

SYNTHESIS AND CHARACTERIZATION OF MENTHOL-BASED HYDROPHOBIC DEEP EUTECTIC SOLVENTS

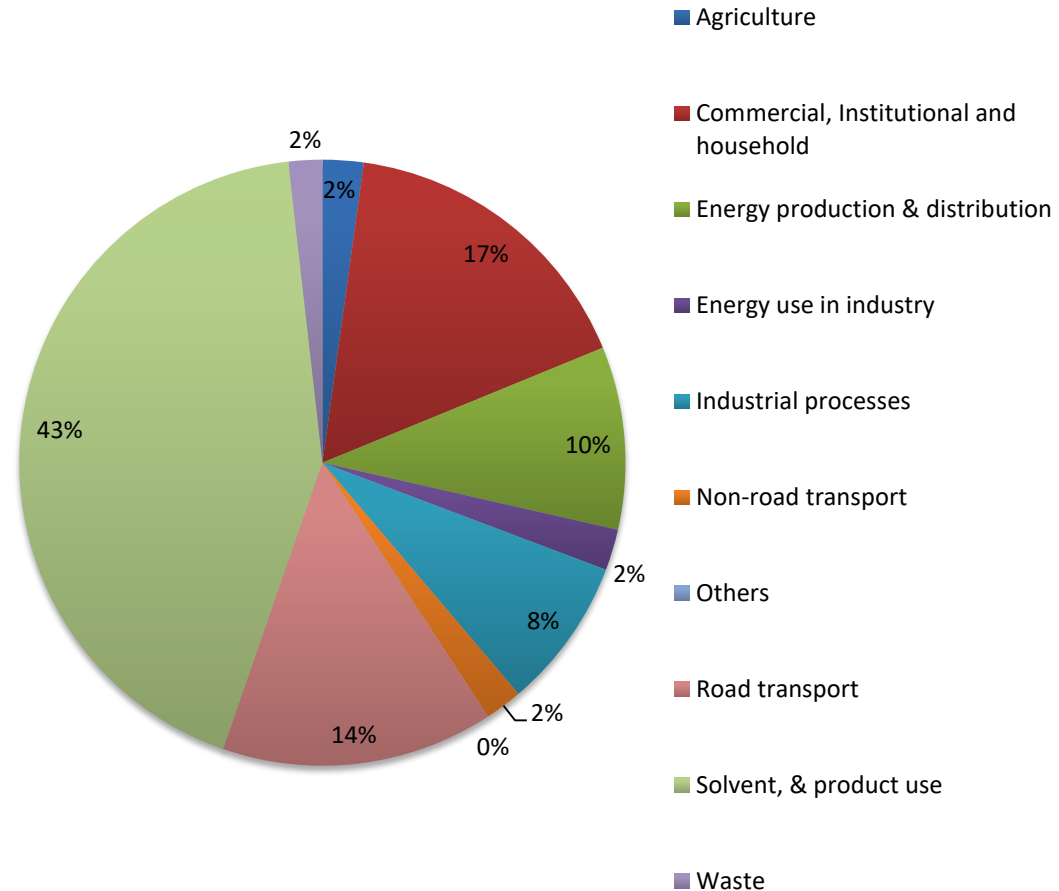
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SOLVENT DEMAND

European Environment Agency 2015 Report on NMVOC

- ❑ 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

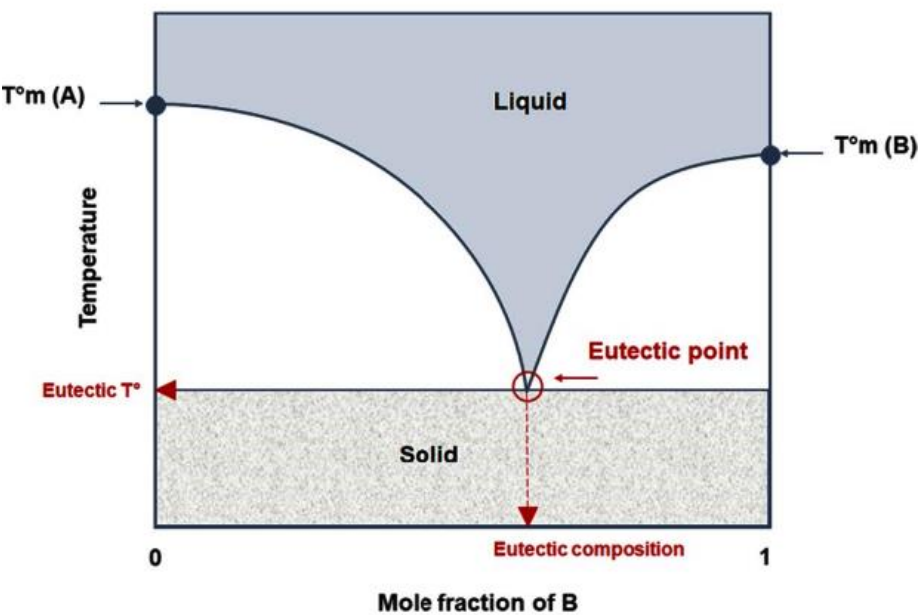


Figure 1: Phase Diagram for DES

STATE OF THE ART ON DESs

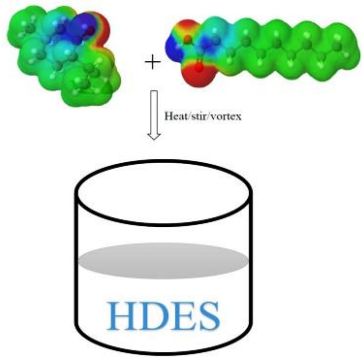


Figure 2: Pictorial s of DES Preparation

Characteristics of DESs

Classification of DES

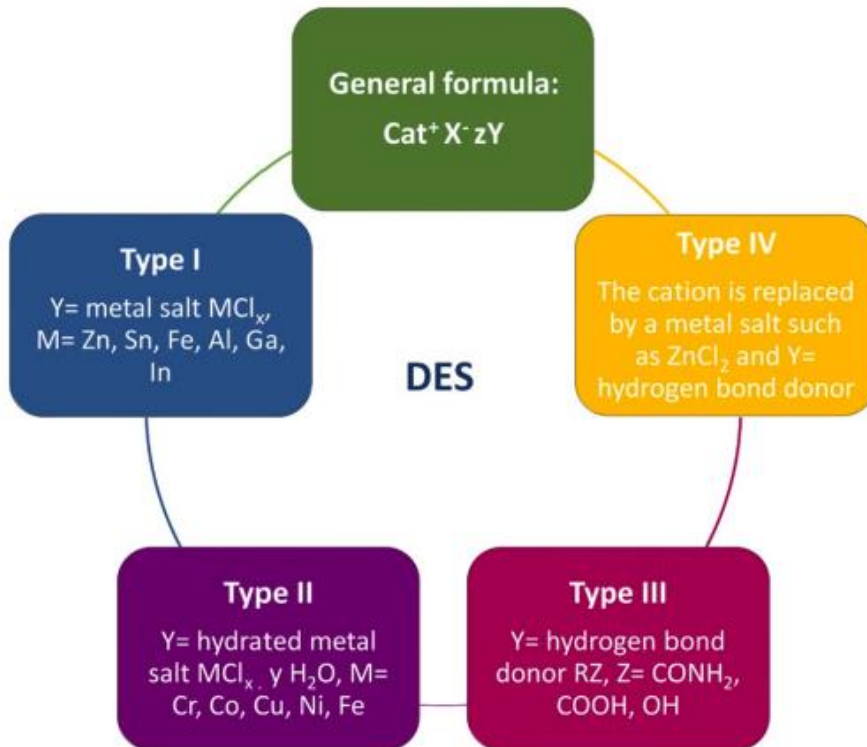


Figure 3: Classes of DES

❖ **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**

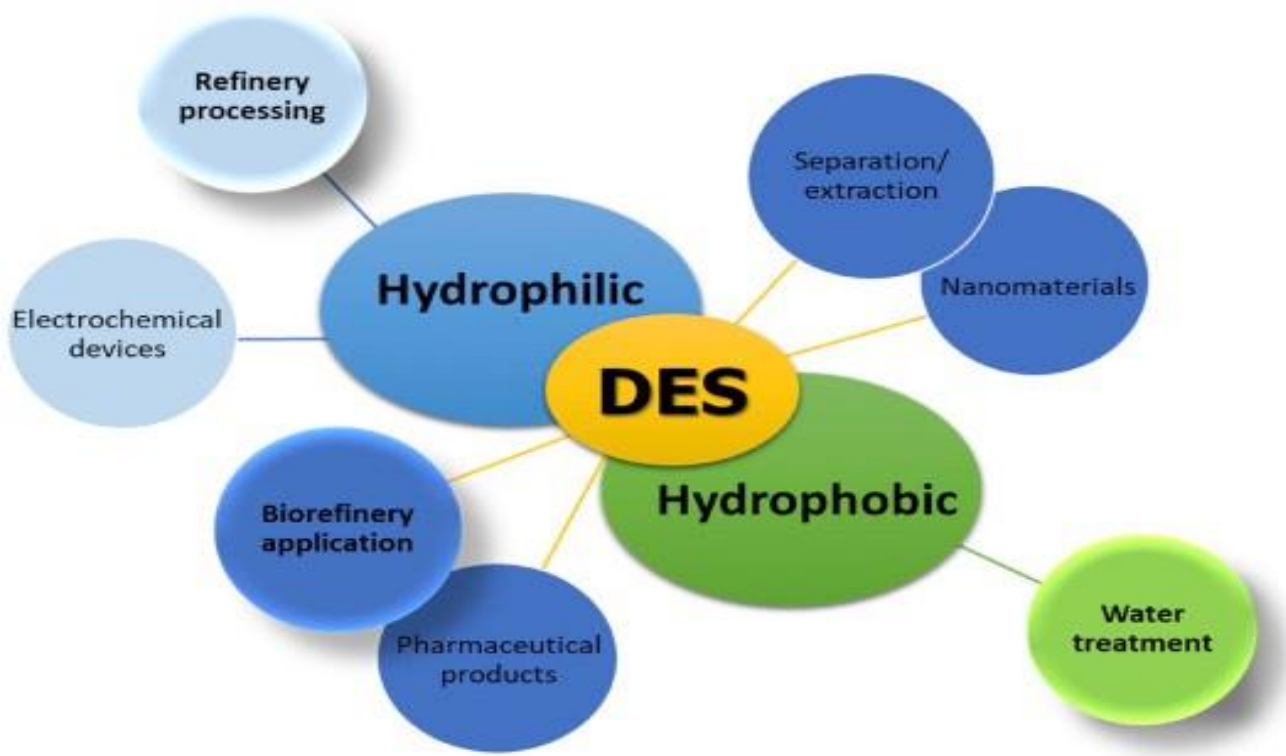


Figure 4: Various Applications of DES

METHODS

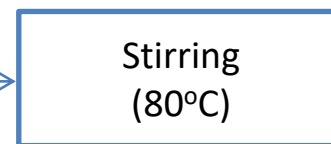
Table 1: Composition of prepared Deep Eutectic Solvents

HBA	HBD	Abbreviation	Mole ratio	Physical appearance
Menthol	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

Hydrogen Bond Donor (HBD)

C₈, C₁₀, and C₁₂

Hydrogen Bond Acceptor
(HBA)- Menthol and Thymol



DES

↓
Characterization
(FTIR, Density,
Viscosity)

Figure 5: Experimental method for DES preparation and characterization

RESULTS

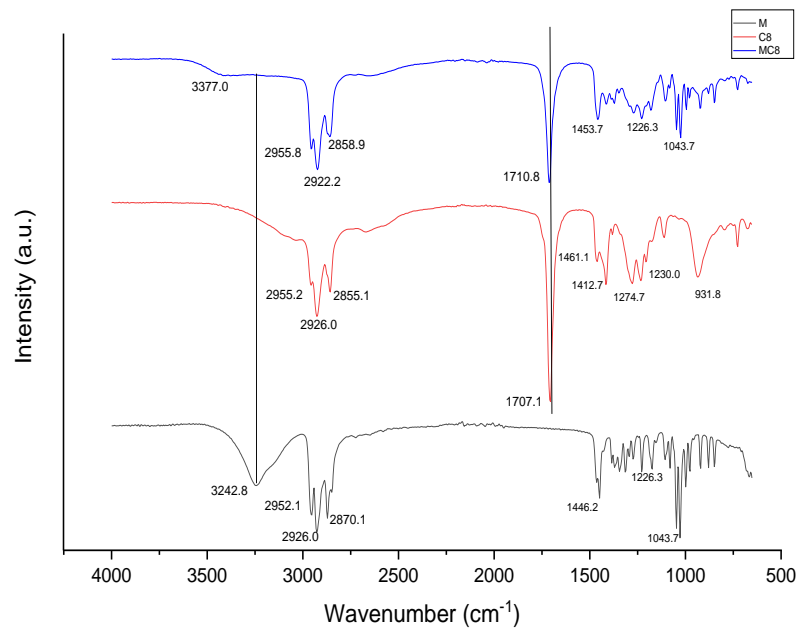


Figure 6: FTIR Spectra for Menthol, Octanoic acid, and MC8 HDES

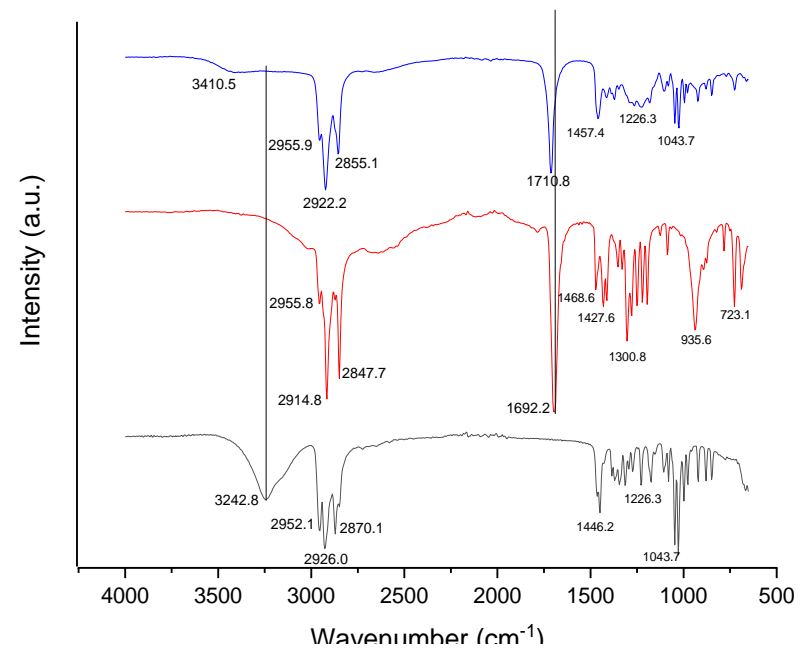


Figure 7: FTIR spectra for Menthol, Decanoic acid, and MC10 HDES

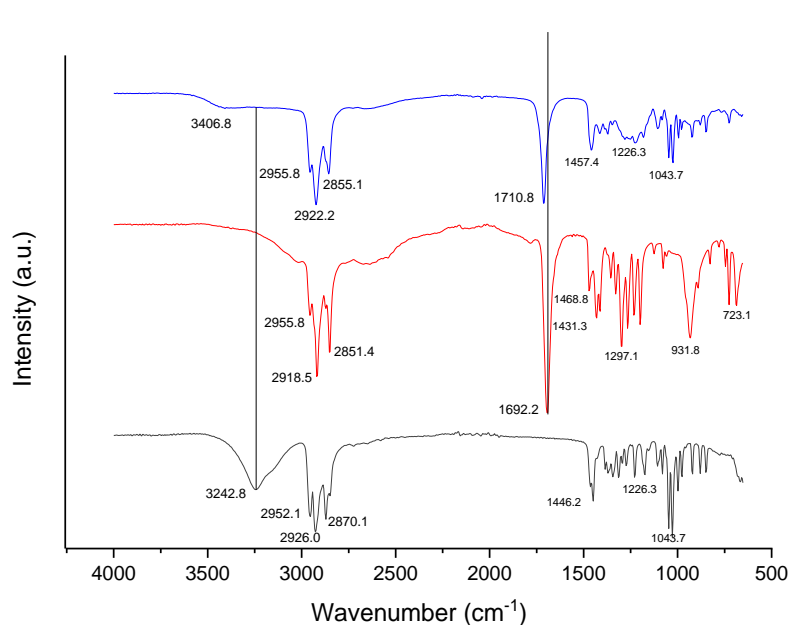


Figure 8: FTIR spectra of Menthol, Dodecanoic acid, and MC12 HDES

Physicochemical properties

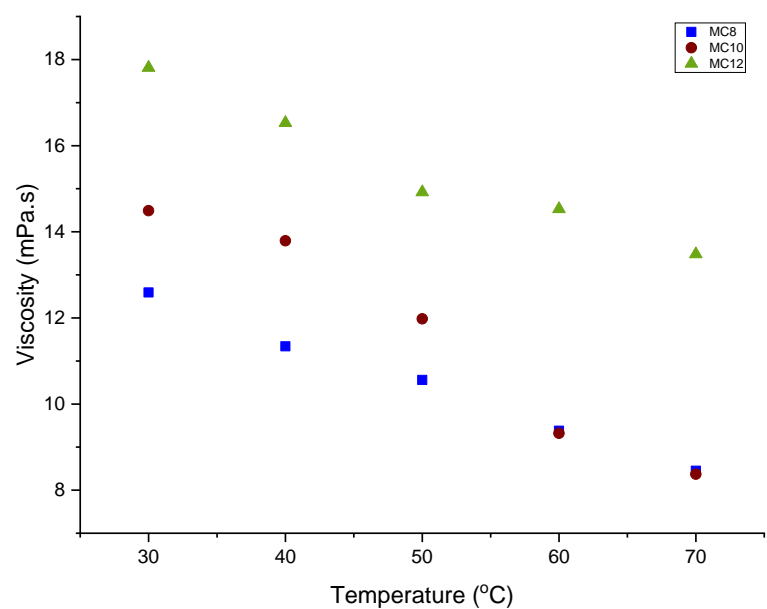
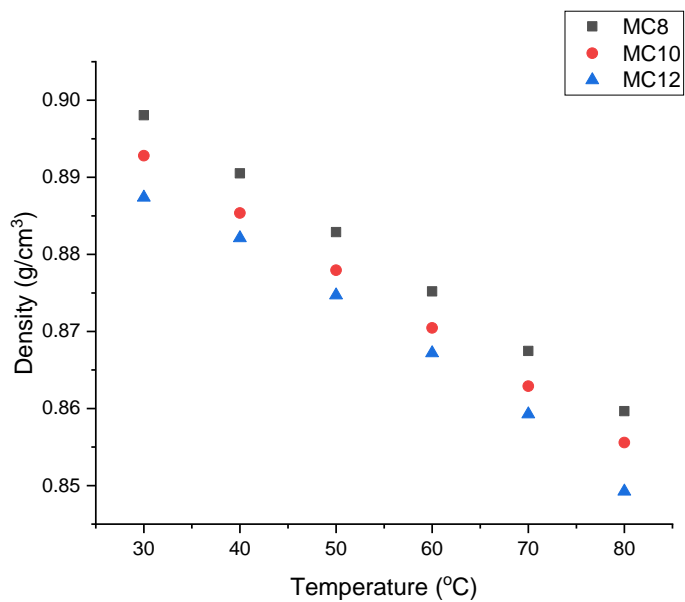


Figure 9: Density-temperature graph for Menthol based HDESs

Figure 10: Viscosity-temperature graph for Menthol based HDESs

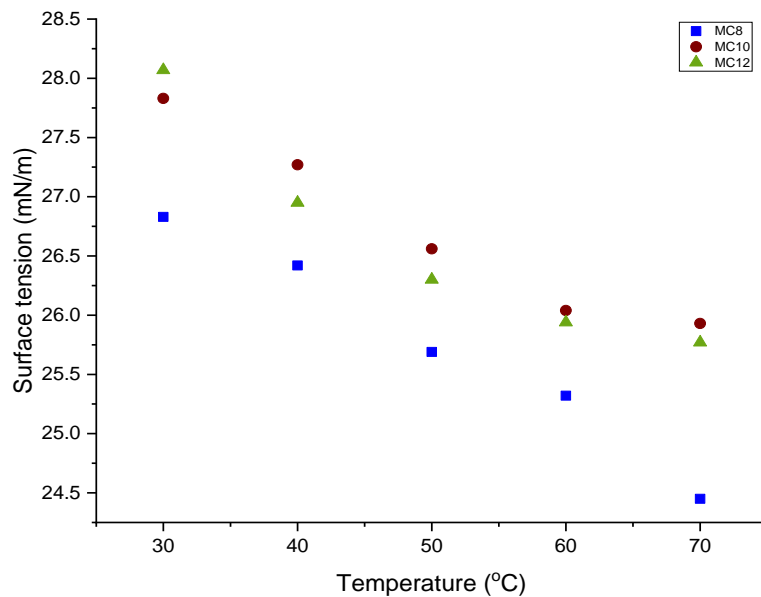


Figure 11: Surface tension-temperature graph for Menthol based HDESs

Test for hydrophobicity

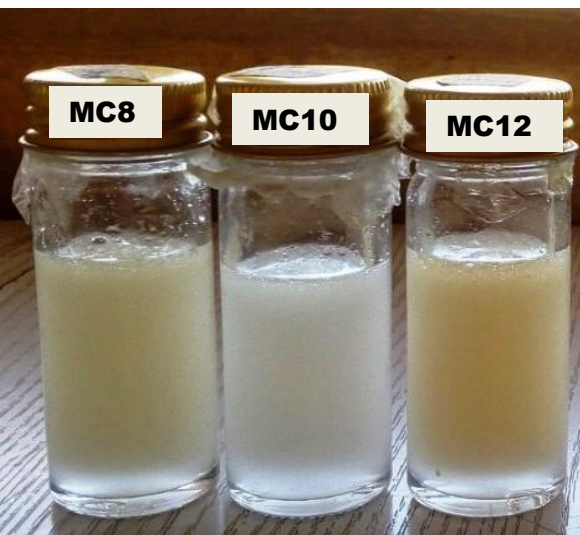


Figure 12: HDESs in water immediately after agitation

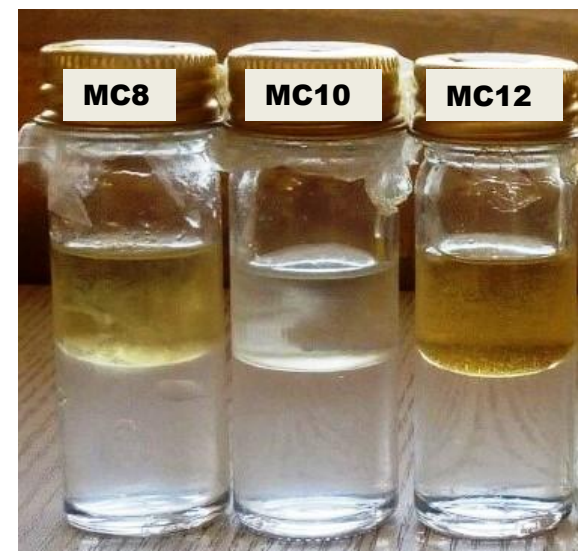


Figure 13: HDESs in water 24 hours after agitation

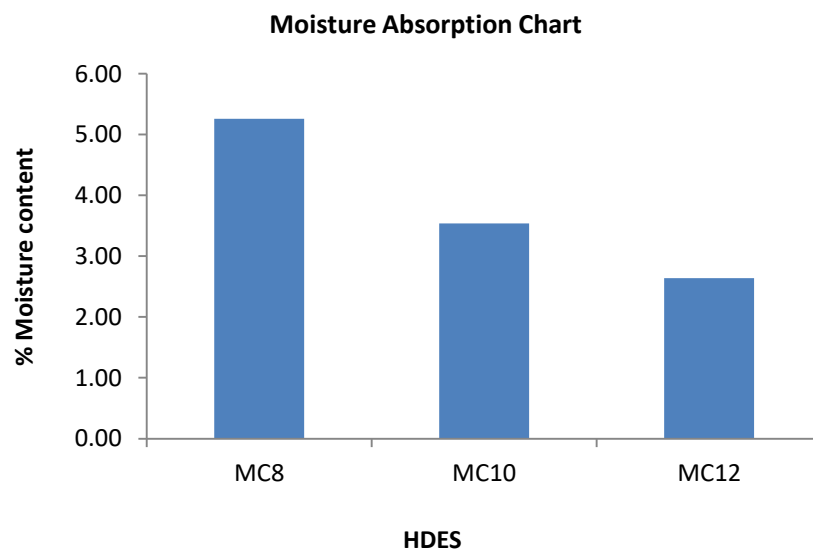


Figure 14: Percentage Moisture content graph for HDESs (Test for hydrophobicity)

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 0.890 – 0.925kg/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 2.64 – 5.26 %. The degree of hydrophobicity increased with an increase in the alkyl chain of the organic acids (HBDs).

THANK YOU FOR LISTENING!