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SYNTHESIS AND CHARACTERIZATION OF THYMOL-BASED HYDROPHOBIC DEEP EUTECTIC SOLVENTS

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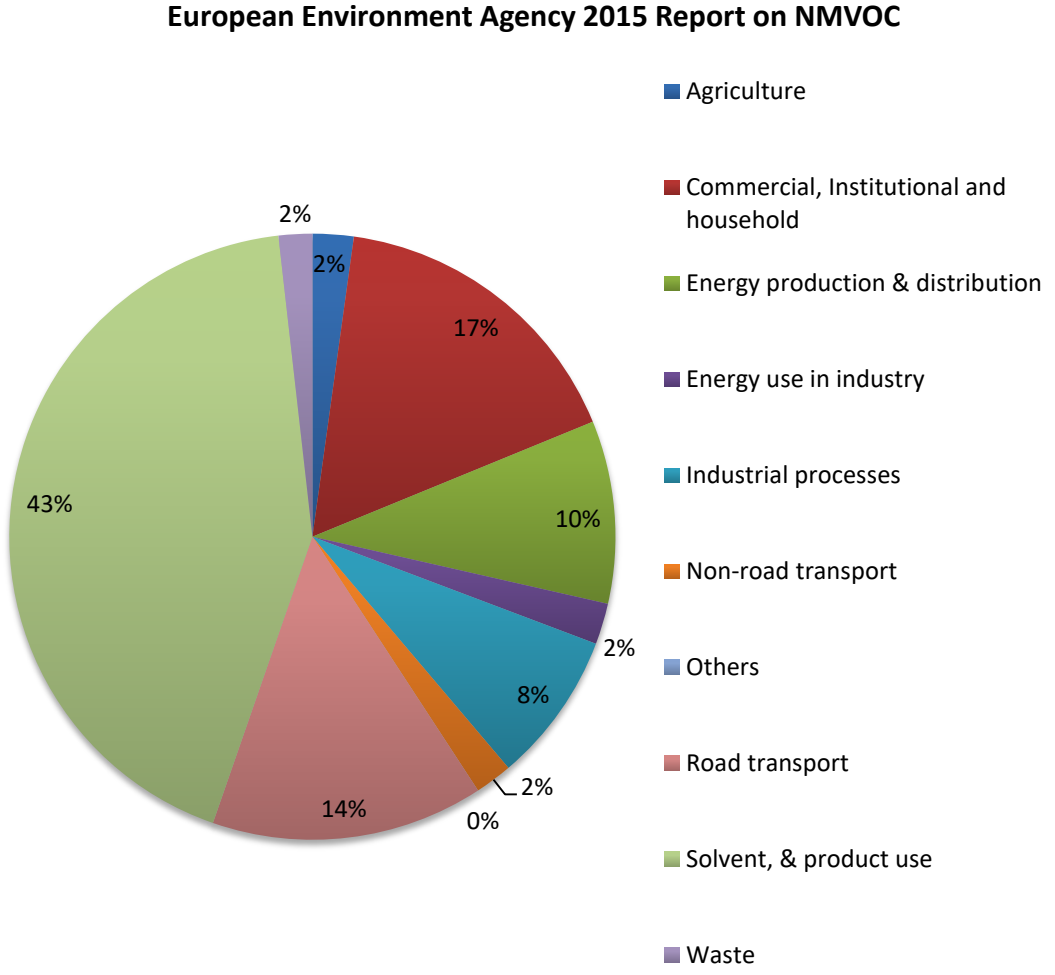
SOLVENT DEMAND

- Industrial demands for solvents by 2026.
- Conventional solvents
- Sustainable Development Goal 13 (Climate action)
- Projected increase in the search for greener solvents as renewable substitutes.
- Solvent market targets biodegradable and ecosystem-friendly solvents.



❖ https://www.eea.europa.eu/data-and-mapsgov/region1/airquality/voc.html

❖ Globenewswire. Reports and Data (June 06, 2019). https://www.globenewswire.com/newsrelease/2019/06/06/1865517/0/en/Solvents-Market-To-Reach-USD-17-99-Billion-By-2026

❖ Clarke, C.J., Tu, W.C., Levers, O., Bröhl, A. and Hallett, J.P. (2018). Green, and Sustainable Solvents in Chemical Processes. *Chem Rev.* , 118(2):747–800.

INTRODUCTION

❑ Deep Eutectic Solvents (DES)

- Hydrogen bonding between a HBD and HBA
- MP lower than that of each component
- Liquid between 25°C and 150°C

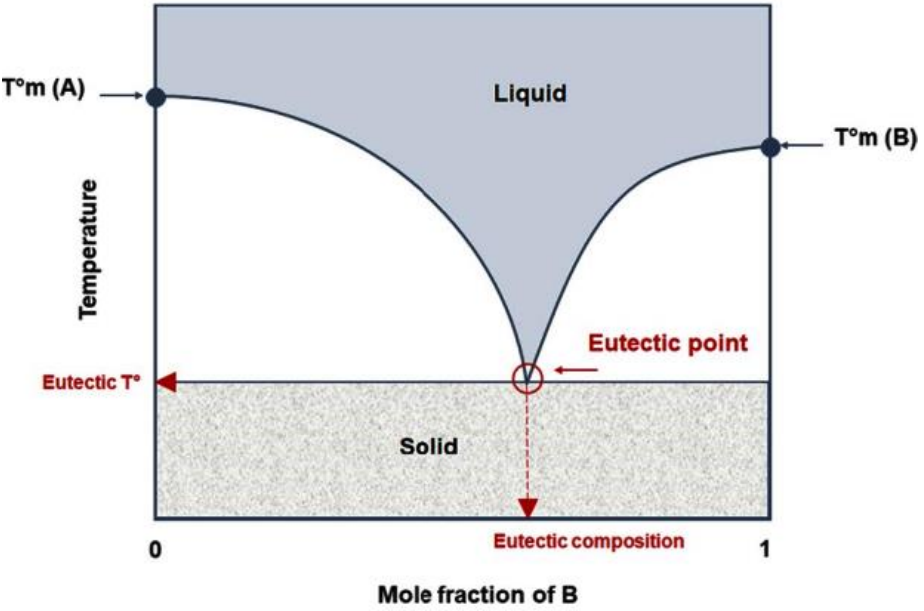


Figure 1: Phase Diagram for DES

❖ Smith, E.L., Abbott, A.P., and Ryder, K.S. (2014). Deep Eutectic Solvents (DESs) and Their Applications. *Chem Rev*., 114:11060–82.

STATE OF THE ART ON DESs

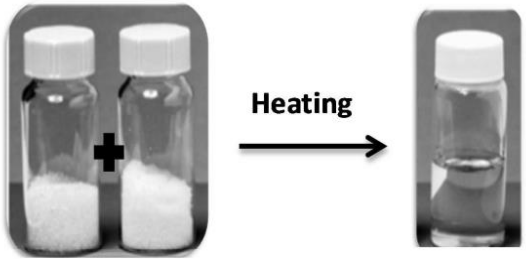


Figure 2: Pictorial s of DES Preparation

- Characteristics of DESs
- Classification of DES

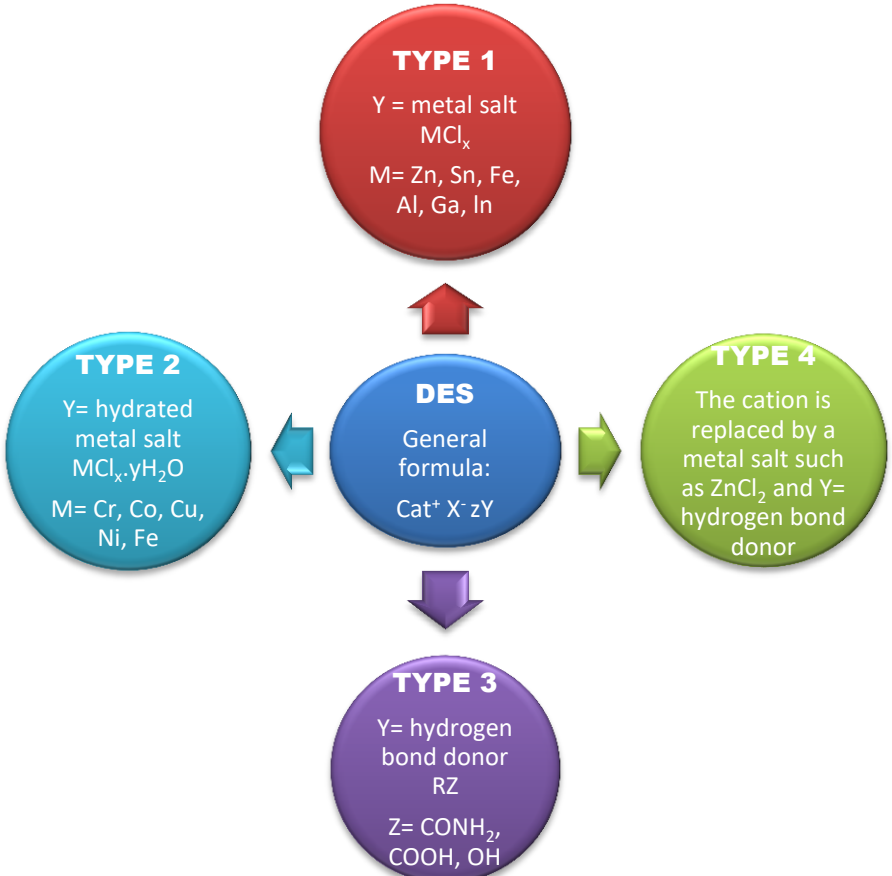


Figure 3: Classes of DES

❖ **Sub-class of Type III DES :**

a. Natural DESs
b. Carboxylic acid-based DESs
c. Therapeutic DESs

❖ **Classification based on affinity for water:**

a. Hydrophobic DES (HDES)
b. Hydrophilic DES

❖ **Applications of DESs**

Figure 4: Various Applications of HDES

Florindo, Catarina, Branco, L. C., & Marrucho, I. M. (2019). Quest for Green-Solvent Design: From Hydrophilic to Hydrophobic (Deep) Eutectic Solvents. *ChemSusChem*, *12*(8), 1549–1559. https://doi.org/10.1002/cssc.201900147

METHODS

Table 1: Composition of prepared Deep Eutectic Solvents

HBA	HBD	Abbreviation	Mole ratio	Physical appearance
Thymol	Octanoic acid	MC_8	1:1	Pale yellow liquid
	Decanoic acid	MC_{10}	1:1	Colourless liquid
	Dodecanoic acid	MC_{12}	1:1	Golden yellow liquid

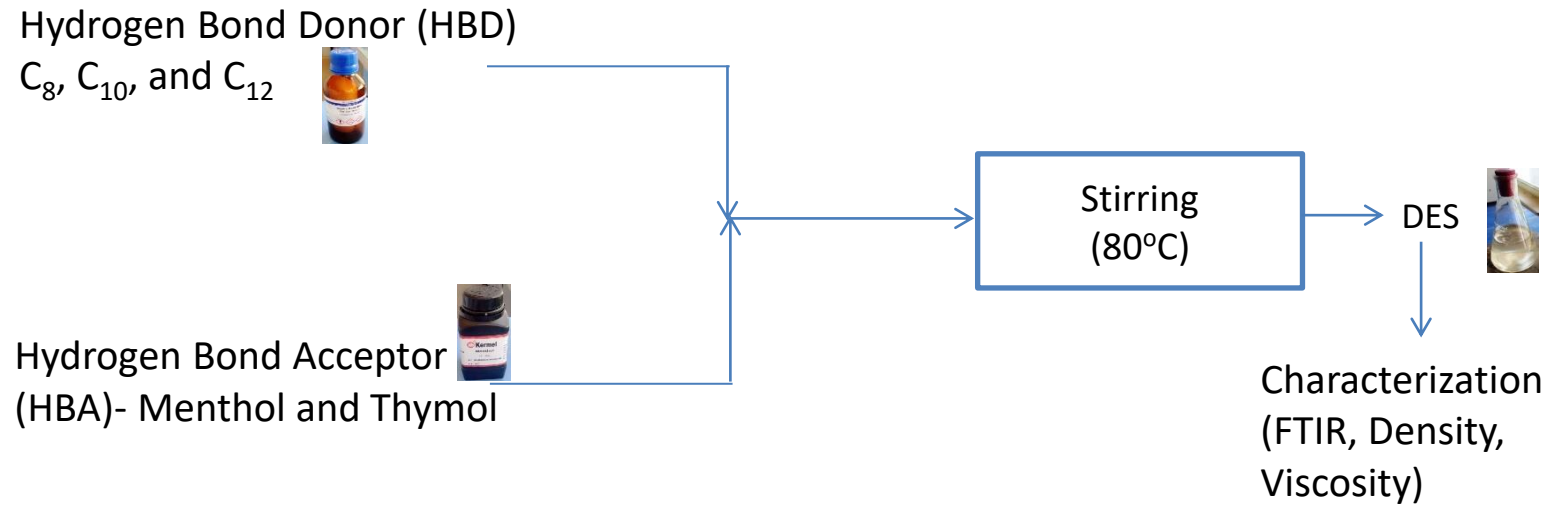


Figure 5: Experimental method for DES preparation and characterization

RESULTS

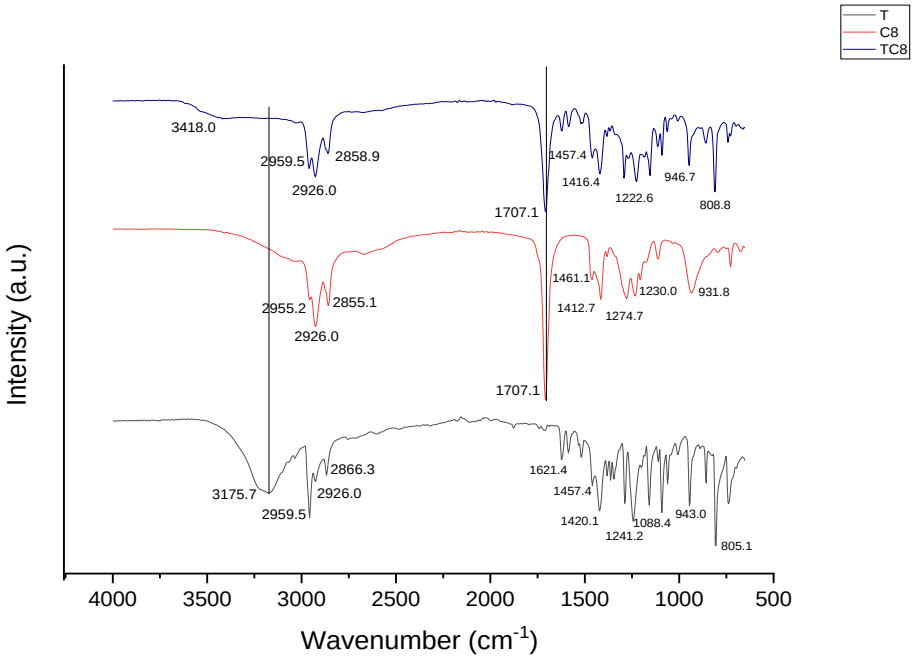


Figure 5: FTIR Spectra for Thymol, Octanoic acid, and MC8 HDES

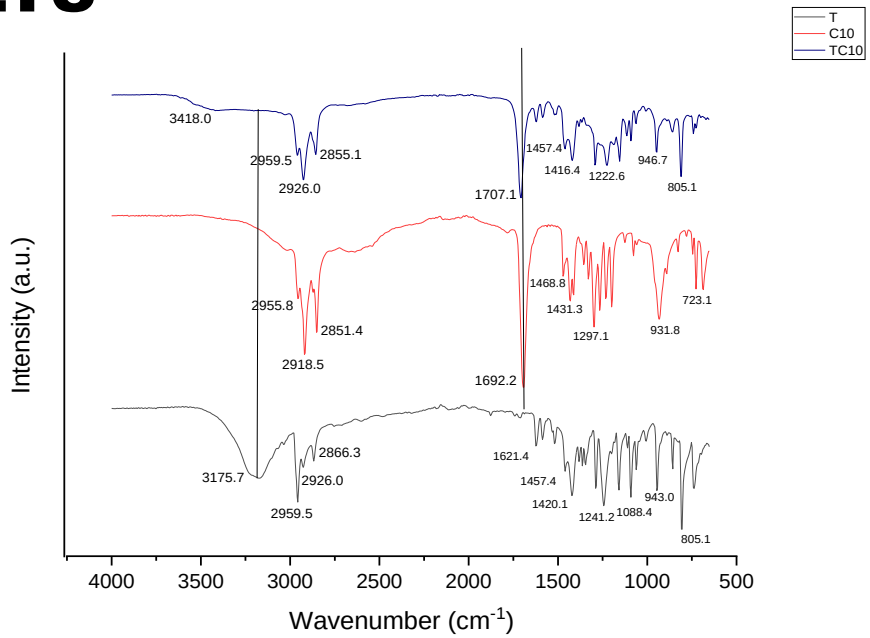


Figure 6: FTIR spectra for Thymol, Decanoic acid, and MC10 HDES

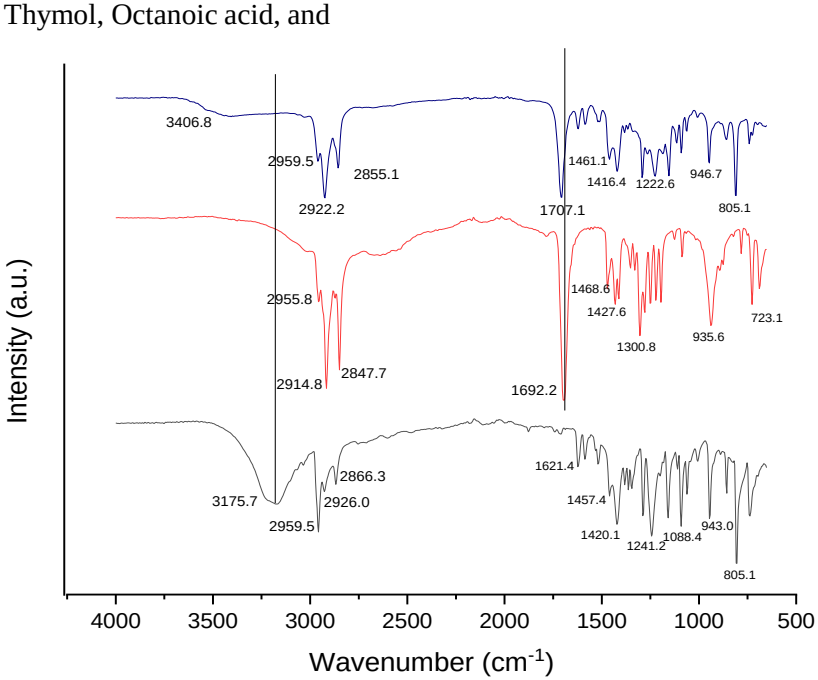


Figure 7: FTIR spectra of Thymol, Dodecanoic acid, and MC12 HDES

Physicochemical properties

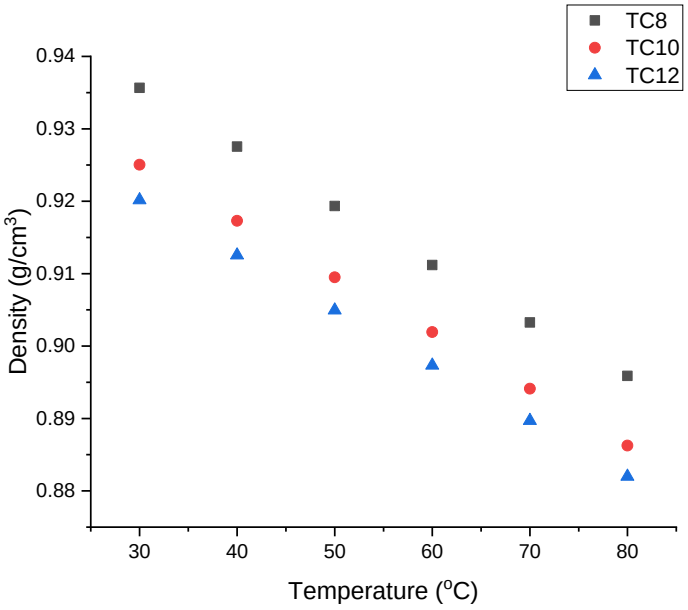


Figure 8: Density-temperature graph for Thymol based HDESs

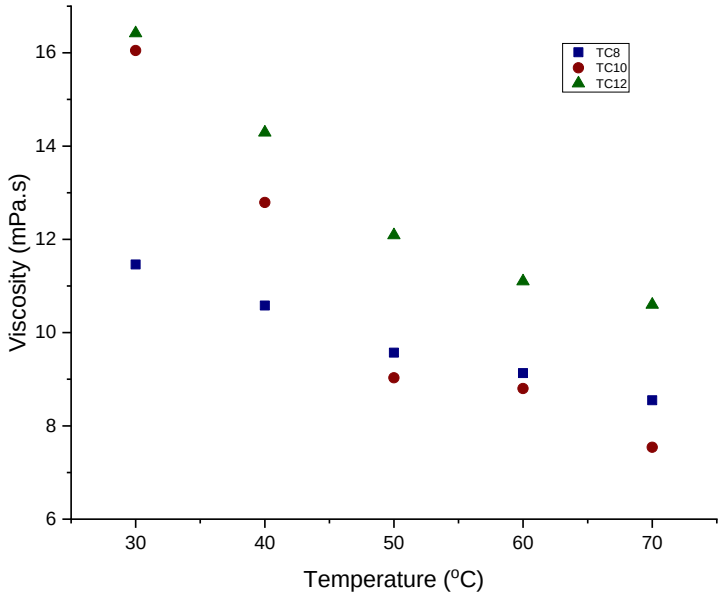


Figure 9: Viscosity-temperature graph for Thymol based HDESs

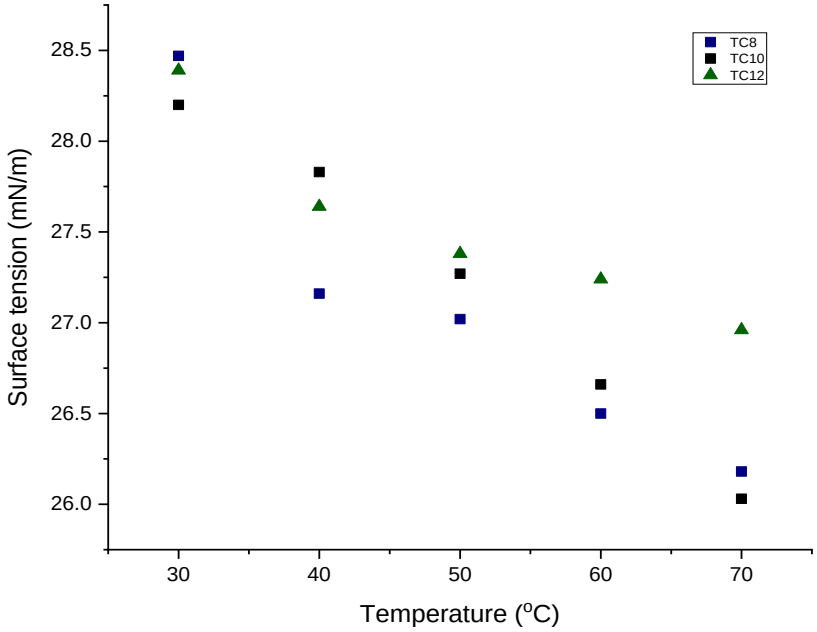


Figure 10: Surface tension-temperature graph for Thymol based HDESs

Test for hydrophobicity

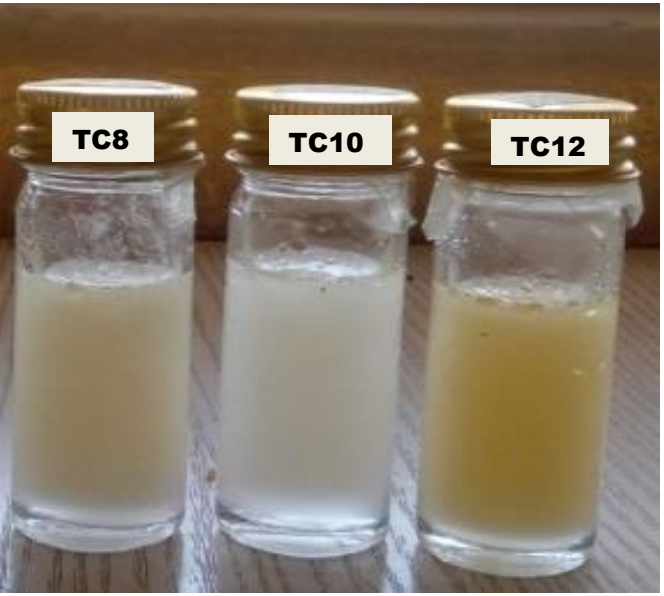


Figure 11: HDESs in water immediately after agitation

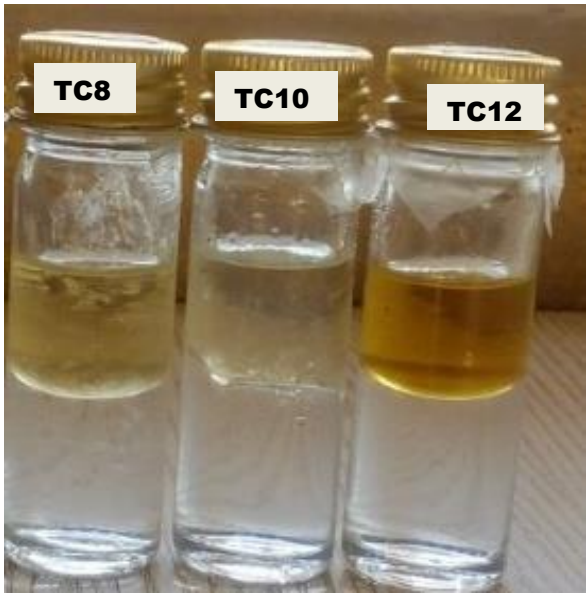


Figure 12: HDESs in water 24 hours after agitation

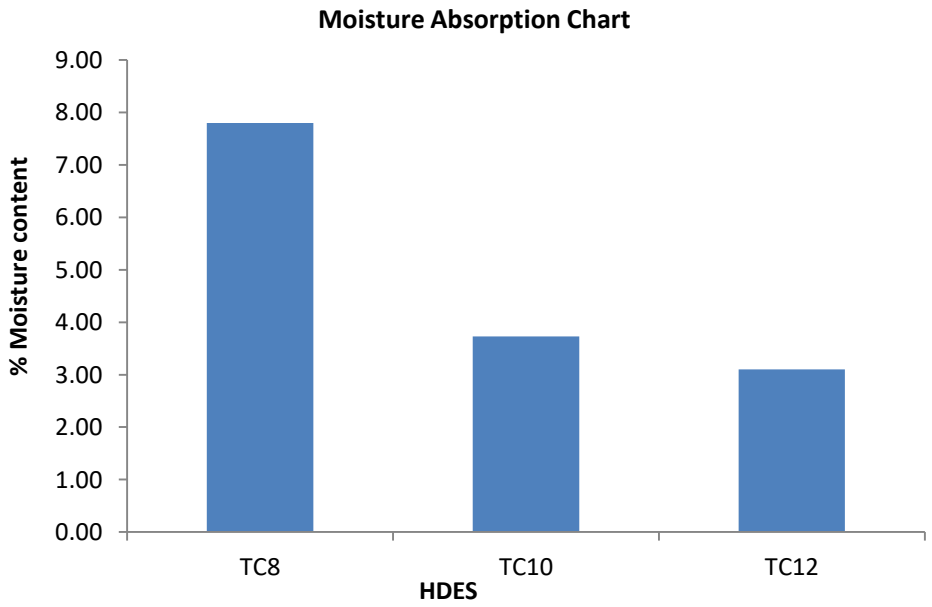


Figure 13: Percentage Moisture content graph for HDESs (Test for hydrophobicity)

CONCLUSION

- Three (3) deep eutectic solvents from menthol and long-chain organic acids were successfully prepared.

- FTIR spectra of the DESs revealed remarkable shifts in their O-H stretching bands and C=O stretching bands when compared with their precursors. The changes in the OH stretching bands and C=O stretching bands resulted from the intermolecular hydrogen bond formed between the starting materials to give DESs.

- The density of DESs was found to be between 0.925 – 0.940kg/l. All densities were found to decrease with an increase in temperature.

- The viscosity of the HDESs was found to be <20 mPa.s and decreased with stepwise increase in temperature.

- Similarly, the surface tension of DESs also reduced with stepwise increase in temperature and was observed to be <30 mN/m.

- Finally, the extent of moisture absorption into the matrix of the DESs was determined to be between 3.10 – 7.80 %. The degree of hydrophobicity increased with an increase in the alkyl chain of the organic acids (HBDs).

THANK YOU FOR LISTENING!