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Unlocking the potential of recycled waste cooking oil for a sustainable volatile organic compound absorption process

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ABSTRACT

Finding suitable absorbents to remove volatile organic compounds (VOCs) from industrial polluted air remains a pressing scientific and industrial challenge. Most VOCs are hydrophobic and water demonstrates limited mass transfer in this case. In this study, we evaluated the feasibility of using recycled waste cooking oil (RWCO) to absorb VOCs for the first time. We compared the performance of RWCO with that of two deep eutectic solvents (DESs), including a hydrophobic DES prepared from decanoic and dodecanoic acids (C10:C12) and a supramolecular DES based on randomly methylated β-cyclodextrin (RAMEB) and levulinic acid, and with a conventional solvent, propylene glycol (PG), in absorbing four hydrophobic VOCs, toluene, limonene, siloxane D4, and decane. The vapor-liquid partition coefficients (K) of the VOCs alone and in mixture were determined using static headspace gas chromatography (SH-GC). A lab-scale bubbling device was used to measure absorption capacities under simulated industrial conditions. Results from the static method showed that RWCO performed similarly to the hydrophobic DES C10:C12, exhibiting the highest absorption affinities for the tested VOCs, with K values up to 208 000 times lower than in water. Dynamic absorption studies corroborated these results, highlighting RWCO and C10:C12 as the most effective absorbents, with an absorption capacity up to 0.884 mg/g for toluene at 100 ppm. This study also demonstrated that bubbling nitrogen in addition to heating at 60°C reduced regeneration time from 48 hours to 2.5 hours. The RWCO and C10:C12 absorbents retained over 99.5% of their initial absorption capacity after undergoing twelve consecutive absorption-desorption cycles. These results suggest that RWCO could be an effective and sustainable absorbent for VOCs abatement while complying with circular economy concept.

Introduction

The emission of volatile organic compounds (VOCs) from industrial processes is a significant environmental and health concern, leading to stringent regulations worldwide [1–3]. Consequently, developing sustainable, and economically viable technologies for VOC abatement is a pressing scientific and industrial challenge. Among the various available techniques, gas-liquid scrubbing (*i.e.* absorption) is an attractive method for treating industrial exhausts. The effectiveness of this process largely depends on the choice of the solvent, which must exhibit a high affinity for the target pollutants and possess specific properties, such as low vapor pressure and viscosity. Solvents must also be safe and cost-effective for industrial applications [4,5].

Water, while inexpensive and environmentally benign, is however, largely ineffective for capturing hydrophobic VOCs, such as aromatics, alkanes, and siloxanes common in industrial effluents and biogas, due to their very low water solubility [6]. Thus, various non aqueous solvents were evaluated for absorption processes [7–10]. Although organic solvents can exhibit a high affinity for VOCs, they often have significant drawbacks, such as high volatility, which can lead to secondary air pollution, as well as potential toxicity. In addition, substantial energy input is often required for their regeneration, and solvent losses can occur during the process due to their high vapor pressure, impacting operational costs and sustainability. These challenges have created a

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clear need for a new class of solvents that combines high performance with economic and environmental viability [4].

To overcome these limitations, the use of designer solvents has been evaluated. Due to their negligible vapor pressure, high thermal stability, and good absorption capacity for many VOCs, ionic liquids (ILs) have been extensively studied [11–13]. However, their widespread industrial adoption has been hindered by several major drawbacks, including high manufacturing costs, significant viscosity that can impede mass transfer rates, and growing concerns over their toxicity and poor biodegradability [14]. Since their discovery in 2003 [15], deep eutectic solvents (DESs) have emerged as a promising solution for various applications, including catalysis, solubilizing active compounds and gases, and extraction processes [16–20]. DESs are obtained by simply mixing a hydrogen bond acceptor (HBA) and a hydrogen bond donor (HBD), and are gaining significant attention because they are generally inexpensive, easy to prepare, and often biodegradable and less toxic than ILs [21]. Several studies have explored the potential of DESs for VOC absorption [22–26]. Hydrophobic DESs made from fatty acids were found to be very advantageous for VOCs absorption [27,28]. Supramolecular DESs based on cyclodextrins (CDs), cage molecules, have also shown promising results [29–31]. However, not all DESs can be used for VOC absorption. Some studies have used volatile or semi-volatile compounds to form eutectic mixtures and partial evaporation of these compounds could be observed under dynamic condition. In addition, some toxic HBDs or HBAs were also used [32]. Recently, the use of vegetable oils was evaluated and showed promising results for the absorption of hydrophobic VOCs [2]. These oils are widely available and non-toxic, but since they are used in food, cosmetics and pharmaceuticals, competition for them could be strong. Worldwide, the annual production of waste cooking oils (WCOs) ranges from 41 to 67 million tons [33]. There is a need to find new valorization pathways. However, impurities in raw waste oils may hinder their further use as VOC absorbents [9]. Recycled waste cooking oils (RWCO) appear to be a valuable raw material for various industrial applications such as lubricants and plasticizers [34,35]. However, to the best of our knowledge the use of RWCO as VOC absorbents has not been yet evaluated as a valorization strategy.

This study aims to evaluate the potential of RWCO compared to that of promising selected solvents under conditions close to those of industry by performing dynamic absorption and evaluating the absorption capacity of the selected solvents for VOC mixtures. Indeed, most absorption studies have focused on the determination of partition coefficient in static mode while very few have examined the ability of absorbents to trap VOCs under dynamic conditions similar to those in industrial scrubbers [28,36]. We recently demonstrated that the laboratory-scale dynamic setup developed in our laboratory [37] yielded results that agreed well with those obtained in industrial scrubbers [38]. In addition, since exhausts generally contain more than one VOC, it is also necessary to evaluate the absorption capacity for VOCs mixtures [36]. Four key VOCs found in exhaust gases and biogas, toluene, limonene, siloxane D4, and decane were selected. The performance of RWCO was compared to three promising absorbents, a hydrophobic DES based on decanoic and dodecanoic acids (C10:C12), a supramolecular DES based on randomly methylated β-CD (RAMEB:levulinic acid, RAMEB:Lev) and a conventional solvent (propylene glycol, PG). Finally, recognizing the critical importance of sustainability and cost-effectiveness in industrial processes, we evaluated different methods to regenerate the most promising solvents to determine their potential for reuse in multiple cycles. The results of this study will help improving the design and modeling of air treatment systems that use organic liquids for absorption.

Materials and experimental section

Materials

Toluene (>99.8%), limonene (>99%), octamethylcyclotetrasiloxane (siloxane D4) (>99%), decane (>99%), decanoic acid (C_{10}) (>98%), dodecanoic acid (C_{12}) (>98%), propylene glycol (PG) (>99%), levulinic acid (Lev) (98%) and randomly methylated β-cyclodextrin (RAMEB) were purchased from Sigma Aldrich. Industrial aqueous solution of propylene glycol (16% water content) was purchased from Progalva Energies (France). Waste cooking oil was supplied by a local catering company from Milan (Italy). The recycling following recycling procedure, adapted from [35] was used. WCO (1 kg) was stirred for 1 h at 50°C in the presence of 20 wt% of distilled water (0.2 kg). After decantation, the water was removed and the oil dried with Na_2SO_4, filtered, and stirred under vacuum for 1h. The RWCO employed in this work was previously characterized in detail in our earlier study [35]. In that work, its chemical composition, rheological behavior, and oxidative stability were determined by ^{1}H NMR, UV–VIS spectroscopy, and rheometry. The recycled oil consisted mainly of C16–C18 triglycerides with minor free fatty acids, showing a viscosity of 56 mPa·s at 30°C and an iodine number of 82.4, comparable to refined vegetable oils. The absence of polar contaminants and the confirmed oxidative stability indicate that the recycled oil represents a clean and reproducible hydrophobic matrix suitable for VOCs absorption studies.

High purity gases, nitrogen, air, helium and hydrogen were used for chromatographic analysis. Table 1 shows the structures and water solubility of selected VOCs.

The absorbents used in this study are listed in Table 2. The DESs were prepared by stirring the two components (HBA and HBD) at the desired molar ratio at 30°C until a clearly homogeneous liquid was formed. The water content of the studied absorbents was determined by Karl Fischer titration (Mettler Toledo C20S KF Titrator). Viscosity and density of the absorbents were determined in previous studies [27,35,39].

Vapor-liquid partition coefficients

Evaluating the vapor-liquid partition coefficient ($K= C_G/C_L$) is crucial for further industrial applications. The K value for the VOCs in the studied absorbents was measured experimentally using static headspace gas chromatography (SH-GC) as described previously [23,27].

All measurements were performed with a Thermo Scientific Tri-Plus^{TM} 500 headspace autosampler coupled to a Trace 1300 gas chromatograph equipped with a flame ionization detector and a DB 624 column using nitrogen as carrier gas. The GC column temperature was set at 80°C for toluene and at 120°C for limonene, siloxane D4 and decane.

The studied VOCs were added at a known concentration in a precise mass of solvent (about 3.5 g) placed in 20mL vials. Samples were equilibrated during 24 hours under magnetic stirring to reach equilibrium before SH-GC analysis. VOCs will distribute between both phases according to the thermodynamically controlled equilibrium constant. All experiments were performed in triplicate and the partition coefficients did not vary by more than 5%.

Lab scale dynamic absorption

A dynamic bubbling setup was designed previously in our laboratory to mimic industrial absorption processes based on a scrubber [36–38]. Polluted gas is generated by injecting liquid VOCs at various rates into a nitrogen stream. The flow rates of the stream are monitored by mass flow controllers. In this study, since VOC mixtures were used, the gas concentrations of the inlet and outlet gas were monitored with a Micro GC Fusion (Chemlys). The temperature gradient was as follows: an initial temperature of 140°C for 15 s, increasing to 200°C at a rate of 1°C/s and held at 200°C for 45 s. The sample inlet temperature was set at 70°C.

A precise mass (20 g) of absorbent is used in each experiment. The temperature is maintained at 30°C using a thermostated cable. The gas flows through the by-pass and is analyzed by the GC. Once the

Table 1
Studied VOCs and their water solubility.

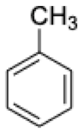
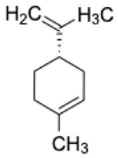
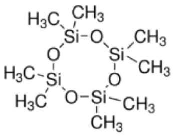
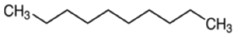
VOCs	Toluene	Limonene	Siloxane D4	Decane
Structure				
Water solubility (mg L^{-1}) 25°C	526	7.57	0.056	0.052

Table 2
List of absorbents used in the study, their abbreviation, molar ratio in the case of DESs, water content and viscosity.

Absorbent		Molar ratio	Abbreviation	Water content (%)	Viscosity at 30°C (mPa.s)
Recycled waste cooking oil			RWCO	0.04	56.0[a]
Propylene glycol			PG	0.52	30.6[b]
			PG16	16.00	20.2[a]
DESs					
HBA	HBD				
Decanoic acid	Dodecanoic acid	2:1	C10:C12	0.05	9.1[b]
RAMEB	Levulinic acid	1:30	RAMEB:Lev	2.00	212.9[c]

[a] ref [35];
[b] ref [27];
[c] ref [39]

concentration stabilizes, both valves are switched and the gas flows through the absorbent. The outlet gas concentration is monitored until the absorbent is fully saturated (outlet gas value equal to initial inlet value). For the desorption process, the feed (inlet) is removed, and nitrogen gas is used to strip and regenerate the saturated solvent. The amount of absorbed VOC can be determined by integrating the obtained curves. The effect of the initial toluene concentration was investigated at five different flow rates (10, 20, 40, 80 and 100 μL.hr^{-1}). Absorption/desorption experiments were repeated three time.

Regeneration study

After analysis, the vials containing the VOC-loaded solvents were uncapped and regenerated. Three regeneration methods were evaluated: heating, nitrogen bubbling and a combination of both. Total VOC removal was verified by analyzing the vial over time until no toluene could be detected by GC. Then, we evaluated the absorption capacities of the solvents using the previously described procedure.

Results and discussion

Partition coefficient

VOC alone

The partition coefficients obtained at 30°C in this study and in previous studies are given in Table 3. The decrease in K values observed for all VOCs in all solvents compared to water indicated that a greater amount of VOC was absorbed in these solvents. Furthermore, the K values of VOCs in the hydrophobic DES and in RWCO are lower compared to other studied absorbents.

The obtained K values for toluene in the studied solvents ranged from 4.48E-04 to 1.48E-02 (up to 656-fold lower than in water). RWCO exhibited similar or lower K values compared to the C10:C12 DES and lower K values than the conventional solvent PG. Among the DESs C10:C12 has the lowest K value. The same observation is true for limonene, siloxane D4 and decane, with a up to 23 800, 12 800 and 208 000-fold decrease of K, respectively.

Regarding the absorption capacities of RAMEB:Lev, the lowest value of K for limonene compared to toluene and of decane compared to siloxane D4 could be explained by the formation constant of these VOCs with β-CDs. Indeed, previous studies have determined a formation constant value of 171 M^{-1} for toluene, 4386 M^{-1} for limonene, 8 M^{-1} for siloxane D4 with RAMEB and of 100 M^{-1} for β-CD/decane [30,37,40,41]. As observed previously, the use of the commercial solution of PG containing 16% of water lead to higher partition coefficient compare to PG [27].

Table 3
Partition coefficient (K) for toluene, limonene, siloxane D4 and decane in water, and in the studied solvents at 30°C. (Error <5%).

Absorbent	Toluene	Limonene	Siloxane D4	Decane
Water	0.294[a]	2.32[a]	4[a]	21
C10:C12	5.14E-04[a]	1.13E-04[a]	3.14E-04[a]	1.11E-04
RAMEB:Lev	1.00E-03[b]	6.18E-04	1.36E-02	6.42E-03
Propylene glycol	3.31E-03[a]	1.42E-03[a]	1.23E-02[a]	6.19E-03
Propylene glycol 16	1.48E-02[a]	1.17E-02[a]	2.81E-02[a]	3.97E-02
Recycled waste cooking oil	4.48E-04	9.73E-05	4.45E-04	1.01E-04

[a] ref [27];
[b] ref [29]

Toluene is the most widely studied VOC in the literature and serves as a model for hydrophobic VOCs. The K value obtained in the RWCO is among the lowest reported in the literature for DESs [23], ILs [42,43] or oils [2,44]. Few studies have evaluated VOC absorption in vegetable oils. A recent study evaluated different vegetable oils for the absorption of hydrophobic VOCs. The data obtained ranged from 1.4 E-02 to 4.4 E-04 for toluene [2]. In addition, the partition coefficient obtained for butane, pentane, hexane and heptane decreases with increasing carbon chain length. The smallest K value obtained for heptane is 1.1.E-03 in peanut oil. The value of 1.01E-04 obtained for decane in this study is therefore consistent with values for shorter-chain alkanes. Therefore, since recycling WCO does not alter its efficacy, VOC absorption could be an interesting output for RWCOs.

Mixture of VOCs

Since real gaseous effluents and raw biogas generally contain a wide variety of VOCs, we examined how VOC mixtures affect the partition coefficient of individual VOCs. A mixture of the four VOCs in equimolar amounts was prepared to mimic a real sample. Various amounts of this stock solution were then added to vials that had previously been filled with the absorbent. The K values for toluene, limonene, siloxane D4, and decane both individually and as a mixture at 30°C are shown in Table 4.

Most of the K values are in the same order of magnitude as those obtained for the individual VOCs. We can observe a slight increase in the K value of toluene, limonene and decane in the case of RWCO as well in the case of RAMEB:Lev. Concerning the polar absorbents, we can observe a slight decrease in the partition coefficient for VOCs in the mixture. This could be due to the solvent + VOCs solution becoming more hydrophobic overall. This phenomenon has been observed in a previous study [36]. However, these results demonstrate that the absorbents maintain good affinities with each VOC compound in the presence of other VOCs, with no evidence of competitive effects between VOCs. The absence of competitive effects in VOC mixtures indicates that the VOCs dissolve in the absorbent through physical processes [36].

Lab-scale dynamic absorption

Effect of VOC initial concentration

The effect of the initial VOC concentration on the absorption was investigated for toluene and C10:C12 using five different flow rates of 10, 20, 40, 80 and 100 $\mu L.hr^{-1}$ (corresponding to 50, 100, 204, 410 and 525 ppm) with nitrogen flow rate at 10 $L.hr^{-1}$ at 30°C. As shown in Fig. 1 the absorption capacity of C10:C12 increases with toluene concentration, which is consistent with a physical absorption process. A similar tendency can be found in the literature [28]. In addition, the value obtained for toluene at 410 ppm of 3.94 mg/g is similar to the value obtained in a previous study (3.54 mg/g) [28]. These results validate our setup device and allow us to compare the absorption capacity of the other absorbents and VOCs.

Determination of absorption capacities for VOC alone and in mixture

The absorption capacities of the studied absorbents for toluene, limonene and siloxane were evaluated at 30°C, with a VOC flow rate of 20 $\mu L.h^{-1}$ and a N_2 flow rate of 10 $L.h^{-1}$ (Fig. 2). Under these conditions, the concentrations of toluene, limonene, and siloxane D4 were 100, 120, and 50 ppm, respectively.

The C10:C12 DES showed the best affinity for all the studied VOCs. The absorption capacities obtained in dynamic mode are in good agreement with the K values obtained in static mode. In static mode C10:C12 and RWCO exhibit similar K values. The lowest results observed for RWCO in dynamic adsorption (0.57 $mg_{toluene}/g_{RWCO}$), could be explained by its higher viscosity, as viscosity significantly influences hydrodynamics and mass transfer in gas-liquid contactors [4]. Indeed, at 30°C the viscosity of RWCO is 56.0 mPa.s, while the viscosity of C_{10}:C_{12} is only 9.1 mPa.s. RAMEB:Lev and PG16 exhibit a lower absorption capacity.

For the C10:C12 DES, a slight decrease in toluene absorption capacity was observed from 0.88 to 0.77 $mg_{toluene}/g_{C10:C12}$ in the mixture. Meanwhile, the absorption capacities of limonene and siloxane remained stable at 0.33 and 0.34 $mg_{limonene}/g_{C10:C12}$ and 0.22 and 0.23 $mg_{siloxaneD4}/g_{C10:C12}$ for the VOC alone and in the mixture, respectively. The same behavior was observed in RWCO: the absorption capacity of toluene in the mixture was slightly lower than that of the VOC alone at 0.39 $mg_{toluene}/g_{RWCO}$ and 0.57 $mg_{toluene}/g_{RWCO,}$ respectively. The absorption capacities for limonene were 0.228 and 0.195 $mg_{limonene}/g_{RWCO}$ for VOC alone and in the mixture, respectively, and for siloxane D4 they were 0.134 and 0.121 $mg_{siloxaneD4}/g_{RWCO}$, for VOC alone and in the mixture, respectively. Once again, the results obtained demonstrated that the use of a VOC mixture does not significantly affect the absorption capacity of absorbents.

Solvent regeneration

Replacing solvents can be costly, so they must be recycled after use. Regeneration is crucial for sustainability, cost-effectiveness, and process efficiency, and is a critical issue for many industrial operations. However, most studies fail to consider this parameter. Thermal desorption (heating and stirring) is the most widely studied regeneration methods. From an industrial point of view, however, the regeneration is too long. Therefore, to ensure a shorter regeneration time, we evaluated the effect of bubbling nitrogen with or without heating and compared the results with those of thermal desorption. These three methods can effectively remove toluene from DESs [24].

First, we evaluate the time required to regenerate the RWCO and C10:C12 absorbents. Then, we performed several absorption/desorption cycles. In a previous study, the regeneration of the C9:C12 DES loaded with toluene was evaluated at 48 hours by thermal desorption at 60°C [27]. The regeneration time of the RWCO and C10:C12 can be reduced to 2.5 hours by bubbling nitrogen and heating at 60°C. No significant changes in the absorption capacity were observed. The RWCO and C10:C12 absorbents maintained up to 99.5% of their absorption capacity over twelve absorption-desorption cycles. The reversibility of the process at moderate temperature is further evidence of a physical absorption process. The weight loss of RWCO and C10:C12 was less than 1% for all three types of regeneration. Bubbling nitrogen with heating was the most effective method due to its shorter regeneration time.

Conclusion

This study evaluated the performance of RWCO in absorbing VOCs, both individually and in mixtures, under static and dynamic conditions in comparison with other promising absorbents. Among the tested absorbents, RWCO and the hydrophobic DES C10:C12 demonstrated superior absorption affinity for toluene, limonene, siloxane D4, and decane. Dynamic absorption experiments confirmed the high potential of RWCO. RWCO showed comparable K values in static mode to the hydrophobic DES C10:C12, but slightly lower absorption capacity in dynamic mode due to its higher viscosity. However, RWCO has the advantage of being ready to use. The absorption capacities of the studied solvents were not significantly reduced when exposed to a mixture of VOCs, indicating their potential for treating complex emissions. Both RWCO and C10:C12 proved to be stable, retaining over 99.5% of their initial absorption capacity after twelve consecutive absorption-desorption cycles with minimal weight loss. These results suggest that

Table 4

Partition coefficient (K) of the VOC alone and in mixture in C10:C12, RWCO, RAMEB:Lev and propylene glycol at 30°C. (Error <5%).

Absorbent	Toluene		Limonene		Siloxane D4		Decane	
	Alone	In mixture	Alone	In mixture	Alone	In mixture	Alone	In mixture
C10:C12	5.14E-04	4.40E-04	1.13E-04	1.13E-04	3.14E-04	2.20E-04	1.11E-04	1.30E-04
RAMEB:Lev	1.00E-03	1.10E-03	6.18E-04	6.20E-04	1.36E-02	1.60E-02	6.42E-03	6.70E-03
Propylene glycol	3.31E-03	3.21E-03	1.42E-03	1.32E-03	1.23E-02	1.00E-02	6.19E-03	5.00E-03
Propylene glycol 16	1.48E-02	1.36E-02	1.17E-02	1.10E-02	2.81E-02	2.61E-02	3.97E-02	4.02E-02
RWCO	4.48E-04	5.40E-04	9.73E-05	1.50E-04	4.45E-04	4.45E-04	1.01E-04	1.40E-04

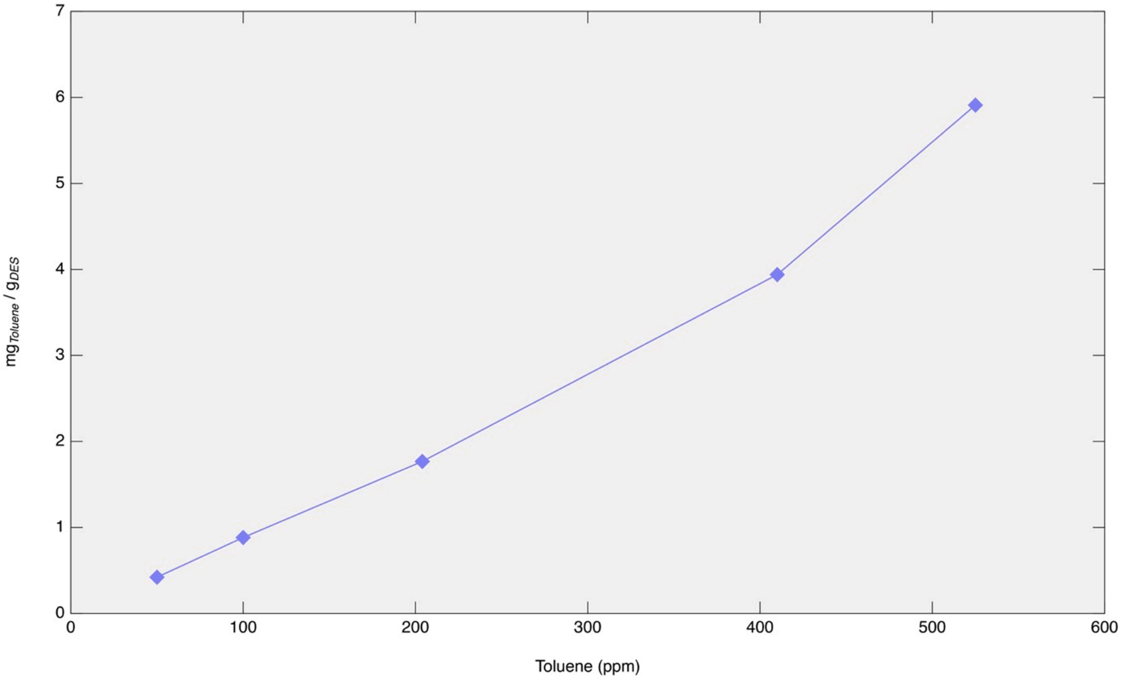


Fig. 1. Absorption capacity of C10:C12 for different initial toluene concentrations at 30°C.

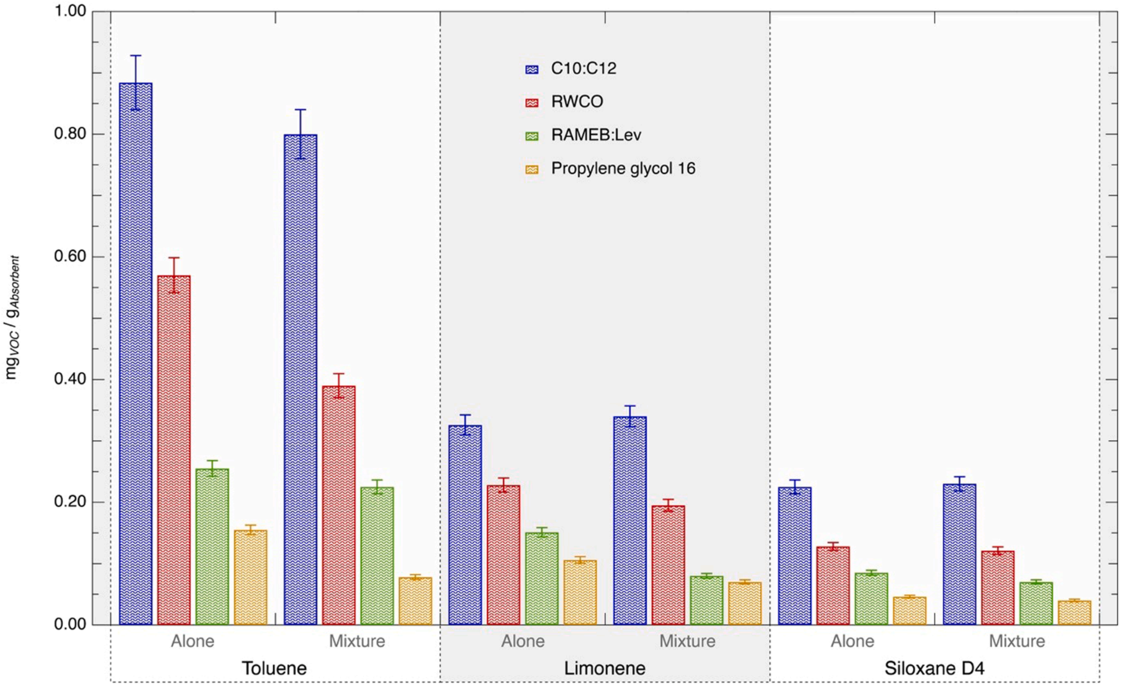


Fig. 2. Absorption capacity of VOC alone and in mixture in C10:C12, RWCO, RAMEB:Lev and propylene glycol 16 at 30°C.

RWCO and hydrophobic DESs such as C10:C12 are highly effective and regenerable physical absorbents for VOC abatement. The performance of RWCO, coupled with the advantage of using recycled materials, presents a promising approach to developing economical and environmentally friendly VOC capture technologies for industrial settings.

CRediT authorship contribution statement

P. Villarim: Writing – original draft, Investigation, Data curation. **A. Ahmad Kassem:** Writing – review & editing, Investigation. **A. Mannu:** Writing – review & editing, Funding acquisition. **A. Mele:** Writing – review & editing, Funding acquisition. **J. Zemmouri:** Writing – review & editing, Supervision, Funding acquisition, Conceptualization. **S. Fourmentin:** Writing – original draft, Supervision, Resources, Methodology, Funding acquisition, Conceptualization.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Data availability

Data will be made available on request.

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