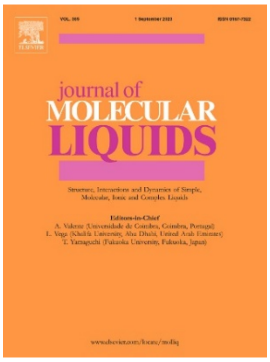
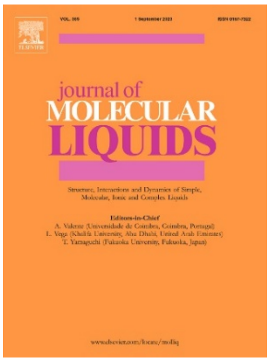
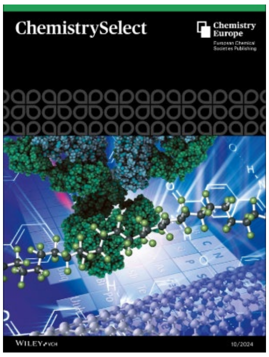
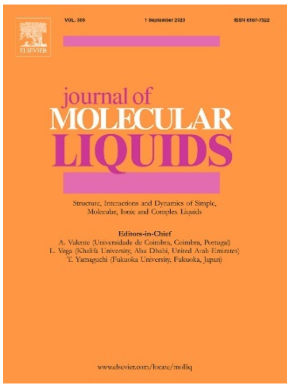
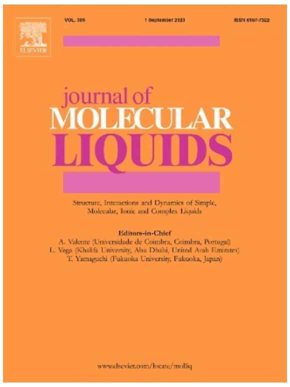
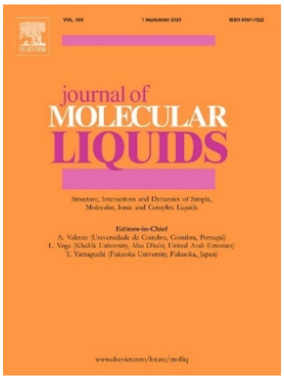
List of Publications & List of Conference Symposia/Seminars/ Workshops/Webinars

❖ List of Publications (Related to Thesis)

1.		**Title: Micellization of conventional and gemini surfactants in aquoline: A case of exclusively water based deep eutectic solvent** **Darshna Hirpara**, Brijesh Patel, Vishwajit Chavda, Sanjeev Kumar **Journal of Molecular Liquids**, Volume 362, p. 119672 **Available Online:** 24 June 2022 **DOI:** https://doi.org/10.1016/j.molliq.2022.119672
2.		**Title: Micellization and clouding behaviour of an ionic surfactant in a deep eutectic solvent: A case of the reline-water mixture** **Darshna Hirpara**, Brijesh Patel, Vishwajit Chavda, Arpita Desai, Sanjeev Kumar **Journal of Molecular Liquids**, Volume 364, p. 119991 **Available Online:** 2 August 2022 **DOI:** https://doi.org/10.1016/j.molliq.2022.119991
3.		**Title: Curcumin solubility enhancement in natural deep eutectic solvent mediated surfactant aggregates** **Darshna Hirpara**, Vishwajit Chavda, Nirbhay Hirapara, Sanjeev Kumar **ChemistrySelect,** (In peer review) **Manuscript ID: slct.202400130**

❖ List of Publications (Non-related to Thesis)

1.		**Title: Composition triggered Aggregation/Solubilization behaviour of mixed counter charged Gemini Surfactants: A Multi-technique investigations** Brijesh Patel, Sneha Singh, Kushan Parikh, Vishwajit Chavda, **Darshna Hirpara,** Debes Ray, Vinod K. Aswal, Sanjeev Kumar **Journal of Molecular Liquids**, Volume 359, p. 119242 **Available Online:** 28 April 2022 **DOI:** https://doi.org/10.1016/j.molliq.2022.119242
2.		**Title: GO/Ionic Surfactant Inspired Photophysical Modulation of Rhodamine B in Reline with or without Additives** Vishwajit Chavda, **Darshna Hirpara**, Sanjeev Kumar **Journal of Molecular Liquids**, Volume 368, p. 120614 **Available Online:** 18 October 2022 **DOI:** https://doi.org/10.1016/j.molliq.2022.120614
3.		**Title: A Sustainable Approach for the Adsorption of Methylene Blue from Aqueous Background: Adsorbent Based On DES/CGS Modified GO@ZrO$_2$** Vishwajit Chavda, Brijesh Patel, Sneha Singh, **Darshna Hirpara**, V. Devi Rajeswari, Sanjeev Kumar **RSC Sustainability,** Issue 1, p. 2038-2057 **Available Online:** 05 October 2023 **DOI:** https://doi.org/10.1039/D3SU00236E

4.		**Title: Deep Eutectic Solvent/Surfactant-Engineered GO@TiO2 Advanced Material for Sustained and Ultrafast Adsorption of Hazardous Dye from Contaminated Aqueous Environment** Vishwajit Chavda, **Darshna Hirpara**, Vandana Rao, Sanjeev Kumar **ACS Applied Engineering Materials** **Available Online:** 08 January 2024 **DOI:** https://doi.org/10.1021/acsaenm.3c00696
1.		**Review** **Title: Exploring the potential of deep eutectic solvents in pharmaceuticals: Challenges and opportunities** Priyanka A. Shah, Vishwajit Chavda, **Darshna Hirpara**, Vinay S. Sharma, Pranav S. Shrivastav, Sanjeev Kumar **Journal of Molecular Liquids,** Volume 390, p. 123171 **Available Online:** 15 November 2023 **DOI:** https://doi.org/10.1016/j.molliq.2023.123171

❖ Work presented in Conferences/ Seminars/ Workshops

Conferences		
1.		**Title: Micellization and Clouding Phenomenon in Reline-Water Mixture: A case of Anionic surfactant** **Darshna Hirpara,** Sanjeev Kumar **National Conference** on *Recent Advances and Future Trends in Biological, Chemical and Physical Science 2021 (RAFTBCPS-2021)*, **30th -31st July 2021**. **Presented Session: POSTER**
2.		**Title: Clouding and Liquid-Liquid phase Separation in Deep Eutectic Solvent: A case of an Anionic Surfactant** **Darshna Hirpara,** Sanjeev Kumar **International Conference** on *Complex Fluids and Soft Matter 2021 (Compflu-2021)*, **13th -15th December 2021**. **Presented Session: ORAL**
3.		**Title: Micellization Behavior of An Anionic Surfactant in a "Designer Solvent": A Case of Deep Eutectic Solvent** **Darshna Hirpara,** Sanjeev Kumar **International Conference** on *Molecules to Materials (MTM – 2021)*, **17th -18th December 2021.** **Presented Session: POSTER**
4.		**Title: Physico-chemical Studies on Deep Eutectic Solvent: A Case of Reline-water System** **Darshna Hirpara,** A.N. Prajapati, Sanjeev Kumar **International Conference** on *Advanced Materials and Applications (ISAMA-2022)*, **18th July 2022.** **Presented Session: ORAL** Received 1st Rank for best oral presentation

5.		**Title: Association Behaviour of an Ionic Surfactants in Deep Eutectic Solvent and its Components** **Darshna Hirpara,** Sanjeev Kumar **National Conference** on *Modern material and chemical sciences (MEMCS-23),* **6th -7th January 2023.** **Presented Session: ORAL**
6.		**Title: Curcumin and its precursor (Curcuma Longa) solubilization in Conventional and Deep Eutectic Solvents with and without ionic surfactants** **Darshna Hirpara,** Sanjeev Kumar *30th CRSI-NSC & 16th CRSI-RSC* **Symposium Series** *in Chemistry,* **2nd-6th February 2023.** **Presented Session: POSTER**
7.		**PARTICIPATED** **Darshna Hirpara,** Sanjeev Kumar **International Conference** *on Surface Chemistry: Colloids and Interface Aspects with Applications (SCCIA-2022),* **3rd-7th January 2022.**
Workshops		
8.		**Darshna Hirpara** **5-Days Faculty Development Programme** *Accelerating Innovations in Material Science - Surface Characterization,* **18th to 22nd May 2021.**

9.		**Darshna Hirpara** **7-Days Training program** *Synergistic Training program Utilizing the Scientific and Technological Infrastructure* ***(STUTI)*** on Modern Spectroscopic, Thermal and Microscopic Techniques,**21st -27th September 2022.**
10.		**Darshna Hirpara** **One-Day workshop** Early Career Researchers by Inspiring India in Research, Innovation, and STEM Education (iRISE*) on* *IP and Knowledge Management*, **28th September 2022.**
11.		**Darshna Hirpara** **2-Days Training program** Hands-on Workshop *on* *Advanced Characterization of Nanomaterials*, **23rd-24th September 2023.**

Published Research Articles

Journal of Molecular Liquids 362 (2022) 119672

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Micellization of conventional and gemini surfactants in aquoline: A case of exclusively water based deep eutectic solvent

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Fluorescence quenching

A B S T R A C T

Deep eutectic solvents (DES) based on water as the only hydrogen bond donor (HBD) have been introduced recently (*aquoline*). There is not a single report on surfactant association behaviour in aquoline. This study deals with the determination of physical properties (rheology, specific conductance, pH, micro polarity and apparent dielectric constant) of pure aquoline (with different molar ratios of choline chloride (ChCl): water, DES I - DES IV) and association behaviour of ionic surfactants in such aquoline systems. DESs were found Newtonian and highly polar / conducting fluids with nearly neutral pH (6.9–7.1). Micellization behaviour of different ionic surfactants (sodium dodecyl sulphate (SDS), sodium dodecane-1-sulphonate (SDSo), sodium dodecyl benzene sulphonates (SDBS), P, P'-1,4-butanedieyl, P, P'-didodecylester, disodium salt (12-4-12A), cetyltrimethylammonium ammonium bromide (CTAB) or dodecyl trimethyl ammonium bromide (DTAB)) has been studied fluorometrically in various aquolines and compare data with an aqueous medium. In most of the cases, critical micelle concentration (CMC) values have been found lower than water which may be due to the presence of choline chloride (ChCl, one of the components of aquoline), which can interact micelle electrostatically and hydrophobically. For different head groups in anionic surfactant, CMC follows the order 12-4-12A < SDBS < SDS < SDSo, which fits in the Hoffmeister like series of head groups. For cationic surfactants (CTAB and DTAB), CMC shows a similar chain length effect as observed in water. Overall, CMC data allow to propose that CMC decreases with an increase in molar content of water in a typical aquoline under eutectic limit. Micellar aggregation number (Nm), Stern-Volmer constant, micellar polarity, and apparent dielectric constant were also computed. It has been observed that micelles of lower Nm, with high polarity, are formed in a typical aquoline system (DES III). The study may find applications where organized assemblies are required in highly polar solvents.

1. Introduction

About two decades back, a new designer solvent (deep eutectic solvent, DES) was introduced in the scientific literature [1]. DES can be obtained from a eutectic mixture of at least one solid and another solid/liquid component at ambient temperature [2–4]. A continuous interest has been shown by the scientific world in synthesizing and characterizing DESs as there are reports to propose them as the potential solvent of the current century [5,6]. The interest was also shown due to their cost effectiveness, an abundance of the precursors and involvement of environmentally friendly components (owing to the demand of biocompatibility). A new classification has been proposed to categorise DES of type-III: hydrophobic DES and hydrophilic DES [7]. Interaction among DES components imparts high viscosity, low volatility and low conductivity, which can be controlled by nature and composition of individual components [5]. The viscous nature of DESs and their ill-defined structures are serious problems which can affect their applicability in various fields [8]. These shortcomings can be solved by mixing controlled proportion of water (below the DES limit) which can modify various interactions among the components [9]. In a recent report, it has been observed that addition of water to hydrophobic DES resulted in an acidic solvent system which causes an undesirability for synthetic organic medium and related applications [7]. For hydrophilic DESs, water has been proposed as a magical additive in order to search more potential and benign

Abbreviations: DES, Deep eutectic solvent; CMC, Critical micelle concentration; HBD, Hydrogen bond donor; Ch^+, Cholinium ion.

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Micellization and clouding behaviour of an ionic surfactant in a deep eutectic solvent: A case of the reline-water mixture

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Cloud point
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ABSTRACT

The micellization and clouding phenomenon are studied across a wide range of reline (Choline chloride (ChCl)-Urea, 1:2 mol ratio) - water composition using fluorescence spectroscopy. Experiments have also been performed to determine the physical properties (specific conductance (κ) and zero-shear viscosity (η_0)) of water in reline and reline in water. κ and η_0 vary in opposite ways as water composition of the water in reline decreases. Further, pure reline and the reline-water mixtures have been found Newtonian in nature. Critical micelle concentration (CMC) data of Sodium dodecylsulphate (SDS, an anionic surfactant) suggest three regions of CMC variations with water content in the water in reline and reline in water mixtures. Reline-urea-water motifs (H-bonded) or molecular solutions of components (ChCl and urea) dictate CMC in water in reline/reline in water region. Cloud point (CP) data was acquired using SDS + tetra *n*-butyl ammonium bromide (TBAB) in water in reline or reline in water and compared with pure water. CP plots are constructed with respect to [SDS] or [TBAB]. It has been noted that the increase in CP is directly dependent on [SDS] and reversely on [TBAB]. A clouding mechanism for SDS (+TBAB) in reline -water mixture has been interpreted on the basis of competition between tetra-*n*-butylammonium (TBA^+) and cholinium (Ch^+) counter ion for the micellar surface. CP data have also been acquired in the presence of metal salt (cadmium chloride, $CdCl_2$ or zinc sulphate, $ZnSO_4$). These two salts affect CP oppositely, which is explained on the basis of hydrating capacity of metal ions. SDS + TBAB + reline + water form a system that can be potentially used for biphasic extraction (LLPS) of various metals from waste streams or battery waste.

1. Introduction

Deep Eutectic Solvents (DESs) are usually obtained via H-bond exchange between hydrogen bond donor (HBD) and hydrogen bond acceptor (HBA) in the eutectic range [1]. Initially, it was thought that DES is only one kind of ionic liquid [2]. However, later researches show that DES has the potential to act as a solvent of the 21st century [3]. Within the available DESs, choline chloride (ChCl) containing DESs is a particular class and is widely explored due to their desirable properties (nontoxic, biodegradable, biocompatible, nonflammability, etc.) and economy [4]. Among ChCl-based DESs, reline (ChCl: Urea, 1:2 mol ratio) has been used in various applications [5,6]. In infancy time, the general belief was that DES should be strictly anhydrous for an application point of view. However, the full potential of reline could not be realized due to its higher viscosity which poses a processing problem during its transfer. The problem has been resolved by heating [7] or by adding water [8]. An upper boundary of hydration was demarcated (up to which it behaves as water in DES) over that system behaves molecular solution of DES components (DES in water). Water has unique characteristics owing to strong H-bonding interaction [9]. When water is deliberately mixed with DESs, the resulting system can act as a functional fluid with improved properties and higher fluidity. This may be due to the alteration of the hydrogen bond network of reline [10]. Recently, it has been proposed that water can act as an additional HBD which results in a newer DES with additional properties over its precursor DES [11].

The nano-structure of DES, with or without water, can influence aggregation/association behaviour of surface-active agents

Abbreviations: DES, Deep eutectic solvent; CP, Cloud point; LLPS, Liquid-liquid phase separation; CPEM, Cloud point extraction methodology; CMC, Critical micelle concentration; Ch^+, Cholinium ion; TBA^+, *n*-tetra butyl ammonium ion.

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Composition triggered Aggregation/Solubilization behaviour of mixed counter charged gemini Surfactants: A Multi-technique investigations

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ABSTRACT

Aqueous association behaviour of counter charged gemini surfactants has been studied by fluorometry, dynamic light scattering (DLS), Zeta(ζ)-potential and SANS measurements at 303 K. For the purpose, P, P'-1,4-butanedieyl, P, P'-didodecylester, disodium salt, anionic surfactant(12–4-12A) and cationic gemini surfactants: Butanediyl-1,4-bis (N, N-dimethyl-N-tetradecyl-ammonium) dibromide (14–4–14); Ethane-1,2-diyl bis (N, N-dimethyl-N-tetradecyl-ammonium acetoxy) dichloride (14-Eg-14) and (D-isosorbate-1,4-diyl bis (N, N-dimethyl-N-tetradecylammonium acetoxy) dichloride (14-Isb-14) were mixed for varying mole fraction range (x = 0–1). Fluorescence data using pyrene as a probe are used to obtain CMC values which were theoretically treated using regular solution theories. It has been observed that mixing causes non spherical micelles and even vesicle formation was observed in one of the combinations (12–4-12A + 14–4–14) at x = 0.4. Various compositions are used to solubilize polycyclic aromatic hydrocarbon (pyrene, anthracene and phenanthrene) in order to have an idea of solubility enhancement efficacy. Micellar morphology/environment has been used to draw a correlation between apparent dielectric constant (D_{exp}) - composition - solubilization potential. Findings can be used for loading various hydrophobic materials in an appropriate amphiphilic mixture for various applications such as dye solubilization, drug solubilization, drug delivery or drug targeting.

1. Introduction

Organised assemblies resulted from amphiphilic molecules (surfactants, ionic liquids, polymers, drugs, biomolecules etc.,) play an important part in physical and life sciences [1]. To achieve the requirement of the application, different strategies are adopted to search an optimal system with or without (single) combination of more than one component [2–4]. Such strategies are namely architectural changes (chain length, nature of spacer, nature of charge of amphiphile/counter ion etc.),experimental conditions (temperature, pH, additives etc.,) and mixing components/composition [5–13]. Mixing of components (surfactants), in place of single surfactant seems an effect mode to regulate/boost synergistic properties of aqueous organised assemblies. Among the above procedures, mixing of surfactant components is an effective and easy way to get morphologies of desired architecture only by varying the mixing ratio [14–17]. Special effort has been directed towards the obtaining vesicles, since mixing of amphiphiles of counter charges resulted in thermodynamically stable aggregates [2,18].

Gemini surfactant (represented as m-s-m, m and s are carbon numbers in hydrocarbon chain and spacer groups) has been introduced as having potential to work as the surfactant of the 21st century [5,13,19,20]. In last decade, positively charged gemini surfactant has been continuously utilise as one of the members of the mixture, which resulted in mixed micellization and boosted solubilisation efficacies [21–23]. Recently, even mixing of block copolymers(non-ionic components) modifies association phenomenon and drug solubilisation [24]. However, the full potential of mixing gemini surfactants at the application front is yet to be fully exploited [25–28]. Gemini surfactants provide rich functionality due to the presence of spacer group which could be solvophilic or solvophobic, rigid or semi-rigid, lengthy or smaller [29]. The simultaneous presence of gemini surfactants of counter-charges has only been investigated scarcely, and data available are not sufficient to have a good database for full utilisation in the field of bio-

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GO/ionic surfactant inspired photophysical modulation of rhodamine B in Reline with or without additives

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ABSTRACT

Photophysical behaviour of rhodamine B (RB) in deep eutectic solvents (DES, formed by quaternary ammonium salt and hydrogen bond donor (HBD) in a specific eutectic ratio) with or without graphene oxide (GO) or ionic surfactants, is less known. The nature of Reline (choline chloride (ChCl): urea (HBD), 1:2), a well-known DES, has been designed by adding glycerol or water as the second HBD for sustained movement of RB. Effects of GO, surfactant, or GO + surfactant, in controlling RB movement, at various sites (GO surface, surfactant micelle, DES surface, or background solvent), have been fluorometrically reported. The basic nature of Reline (pH = 10.38) causes modification of GO surface (deprotonated site) and nature of RB (cationic → zwitter ionic). Above Reline-inspired changes have been found to modify interactions of RB with GO and/or sodium dodecyl sulphate (SDS, an anionic surfactant) or cetyl trimethyl ammonium bromide (CTAB, a cationic surfactant). SDS (10 mMdm^{-3}, < critical micelle concentration (CMC) in Reline) shows ~ 2.6, 1.6, and 1.4 fold fluorescence intensity enhancement of RB (in water, pure Reline, and methanol, respectively). However, GO and/or CTAB shows quenching behaviour. Further, the fluorescence of RB shows weak dependence on changing the second HBD (water or glycerol). DES-controlled cationic *vs* zwitterionic form of RB is responsible for the interaction and sustained movement towards GO surface, micellar surface, or negatively charged ion-pair formation (with SDS monomers). Findings of the work have implications in searching potential fluorescent levels/sensors for photophysics, photobiology, or wider vehicle means for sustained drug delivery.

1. Introduction

In the last decades, fluorescent chemo-sensors have been preferred over other conventional analytical tools due to their better sensitivity and selectivity [1-3]. Rhodamine dyes (Rhodamine B (RB) and derivatives) have been widely used in single-molecule detection, fluorescence leveling, or for DNA sequencing [4,5]. The choice of solvent medium and dye concentration is a prerequisite for the specific use. In the past, the majority of the work addressed individual solvent effects with a limited concentration range [6-8]. In a later study, it has been reported that solvent polarity affects the linear and non-linear properties of the chromophores [9]. The photophysical properties of rhodamine dyes made them important members of laser dyes [10]. The association behaviour of ionic dyes is distinctly influenced by several factors such as concentration, pH, temperature, and nature of the medium [11-14]. In a few studies, it has been shown that solvent nature governs the photophysical process shown by fluorescent molecules with or without nanomaterials of the graphene family [14-17]. Therefore, a wider window exists to investigate the photophysics of the systems involving carbon-based nanomaterial, dye molecules, and solvents with a mechanism of fluorescence modulation.

In light of the above facts, the role of solvent is decisive in molecular-level interactions of chromophores with biomolecules [18]. Recently, a new solvent system based on the process of the exchange of hydrogen bonds between hydrogen bond donor (HBD) and hydrogen bond acceptor (HBA) has been introduced as the solvent of the 21st century [19]. These solvent systems are greener and more benign and popularly known as '*Deep Eutectic Solvent*' (DES) [20-22]. DESs have many characteristics analogous to conventional ionic liquids (ILs) [22]. Since ILs are reported to form mixed micelles with surfactants, one can expect DES-inspired modification of the solution behaviour of a surfactant

Abbreviations: DES, Deep eutectic solvent; GO, Graphene oxide; HBD, Hydrogen bond donor; RB, Rhodamine B; CMC, Critical micelle concentration.

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A sustainable approach for the adsorption of methylene blue from an aqueous background: an adsorbent based on DES/CGS modified GO@ZrO_2†

Vishwajit Chavda,[a] Brijesh Patel,[a] Sneha Singh,[a] Darshna Hirpara,[a] V. Devi Rajeswari[b] and Sanjeev Kumar *[a]

Gemini surfactants (GSs) and deep eutectic solvents (DESs) belong to two important classes of industrially important materials which can be used to modify the performances of other entities where they are used for functionalization. A graphene oxide-zirconium oxide (GO@ZrO_2) nanocomposite has been synthesised and modified by using a cationic gemini surfactant (CGS, butanediyl-1,4, bis(*N*,*N*-hexadecyl ammonium) dibromide (16-4-16)) or by using a well-known DES (reline, choline chloride : urea, molar ratio 1 : 2). The adsorbent materials were characterized by various physicochemical techniques (FTIR, XRD, TEM, SEM-EDX, and TGA). Methylene blue (MB), a well-known industrially important colouring material, has been used as a model adsorbate to investigate its adsorption/removal from aqueous solution by using the above-modified nanocomposites (NCs, CGS-GO@ZrO_2 and DES-GO@ZrO_2). The adsorption process follows the Langmuir model ($R^2 \approx 0.995$) together with *pseudo*-second order rate kinetics. Adsorption variables were optimised in the light of [NC], [MB], pH, and contact time. DES-GO@ZrO_2 has been found to be a better candidate for the fast removal of MB (~100% at 20 mg L^{-1}, 5 m with 2 mg ml^{-1} DES-GO@ZrO_2) when compared with other similarly modified materials. To economize the method, desorption of adsorbed MB (performed by using ethanol) is necessary. It has been found that the DES-GO@ZrO_2 performs efficiently even after 5 adsorption–desorption series. The findings of the present study can have potential applications in developing an economic strategy for the purification of industrial dye effluents with a concomitant redressal of aquatic pollution.

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Sustainability spotlight

Industrial effluents containing colour and colouring materials pose a significant threat to the aquatic ecosystem, with dyes from textile industries being a major contributor. To save running water sources from contamination, it is desirable to search for effective strategies for removing dyes from industrial wastewater. In response to this challenge, a sustainable and economically viable answer emerges in the form of modified graphene-based composites. This innovative idea not only offers valuable insights but also presents a practical solution for mitigating the harmful effects of dye effluents. By adopting the methodology presented here, one can actively conserve the already present limited potable water resources on mother earth.

1 Introduction

The last 4–5 decades witnessed a sharp increase in the fundamental progress and prospects of various kinds of materials such as nanomaterials, electronic materials, solvent materials, associated materials, membrane materials, and porous materials among others.[1–11] Among these materials, carbon allotropes attract special attention due to their novel properties and potential application in various fields of life.[12–16] Graphene is the most sought-after carbon allotrope, both in its pure form and when incorporated into composite materials or utilized in its derived forms, in various areas of scientific and engineering research.[4,17–20] Graphene is a uni-layer bi-dimensional surface of carbon atoms chemically bonded in the sp^2 configuration with a hexagonal pattern (benzene ring).[17,18] However, graphene oxide (GO) is preferred over graphene due to the presence of functional groups, though graphene has exceptional mechanical, electrical, and thermal properties.[21]

GO spontaneously distributes in an aqueous medium facilitating polluted water treatment.[22,23] Furthermore, GO shows high electronic mobility imparted from oxygenated moieties at the basal plane and edges.[24] However, high surface energy results in agglomeration and lower dispersibility as well as

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Deep Eutectic Solvent/Surfactant-Engineered GO@TiO_2 Advanced Material for Sustained and Ultrafast Adsorption of Hazardous Dye from Contaminated Aqueous Environment

Vishwajit Chavda, Darshna Hirpara, Vandana Rao, and Sanjeev Kumar*

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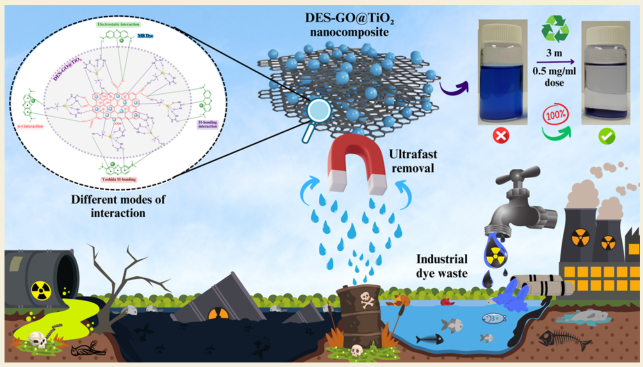

ABSTRACT: Hybrid carbon-based materials are in demand to achieve the desired activities in various fields. However, such materials lack aqueous dispersibility or mechanical strength, which can be addressed by certain modifiers such as surfactants, polymers, metal oxides, and deep eutectic solvents (DESs), among others. Dodecyl trimethylammonium bromide (DTAB) and a typical DES (reline; choline chloride/urea, 1:2) have been employed in conjunction with graphene oxide−titanium oxide nanocomposites (GO@TiO_2 NCs) to get advanced adsorbents (which are characterized by various physicochemical techniques). A model hazardous dye, methylene blue (MB), was used to study the adsorption potential of the above-developed material. The process was optimized in terms of the adsorbent dose, initial MB concentration, pH, and contact time. Various kinetic and adsorption isotherm models were used to analyze the data. Pseudo-second-order kinetics (R^2 = 0.999) has been followed by the Langmuir adsorption isotherm (R^2 = 0.998). Kinetics revealed that 100% MB adsorption was achieved with DES-based NC (DES-GO@TiO_2), which is better than that with both DTAB-based (DT-GO@TiO_2) and pure pristine material (GO@TiO_2). It is proposed that DES (reline) couples TiO_2 on the GO surface with an alternative route to drive MB from an aqueous background. Adsorption data were compared with other similar reported adsorbents, and it was found that the developed DES-based advanced material shows ultrafast MB adsorption (the rate of adsorption has been found to be 222 μg MB per gram of DES-GO@TiO_2 in 1 s). The sustainability and economy of the adsorbent were revealed by repeating numerous adsorption cycles (up to seven times) without losing the adsorption efficiency. The study can be applied in various chemical industries where color or coloring material is involved in the effluent. Further, other similar greener DESs should be used in order to develop sustainable structure−performance relationship for a safer environment.

KEYWORDS: *adsorption, graphene oxide, deep eutectic solvent, surfactant, methylene blue, wastewater treatment*

1. INTRODUCTION

Extensive industrialization and urban development are the main factors behind wastewater discharge, leading to significant water pollution. One significant issue is the release of dyes into water bodies, which pose threats to terrestrial/aquatic life. The disposal of effluents containing used dyes into water bodies is a significant contributor to water pollution. Treatment is crucial for mitigating the harmful effects of dye discharged into wastewater before disposal. However, dye removal remains a challenging problem because even a small amount of dye can lead to health issues, such as allergic reactions, skin irritation, and cancer. Various techniques, including physical, photocatalysis, electrochemical, chemical, adsorption, and biological treatments, have been employed to address this issue. Among these, adsorption stands out as the most effective method because of its simplicity, cost-effectiveness, and versatility in selecting and modifying adsorbent materials. Moreover, it generates no harmful byproducts and can be effectively applied to treat large volumes of water.[1] Various analytical instruments, including gas chromatography−mass spectrometry (GC−MS), liquid chromatography (LC)−MS, and high-performance liquid chromatography with diode-array detection (HPLC-DAD), have been established for dye detection. While these hyphenated techniques offer rapid and precise results,[2] they pose drawbacks such as high cost, complex instrumentation, and the use of organic reagents. Therefore, sustainability demands a method that delivers a fast, accurate, and cost-

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Exploring the potential of deep eutectic solvents in pharmaceuticals: Challenges and opportunities

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ABSTRACT

Deep eutectic solvents (DESs) have gained significant attention over the past twenty years due to their versatile properties and easy preparation methods without the need for extensive purification. DESs show promise as biocompatible options for pharmaceutical applications, particularly in enhancing solubility, stability, and serving as potential drug delivery systems for active pharmaceutical ingredients (APIs). Understanding the behavior of

Abbreviations: 12-4-12A, P, P'-1,4-butanedieyl, P, P'-didodecylester, disodium salt; ACS, american chemical society; ADES, acidic deep eutectic solvent; ADMET, absorption, distribution, metabolism, excretion, and toxicity; AMPA, alkyl methyl phosphonic acids; API, active pharmaceutical ingredients; BADES, bronsted acidic deep eutectic solvent; BCS, biopharmaceutical classification system; LMW, low molecular weight; BDES, alkaline/basic deep eutectic solvent; BE, back-extraction; BSA, bovine serum albumin; CAGE, choline geranate; CD, carbon dots; CD, cyclodextrin; ChAc, choline acetate; ChCl, choline chloride; CMC, carboxy methylcellulose; CMC, critical micelle concentration; CMPA, cyclohexyl methyl phosphonic acids; CPP, critical packing parameter; CTAB, cetyltrimethylammonium ammonium bromide; DEHP, di(2-ethylhexyl) phosphate; DEHPi, di(2-ethylhexyl) phosphite; DEM, deep eutectic monomer; DES, deep eutectic solvent; DLLME, dispersive liquid-liquid microextraction; DMPEG, poly(ethylene glycol) dimethyl ether; DSC, differential scanning calorimetry; DTAB, dodecyl trimethyl ammonium bromide; ee, enantiomeric excess; EME, electro membrane extraction; EMPA, ethyl methyl phosphonic acids; EPA, environmental protection agency; ES, eutectic solvent; GA, glycolic acid; GA, graphene aerogel; GCE, glassy carbon electrode; GCIPR, green chemistry institute pharmaceutical roundtable; GMS, glycerol monostearate; HaCaT, human immortalized epidermal cells; HBA, hydrogen bond acceptor; HBD, hydrogen bond donor; HC-MOF, hollow core microstructure optical fibers; HF-LPME, hollow-fiber liquid-phase microextraction; HME, hot melt extrusion; HMW, high molecular weight; HPBDES/HDES, hydrophobic deep eutectic solvent; HP-β-CD, 2-hydroxypropyl β-cyclodextrin; HPLDES, hydrophilic deep eutectic solvent; HPMCAS, hydroxy propyl methyl cellulose acetate succinate; iBMPA, isobutyl methyl phosphonic acids; IBU, ibuprofen; IL, ionic liquids; IPF, idiopathic pulmonary fibrosis; iPrMPA, isopropyl methyl phosphonic acids; LA, lauric acid; MA, myristic acid; LacA, lactic acid; LADES, Lewis acidic deep eutectic solvent; LC/MS, liquid chromatographymass spectrometry; LCFA, long-chain fatty acids; LevA, levulinic acid; Lys, lysine; MAA, methacrylic acid; MBA, methylamino butyric acid; MC, methyl coumarin; MDES, magnetic deep eutectic solvent; ME, microemulsions; MLC, micellar liquid chromatography; MoN, molybdenum nitride; mp, melting point; MRM, multiple reaction monitoring; MRSA, methicillin-resistant staphylococcus aureus; MTX, methotrexate; NAC, N-acetylcysteine; NADES, natural deep eutectic solvents; NaDES, non-aqueous deep eutectic solvent; NDMA, n-nitroso dimethylamine; NMR, nuclear magnetic resonance; NPOE, 2-nitrophenyl octyl ether; NSAID, nonsteroidal anti-inflammatory drugs; o/w, oil-in-water; OMT, oxymatrine; P123, poly(ethylene glycol)-block-poly(propylene glycol)- block poly(ethylene glycol); PAA, poly-acrylic acid; PALME, parallel artificial liquid membrane; PCA, principal component analysis; PCL, polycaprolactone; PDES, polymerized deep eutectic solvent; PEDES, polymer-embedded deep eutectic system; PEG, polyethylene glycol; PI, precipitation inhibitor; PIL, protic ionic liquid; RTIL, room temperature ionic liquid; PMPA, pinacolyl methyl phosphonic acids; PPG-NH2, poly(propylene glycol)bis(2- aminopropyl ether); PQES, poly-quasi eutectic solvent; PVA, poly-vinyl alcohol; PVP, polyvinyl pyrrolidone K30; PZA, pyrazin-amide; QD, quantum dots; QUE, quercetin; CA, capric acid; SANS, Small-angle neutron scattering; SCF, supercritical fluid; SDDS, supersaturating drug delivery system; SDME, single-drop microextraction; SDS, sodium dodecyl sulfate; SFODME, solidified floating organic drop microextraction; SLE, solid-liquid equilibrium; SLM, supported liquid membrane; SLPD, solid-liquid phase diagram; SUPRADES, supramolecular deep eutectic solvent; TBAB, tetrabutylammonium bromide; T_c, cold recrystallization; TCH, tetrahydro curcumin; T_f, freezing point; TGA, thermogravimetric analysis; Th/Thy, thymol; Da, decanoic acid; THEDES, therapeutic deep eutectic solvent; TIC, total ion current; T_m, melting temperature; TMG, trimethyl glycine; TM-β-CD, heptakis (2,3,6-tri-O-methyl)-β-cyclodextrin; T_{onset}, loss weight; VOS, volatile organic solvent; TSE, twin-screw extruder; UHPLC/MS, ultra-high performance liquid chromatography-mass spectroscopy; UNIQUAC, universal quasi-chemical; USAEME, ultrasound-assisted emulsification-microextraction; VOC, volatile organic compound; NRTL, non-random two-liquid theory; w/o, water-in-oil; WDES, water based deep eutectic solvent; WiS, water-in-salt.

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