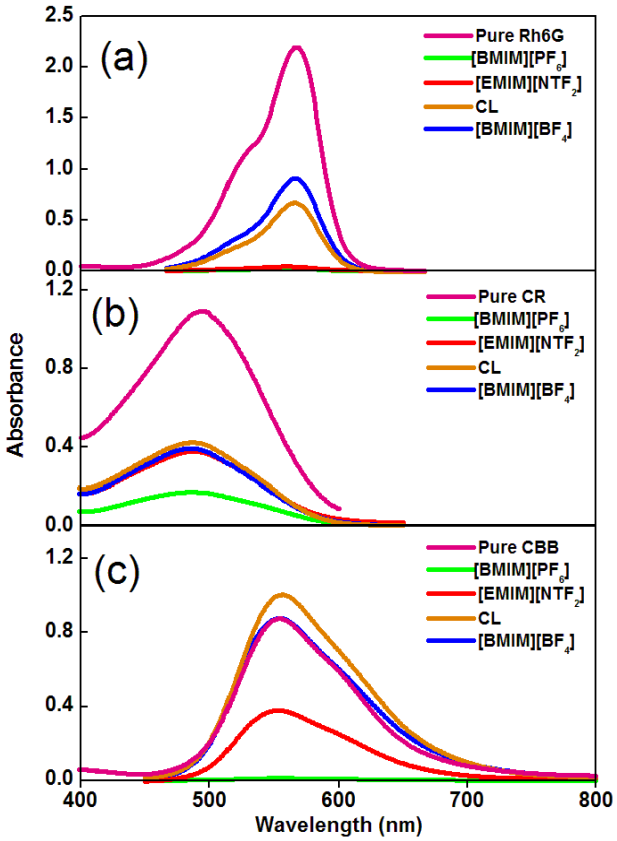
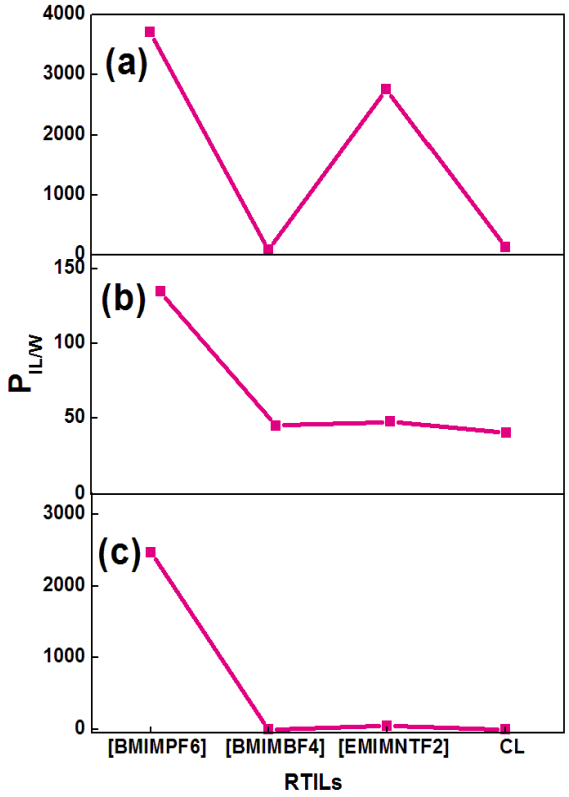


Fig. 3. Plot of partition coefficients $P_{RTIL/W}$ of (a) Rh6G (b) CR (c) CBB against the used RTILs



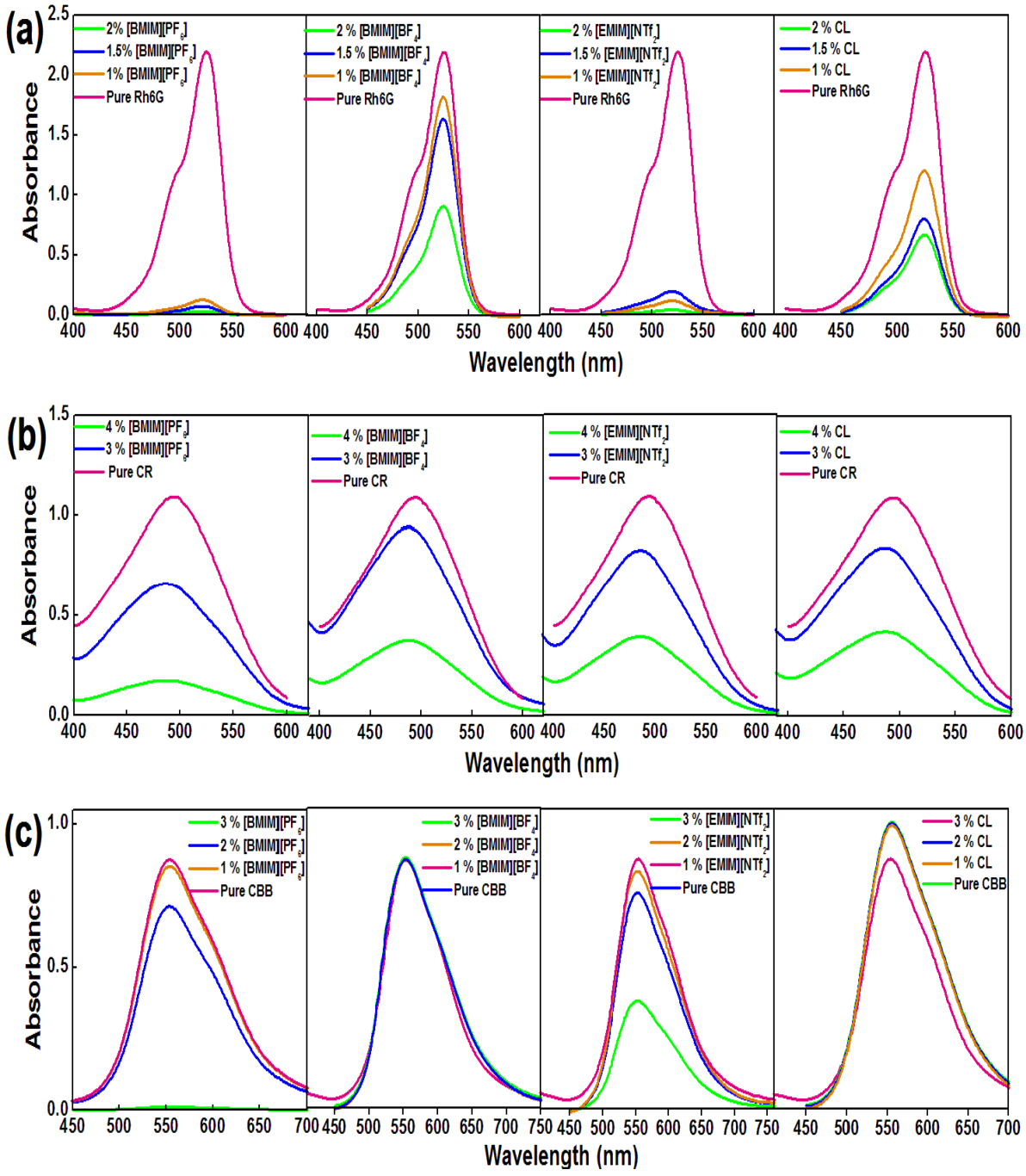
Effect of concentration of RTILs

A minimum amount of 1 % of RTILs was normalized to extract Rh6G, CBB and 3 % of RTILs was normalized to extract CR from aqueous solutions of dyes. However, to observe the effect of concentration of RTILs on the removal efficiency, different amount of RTILs were used for the extraction of dyes from their aqueous solution. Figure 4 illustrates the UV-vis spectra for the removal of dyes using different amounts of each RTIL. It is clear from figure 4 that hydrophobic [BMIM][PF_6] is more efficient in extraction of dyes than other RTILs. Figure 4 (a) shows that the addition of 2 % (0.0704 M) of [BMIM][PF_6] leads to almost complete precipitation (98.69 %) of Rh6G. Figure 4 (b) confirmed that 4 % (0.1383 M) of [BMIM][PF_6] is capable of removing 84 % of CR from its aqueous solution. In the case of CBB, 3% (0.1045 M) of [BMIM][PF_6] results in removal of 98.67 % of the dye from its aqueous solution (figure 4 (c)). Percentage removal of dyes using different concentrations of RTILs is given in figure 5.

Table 2 $P_{RTIL/W}$ of dyes in each RTIL

Contaminates		[BMIM][PF_6]	[BMIM][BF_4]	[EMIM][NTf_2]	CL
Rh6G	$P_{RTIL/W}$	3709.40	79.31	2764.26	125.84
CR		134.57	45.09	47.82	40.22
CBB		2466.42	-0.06	43.88	-4.22

Fig. 4. Plot of absorbance vs wavelength for (a) Rh6G (b) CR (c) CBB using different amount of each RTIL

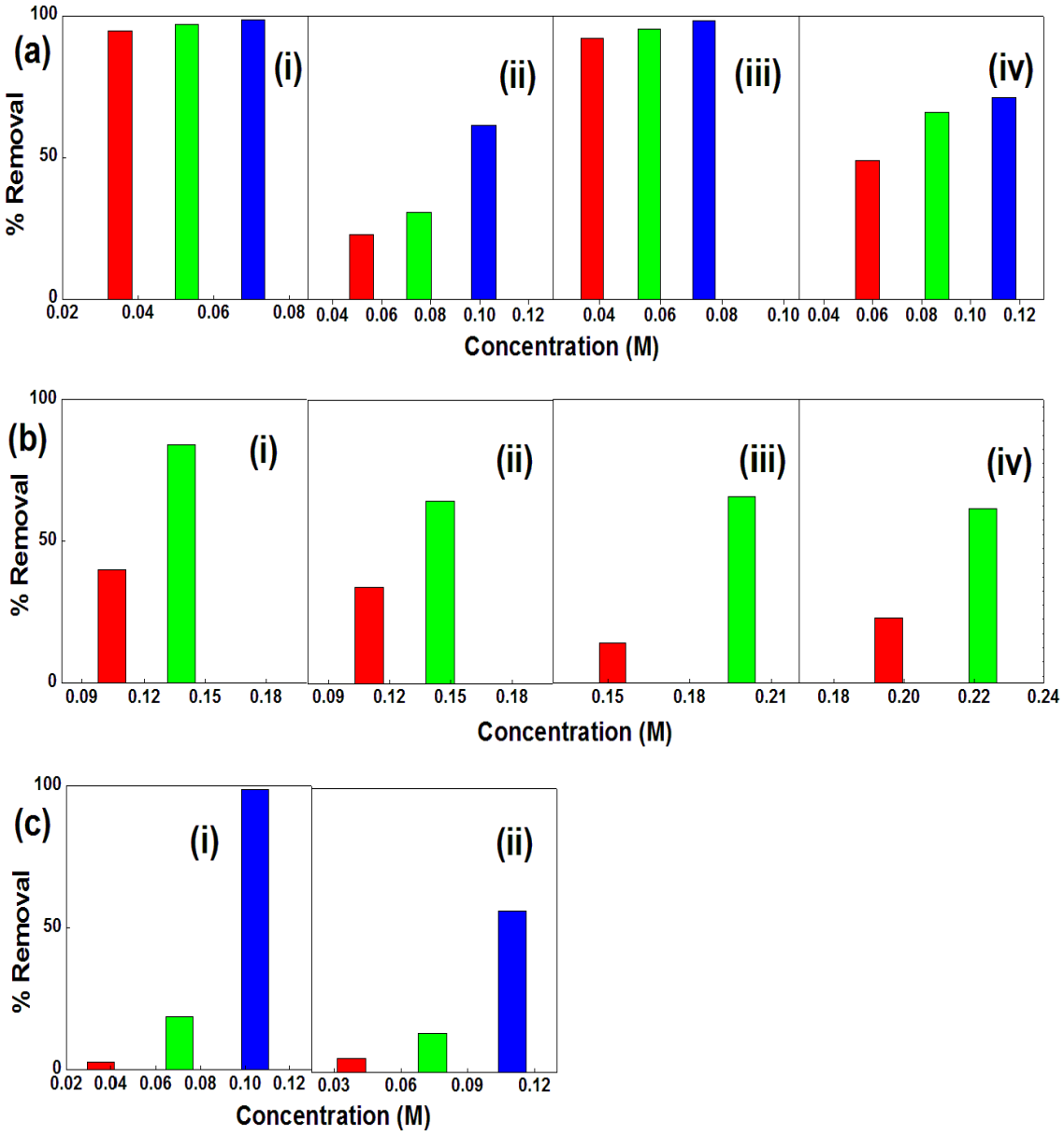


The mechanism of precipitation followed two steps [39-41]:

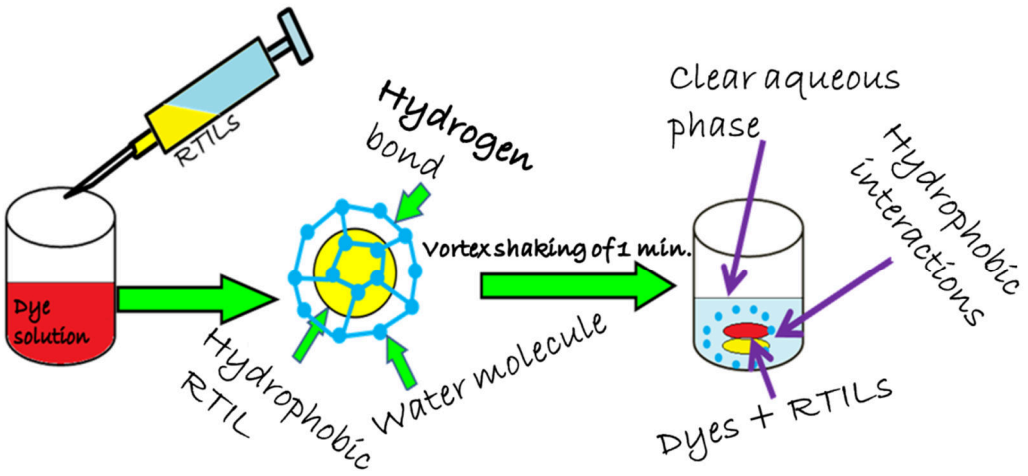
Before forming hydrophobic interactions: When dyes are added in water, these gets solubilize by making hydrogen bonds with water. However, when hydrophobic RTILs are added in aqueous solution of dyes, weak hydrogen bonding between dyes and water molecules started breaking down in order to make a space for hydrophobe. The whole reaction has been endothermic (change in enthalpy, ΔH is positive), as heat is required in breaking of bonds. The presence of the hydrophobic RTIL forced distorted water molecules to make new hydrogen bonds in order to have less contact with the hydrophobe

and hence water form an ice-like cage structure called a clathrate cage around the hydrophobe (scheme 1). The system becomes more ordered that leads to decrease in the entropy of the system; therefore ΔS becomes negative. ΔH of the whole system may be positive, negative or zero, depending on whether the new hydrogen bond making have completely, partially or over compensate the breaking of hydrogen bonds due to the addition of hydrophobe RTIL. However, in the mixing of hydrophobe and water molecules the entropy decrease become so large, that the sign of change in enthalpy ΔH is insignificant in deciding the spontaneity of reaction. Hence, according to Gibbs free energy formula $\Delta G = \Delta H - T\Delta S$, the value of ΔG is positive with a large negative ΔS and small ΔH. The mixing of hydrophobic RTIL with water was not spontaneous process.

Fig. 5. Plot of percentage removal of dyes using different concentration of (i) [BMIM][PF_6], (ii) [BMIM][BF_4] (iii) [EMIM][NTf_2] and (iv) CL for (a) Rh6G, color representation; red= 1%, green= 1.5% and blue= 2% of each RTIL (b) CR, color representation; red= 3%, green= 4% of each RTIL (c) CBB color representation; red= 1%, green= 2% and blue= 3% of each RTIL



Scheme 1. Schematic representation showing the extraction process



Formation of hydrophobic interactions: On vortex shaking of 1 minute, Rh6G having lipophilic character as well [42], CR due to more attraction towards hydrophobic component[43] and CBB having hydrophobic character, started interacting with hydrophobic RTIL via hydrophobic interactions. This process leads to increase in enthalpy (ΔH is positive), as due to hydrophobic interactions some of the bonds of clathrate cage broke down. Due to tearing of clathrate cage, the entropy of the system increases, making ΔS positive. Therefore, with a large positive ΔS and small positive ΔH, the value of ΔG is negative. Hence the hydrophobic interactions are spontaneous. Schematic representation showing the mechanism of dye precipitation is shown in scheme 1. So, the immiscibility of RTIL in the aqueous dye solution makes it efficient in the extraction of dye.

Effect of pH of aqueous solution of dyes

The effect of pH of aqueous solution of dyes on the extraction efficiency of RTILs, has also been studied. It was observed that, pH has no significant effect on the efficiency removal of hydrophilic RTILs; [BMIM][BF_4] and CL, however, on efficiency removal of hydrophobic RTILs, a slight effect of pH for all the dyes has been observed. Out of two used hydrophobic RTILs, [BMIM][PF_6] shows better extraction efficiency than [EMIM][NTf_2], therefore, the effect of pH of aqueous phase on the extraction efficiency was evaluated using [BMIM][PF_6] only. Figure 6 shows the plots of absorbance vs wavelength of supernatant left after the precipitation of dyes in [BMIM][PF_6] at different pH of aqueous phase. Effect of pH value on the percent removal of dyes by using [BMIM][PF_6] and relationship between pH value and $P_{RTIL/W}$ of dyes in [BMIM][PF_6]/aqueous solution is depicted in figure 7. Figures 6 (a) and 7 (a) confirm that Rh6G removal percentage is maximum i.e. 99.28 % in acidic medium with highest $P_{RTIL/W}$ of 7025.47. From these results, it was concluded that in acidic medium, Rh6G remains in its cationic form, due to which electrostatic forces comes into the picture leading to precipitation of Rh6G in RTIL[44]. Whereas, in basic medium as Rh6G remains in its non-ionic form [44], due to which there were no electrostatic forces between RTIL and Rh6G, hence less precipitation occurred (scheme 2). In the case of CR (Figures 6(b) and 7(b)), [BMIM][PF_6] shows its best percentage removal of 92% at pH = 12 having $P_{RTIL/W}$ value 306.86. This precipitation may be due to the reason that, around pH = 12, CR remains in its anionic form and prefers the IL phase, whereas in acidic medium, CR remains in its

zwitterionic form. Therefore, in whole extraction process, for neutral pH, hydrophobicity of RTIL plays its role (scheme 1), and in acidic or basic medium, much stronger electrostatic forces come into picture. Figures 6(c) and 7(c) confirmed that for CBB, [BMIM][PF_6] shows best percentage removal of 99.33 % at pH = 4 with highest $P_{RTIL/W}$ of 4978.70. Being a cationic dye, CBB will remain in its ionic form in acidic medium, and gets precipitate out due to electrostatic forces in [BMIM][PF_6]. Figures 6 and 7 confirm that pH has a very less effect on efficiency removal of [BMIM][PF_6] for all the studied dyes, which may be due to occurrence electrostatic forces, hence, it again supported that hydrophobicity of RTILs plays crucial role in the extraction of dyes.

Fig. 6. Plot of absorbance vs wavelength at different pH of aqueous phase showing precipitation of (a) Rh6G (b) CR (c) CBB in [BMIM][PF_6]

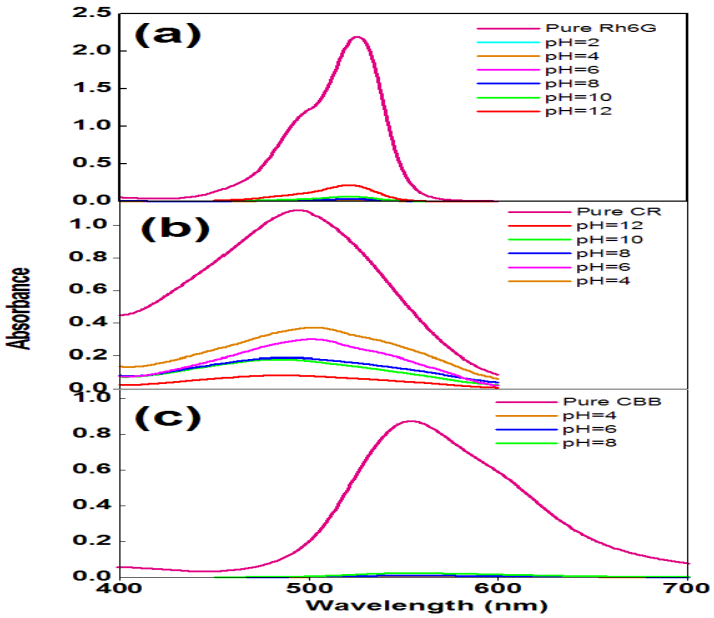
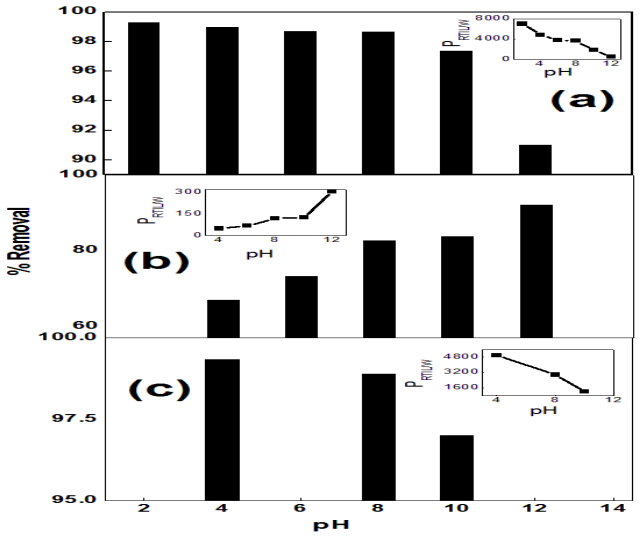


Fig. 7. Plot of pH value vs percentage removal of (a) Rh6G (b) CR (c) CBB by [BMIM][PF_6] (inset; pH value vs $P_{RTIL/W}$ of each dye in [BMIM][PF_6]/aqueous solution)



Removal of mixtures of dyes

We have also investigated the removal of dyes, if all of the four dyes present in one water sample. As observed, hydrophobic RTIL, [BMIM][PF_6] proved to be best extractant, so, we have made an attempt to precipitate mixture of dyes using 2 % of [BMIM][PF_6] from its 10 ml of 3×10^{-5} M aqueous solution. Figure 8 shows the UV-vis spectra showing the removal of dyes using only 2 % of [BMIM][PF_6] with extraction efficiency of 95.07 %.

Recovery and reuse of [BMIM][PF_6]

[BMIM][PF_6] can be separated from RTIL/dye precipitate by washing it with a solvent, in which one of the components of precipitate is soluble and other one is insoluble. A series of solvents were tried and 1-butanol was found to be best solvent for the recovery purpose. [BMIM][PF_6] remained insoluble, while dyes got solubilize in 1-butanol due to their preferential solubility in 1-butanol. In the case of Rh6G, we could achieve 100 % recovery of used [BMIM][PF_6]. Also, we have been able to separate 75.3 % of RTIL from [BMIM][PF_6]/CR precipitate and 87.5 % of RTIL from [BMIM][PF_6]/CBB precipitate by washing it with 1-butanol. The identity of recovered [BMIM][PF_6] was confirmed by comparing its FTIR spectra with spectra of pure [BMIM][PF_6] (figure 9), which were found to be identical. Recovered [BMIM][PF_6] was again used for the extraction of dye via same procedure, and again recovered. In addition to this, we have also verified the identity of precipitated dyes in [BMIM][PF_6]. UV-vis spectra of pure dyes in methanol as well as mixture of precipitated dye + [BMIM][PF_6] in methanol were observed (figure 10). Both UV-vis spectra were found to be same in case of each dye, which confirmed that identity of dyes remains same. This leads us to conclude that extraction process was due to hydrophobic interactions only.

Fig. 8. Absorbance vs wavelength showing 95.07 % removal of dyes

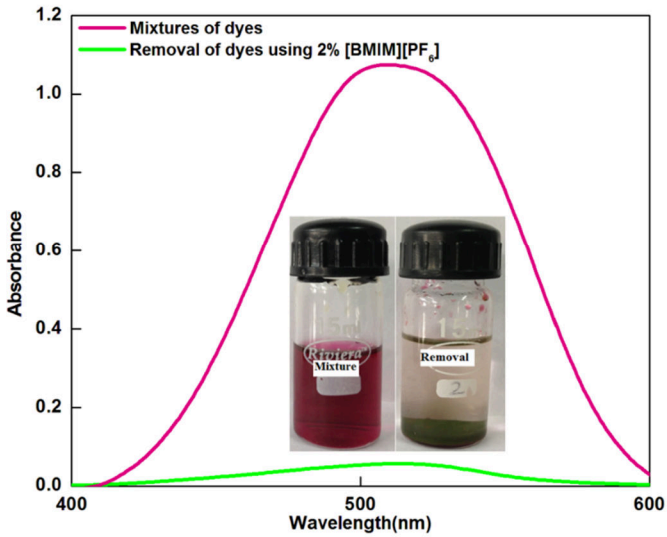


Fig. 9. Comparison of FT-IR spectra of pure [BMIM][PF_6] with recovered [BMIM][PF_6] from dyes

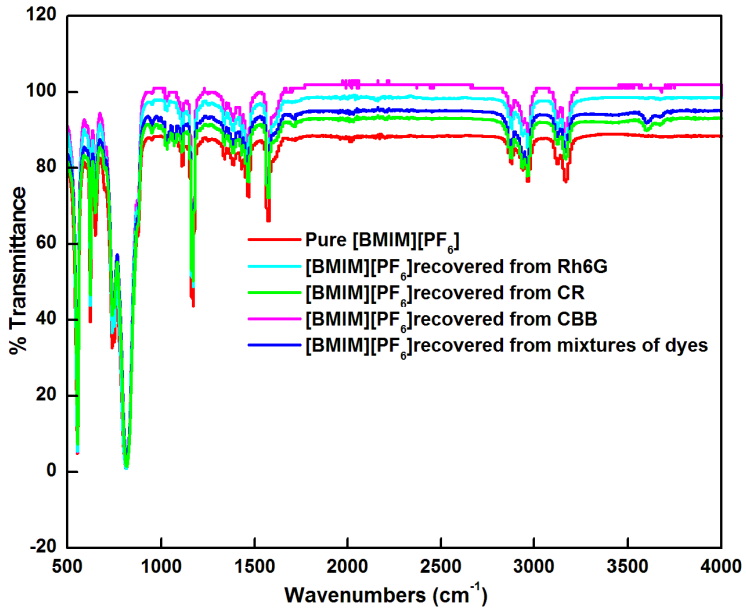
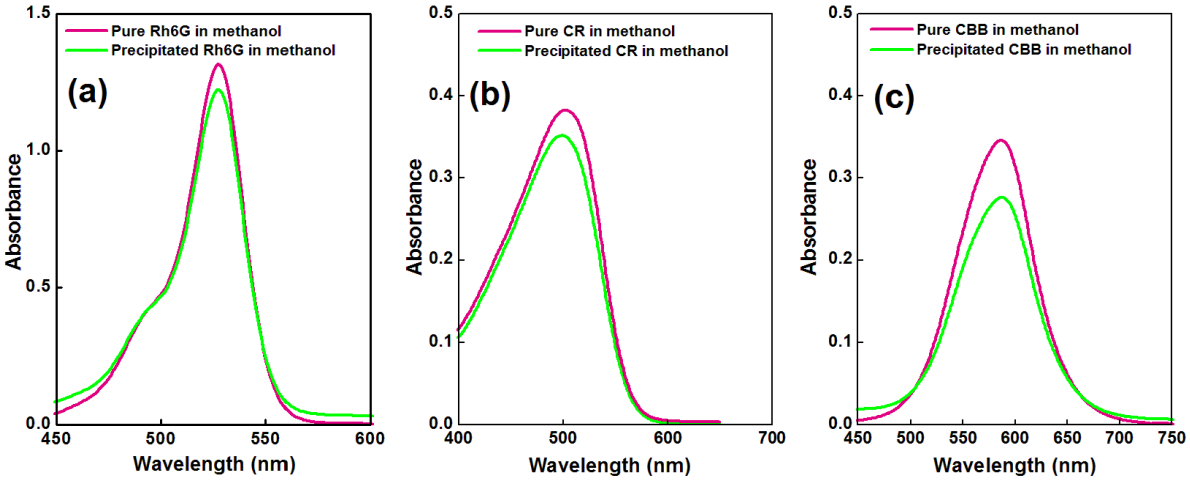


Fig. 10. Plot of absorbance vs wavelength for (a) Rh6G (b) CR (c) CBB in methanol and precipitated dyes + RTIL in methanol



CONCLUSIONS

Hydrophobic RTILs [BMIM][PF_6] and [EMIM][NTf_2] have proven to be best extractant for both cationic as well as anionic dyes in comparison to hydrophilic RTILs; [BMIM][BF_4] and [(C_2H_4OH)-$(CH_3)_3N$][Lactate]. We conclude that hydrophobicity is the key function of extraction processes. In the case of Rh6G, we have been successful in recycling almost 100 % of used [BMIM][PF_6] and reuse it in further extraction process.

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