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Evaluation and Characterisation of Composite mesoporous Membrane for Lactic acid Esterification

BY

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OUTLINE

- Introduction
- Ethyl lactate applications
- Cation-exchange resin for ethyl lactate
- Membrane phenomenon
- Innovation
- Carrier gas transport
- Membrane characterisation
- Methodology
- Results
- Conclusion and future work

INTRODUCTION

Ethyl lactate (EL) plays a major role as a green solvent and also a replacement for most petrochemical solvents. The production of ethyl lactate suffer a major draw-back such as low conversion and purity, which results in the production to be economically and technically uncompetitive. The use of membranes and heterogeneous catalysts for the selective removal of water from the reaction products to shift the equilibrium towards the higher yield of product have attracted a lot of attention.

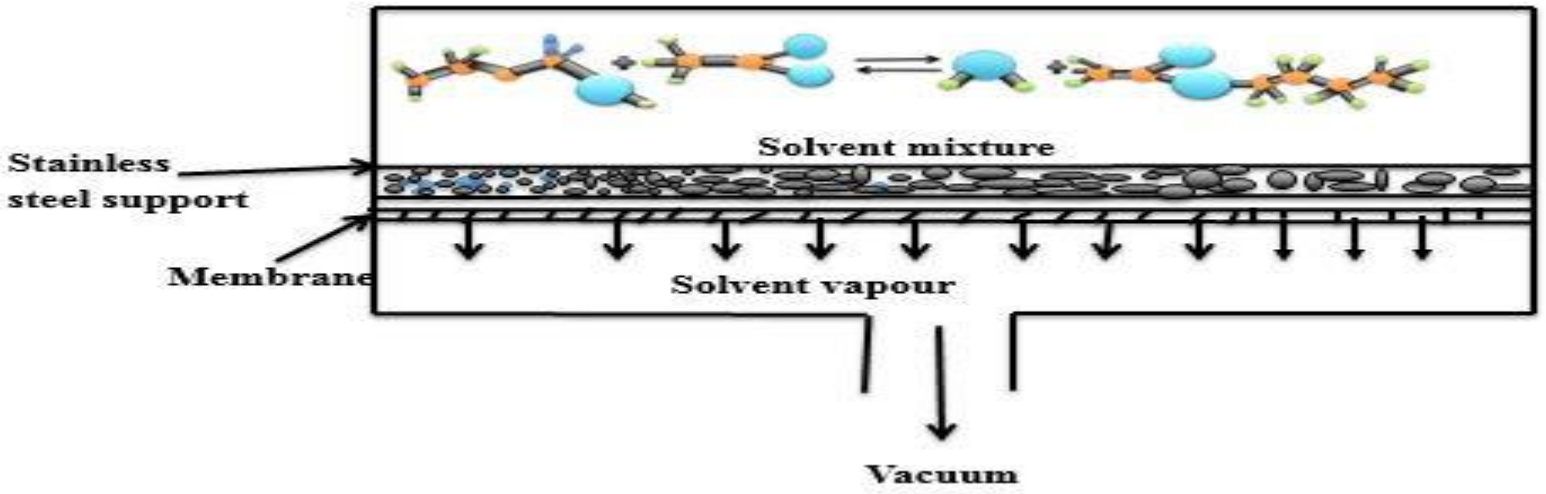


Fig. 1: Esterification process on a catalytic membrane

SOURCE OF ETHYL LACTATE

Ethyl lactate from renewable sources such as those from biomass feedstock. It is a biodegradable solvent with an excellent properties to be applied a green solvent in various applications including pharmaceutical, ink and coatings, food additives and organic synthesis.

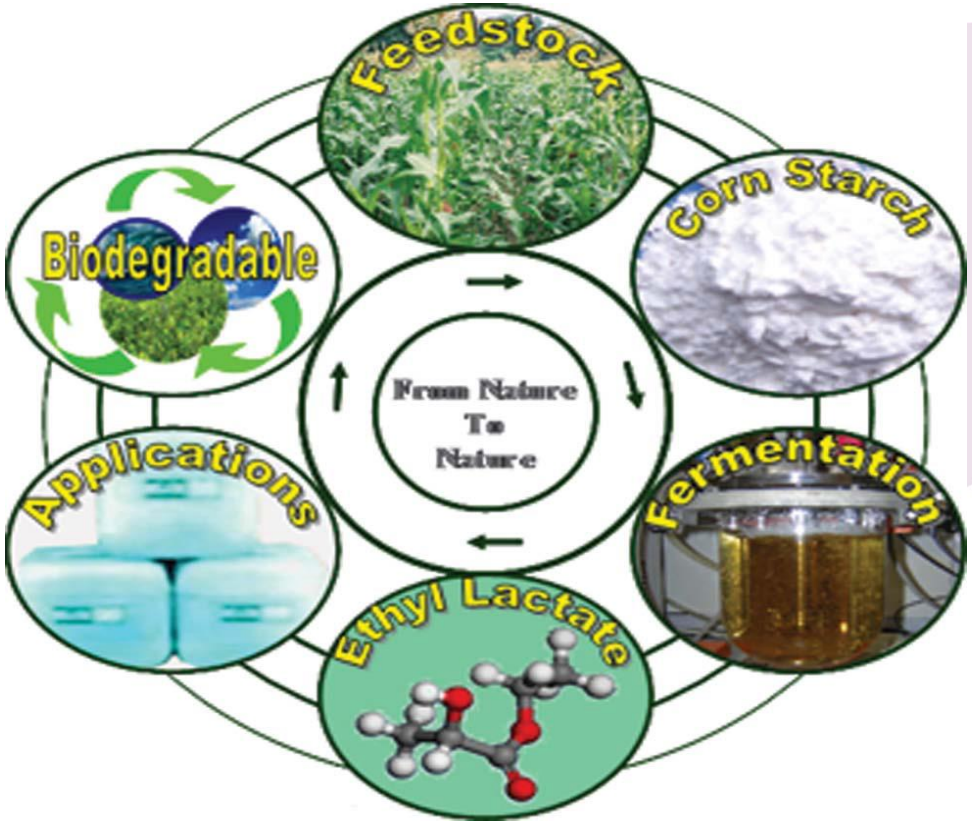


Fig 2: Ethyl lactate life cycle

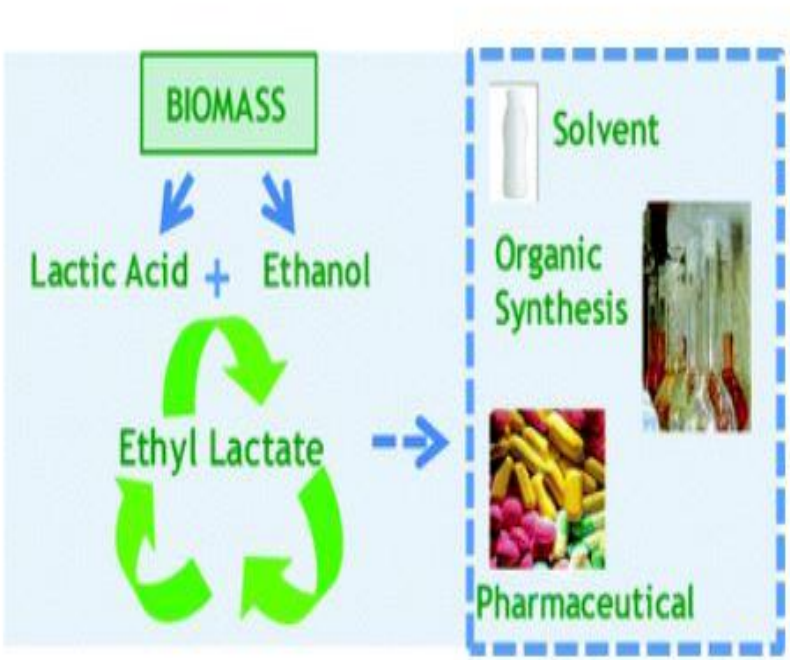


Fig. 3: Synthesis of Ethyl lactate

Application of Ethyl lactate by industries

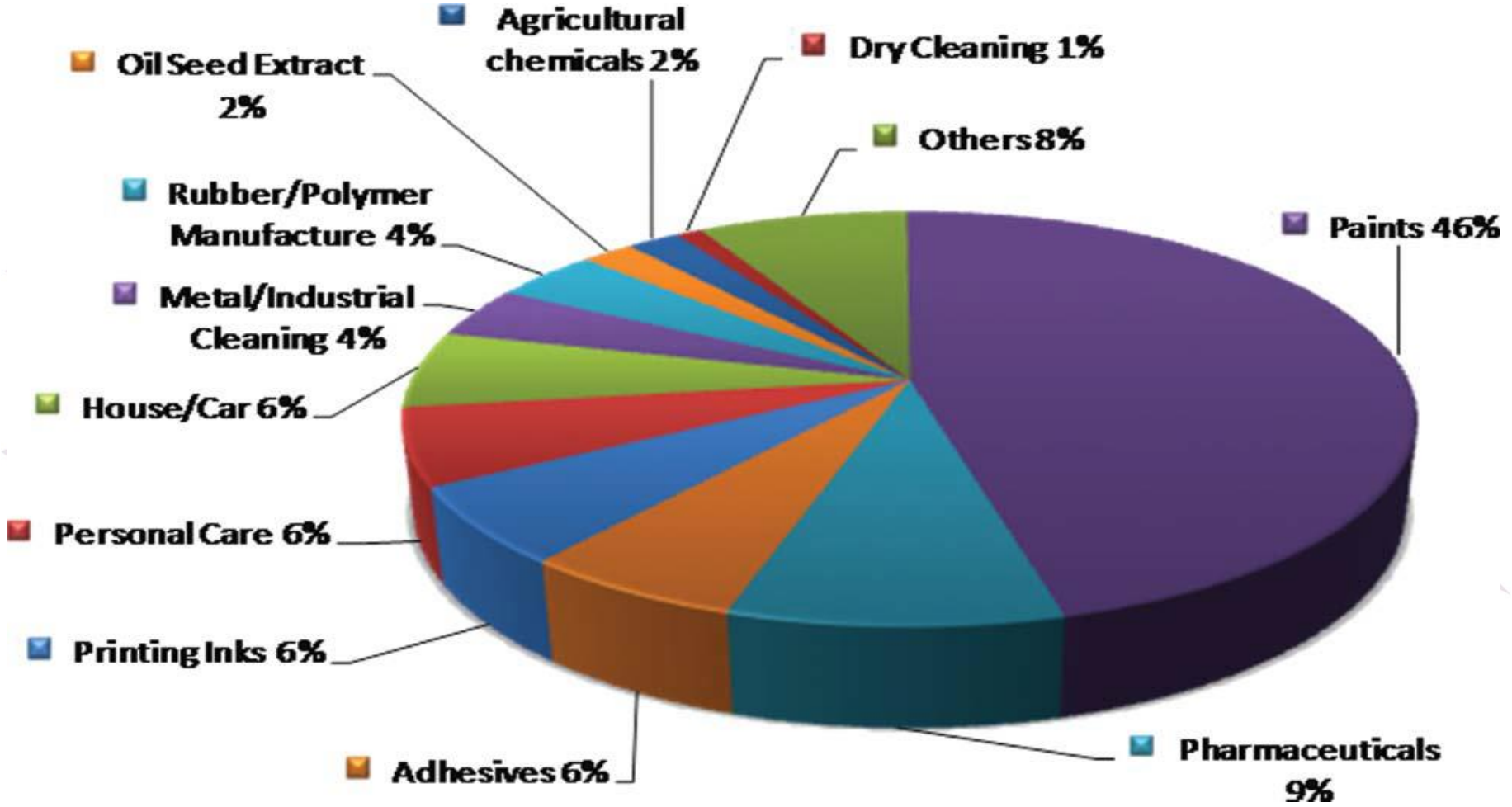


Fig 4: Schematic diagram of Ethyl lactate application by industries

Solid Catalysts for Ethyl lactate Synthesis

Heterogeneous catalysts

Cation-exchange resin

-Amberlyst 36
-Amberlyst 16
-Amberlyst 15
-Dowex 50W8x

Fig. 5: types of heterogeneous catalysts



Dowex 50W8x



Amberlyst 36



Amberlyst 16



Amberlyst 15

Fig. 6: Pictorial diagram of the different catalysts

Membrane Phenomenon

Membrane is a selective or physical barrier that reduces or prevent the mass transfer in a certain material. In the past decade membrane have been regarded as a separation agent. Membranes may be classified as organic or inorganic membranes . The different types of inorganic membrane include:

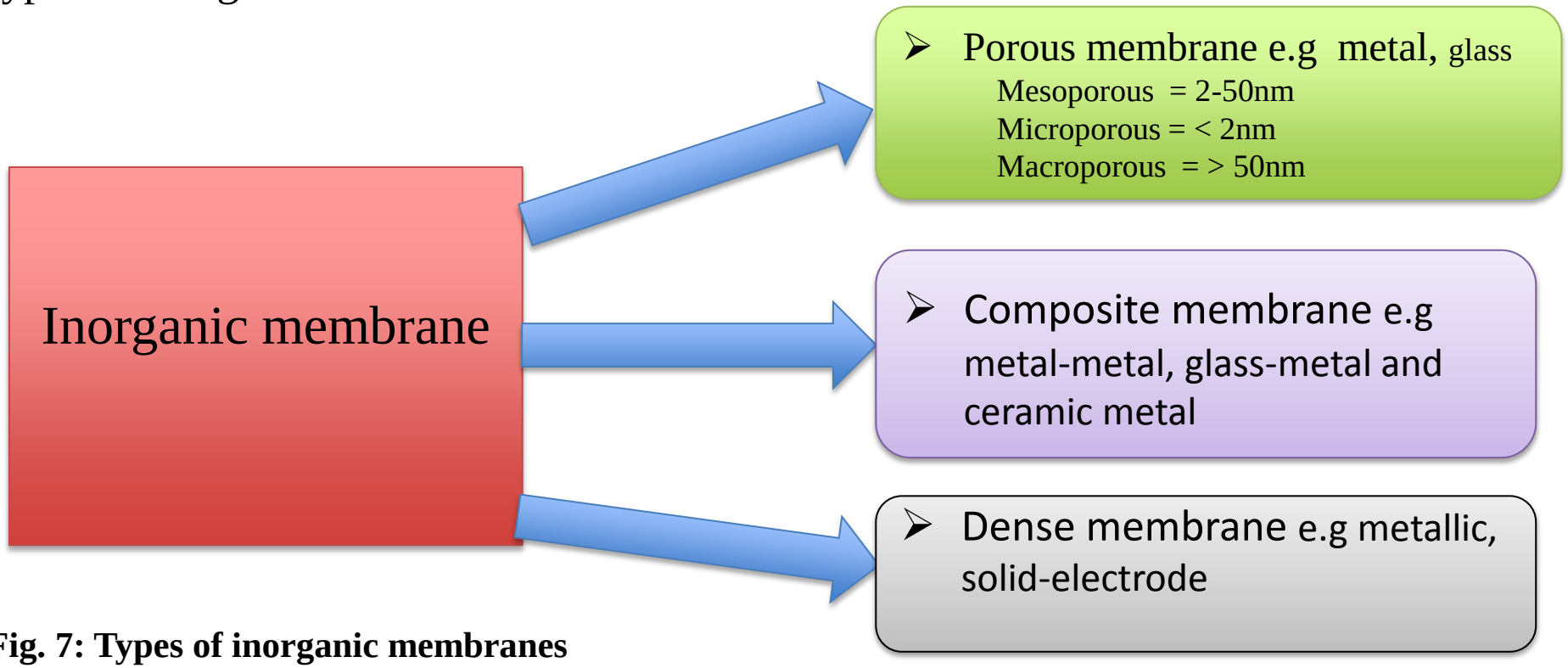


Fig. 7: Types of inorganic membranes

ADVANTAGES OF AN ENHANCED MEMBRANE

- High thermal stability
- Mechanical strength
- Chemical stability
- They allow heterogeneous catalyst

to be deposited easily on the surface.

- Increase in yield of product.
- Cost effective
- Long service life

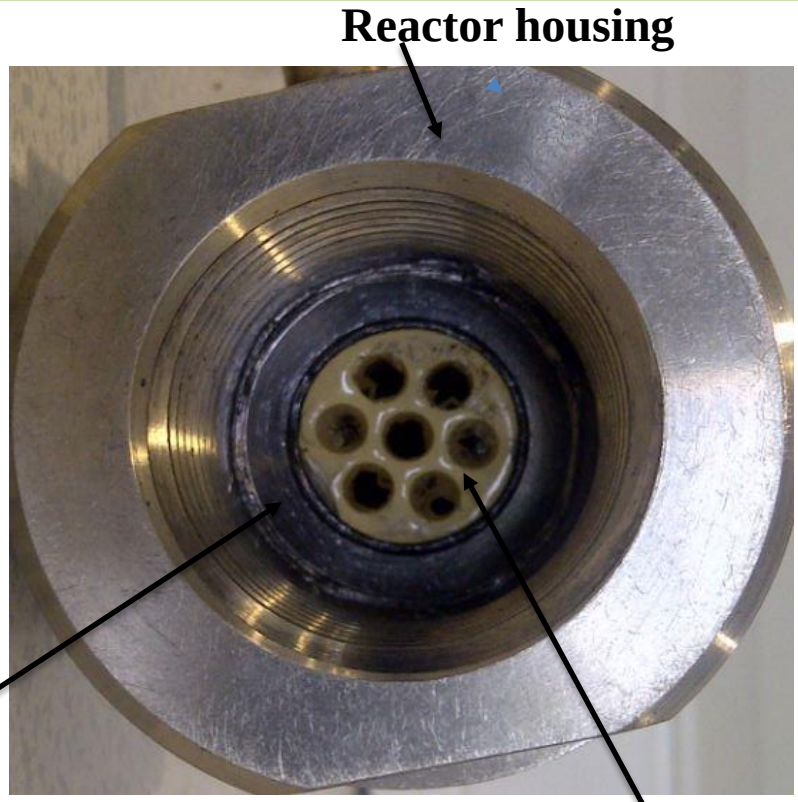


Fig. 8: Pictorial diagram of a porous membrane

INNOVATION

- To characterise resin and ceramic membrane pore size distribution using scanning electron microscopy.
- Transport properties of the inorganic composite ceramic membrane with carrier gases use for ethyl lactate separation.
- To determine the surface area and pore diameter of a mesoporous membrane using liquid nitrogen adsorption.
- To carry out batch process esterification of lactic acid and ethanol and analysed the product using gas chromatograph-mass spectrometer.
- Compare the BET surface area and BJH pore diameter results of the 1st and 2nd dip-coated membranes.

METHODOLOGY

Membrane dip-coating and Carrier Gas Permeation test

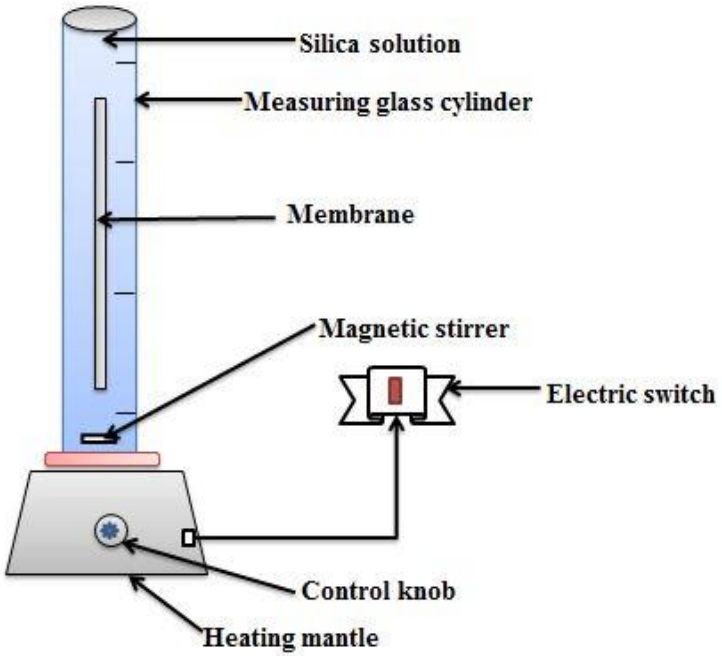


Fig. 9: Membrane dip-coating

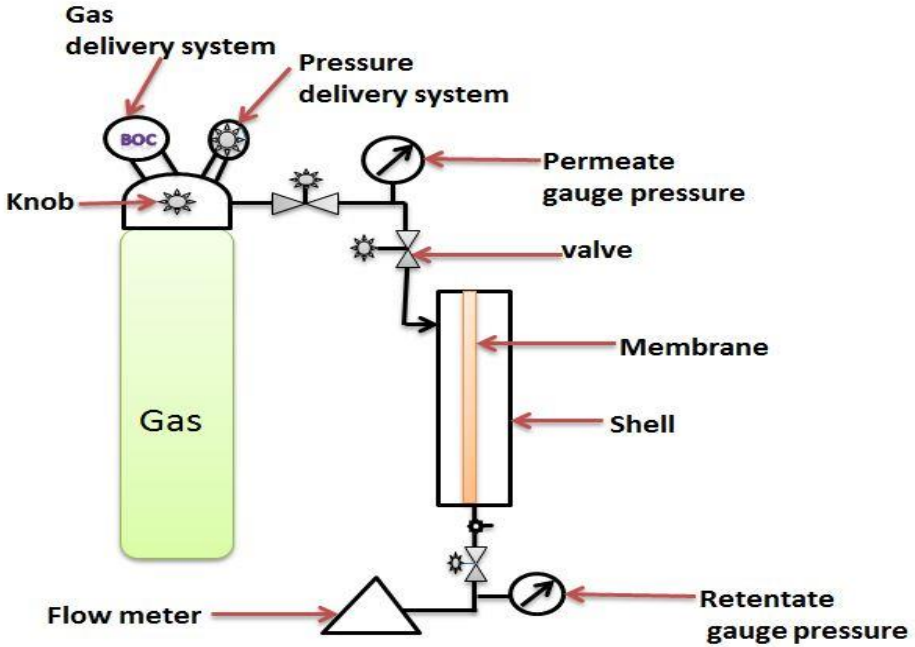


Fig. 10: Permeation test set-up

The four carrier gases used for the experiment include: Helium (He), Argon (Ar), Carbon dioxide (CO_2) and Nitrogen (N_2).



Fig. 11: Air drying of the membrane

Transport Mechanism in inorganic ceramic membrane

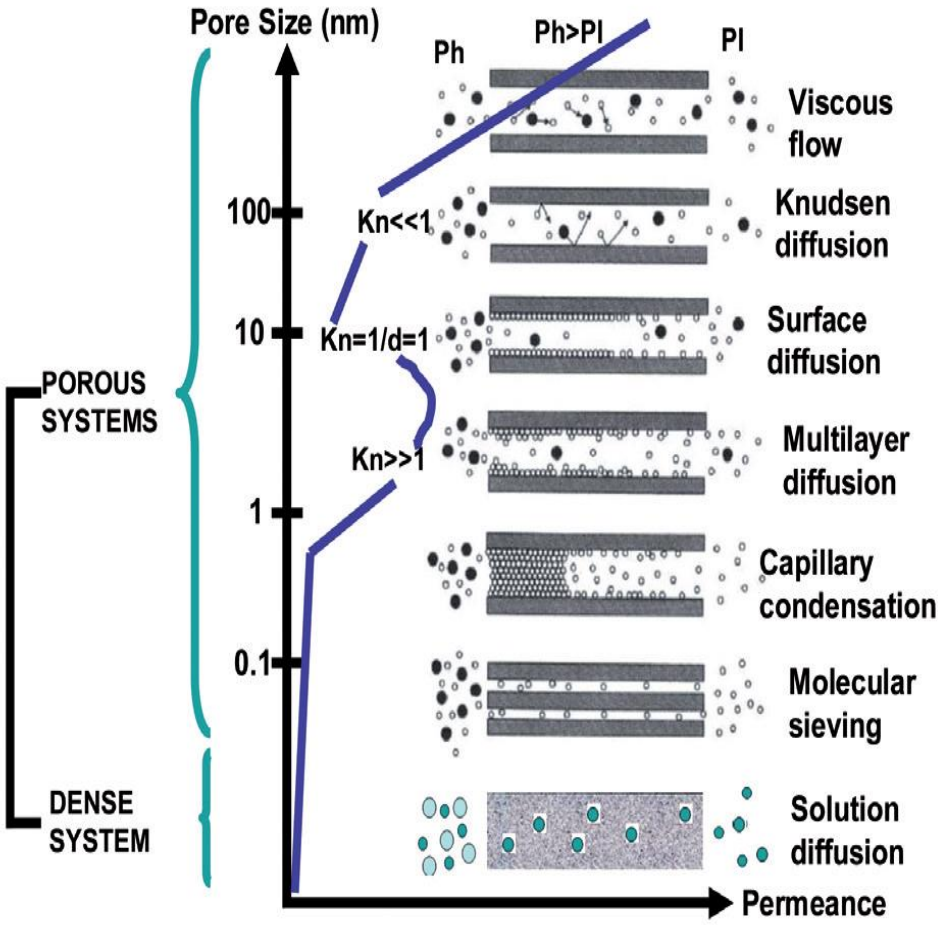


Fig. 12: Transport mechanism in ceramic membrane

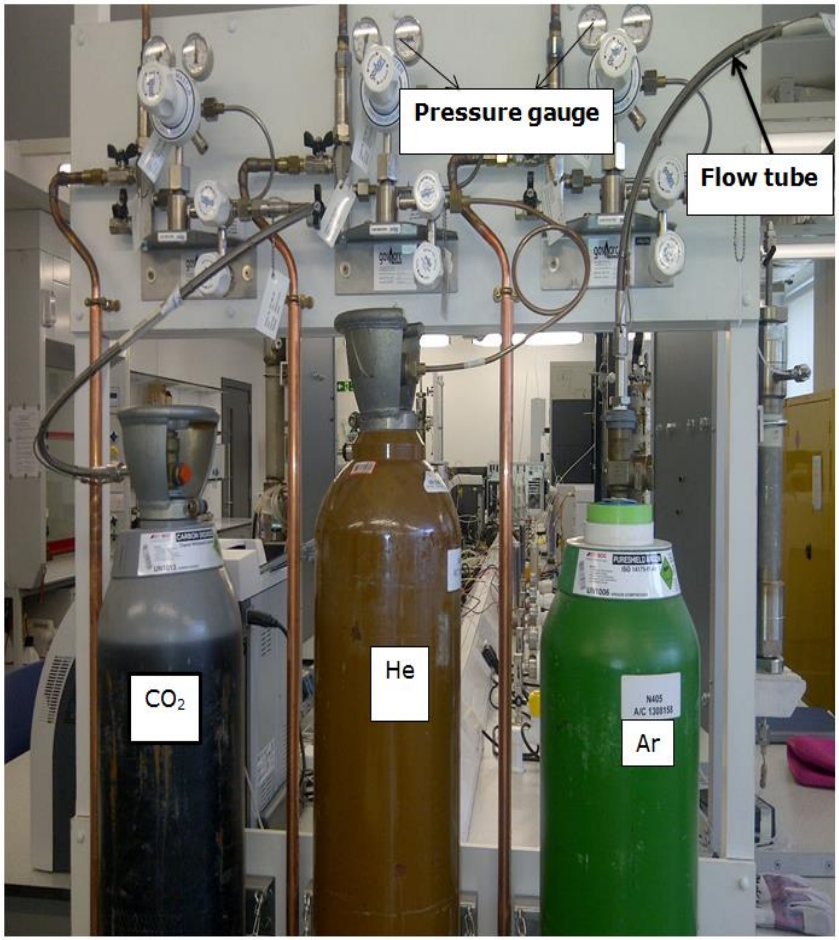


Fig. 13: Gas connection system

Batch Process Esterification Process

Batch process esterification process and GC-MS instrument for the analysis of esterification product

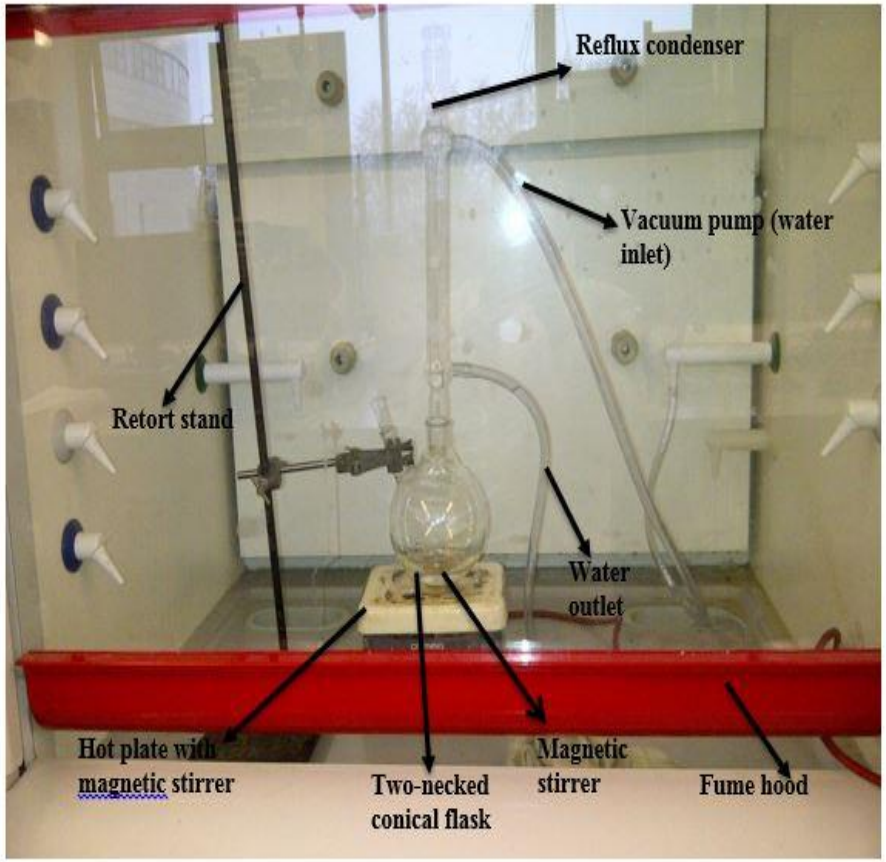


Fig. 14: Batch process set-up



Fig. 15: Auto sample GC-MS instrument

METHODOLOGY

Schematic diagram of ceramic membrane dip-coating technique for liquid nitrogen

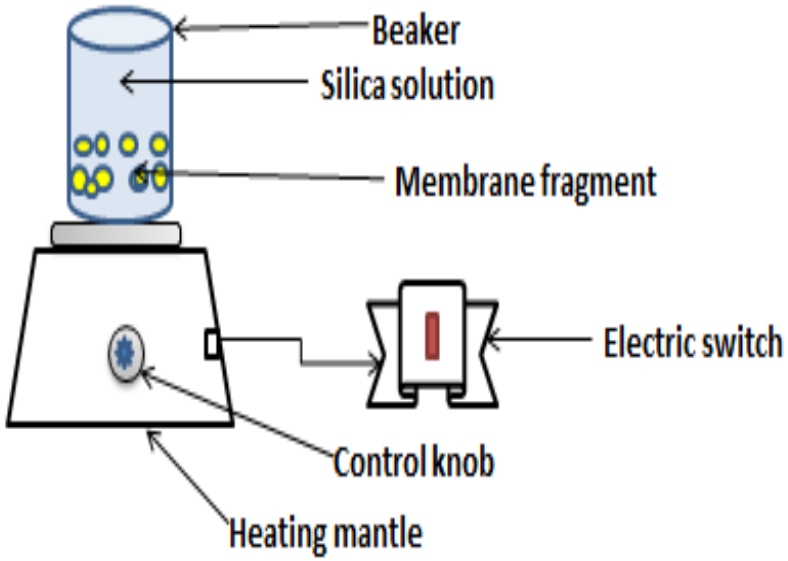


Fig. 16: membrane fragment dip-coating

The sample was found to be expand after the modification process



Fig. 17: Before modification



Fig. 18: After modification

LIQUID NITROGEN ADSORPTION

Sample Preparation before the degassing process

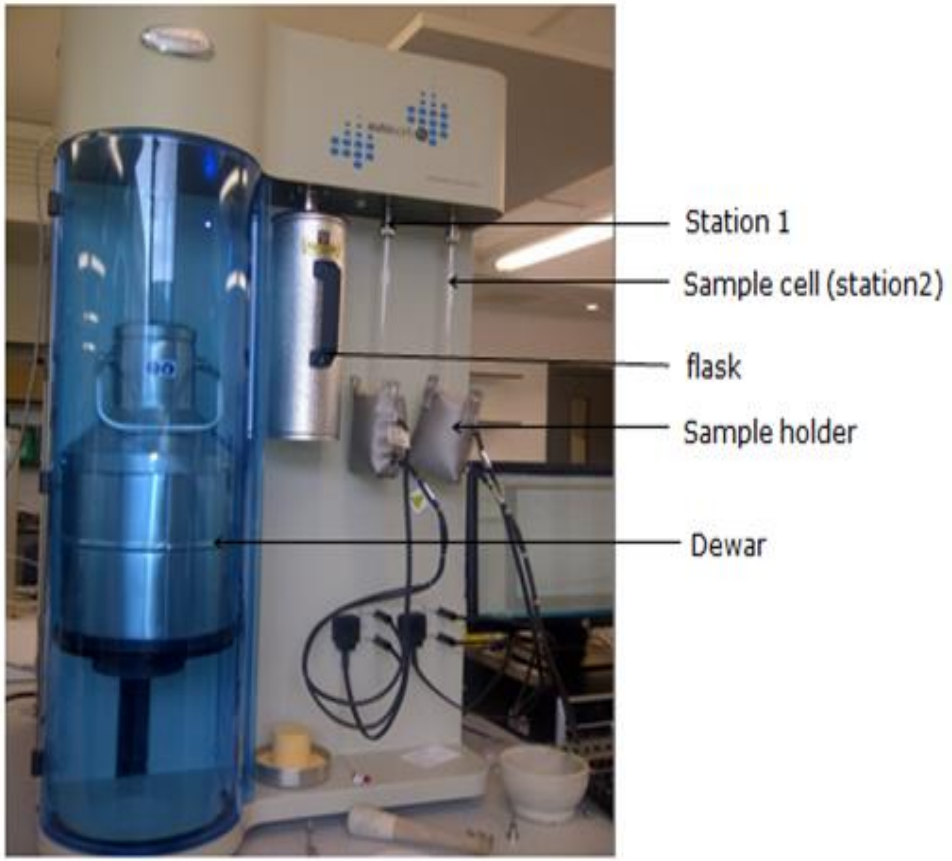


Fig. 19: Liquid nitrogen adsorption

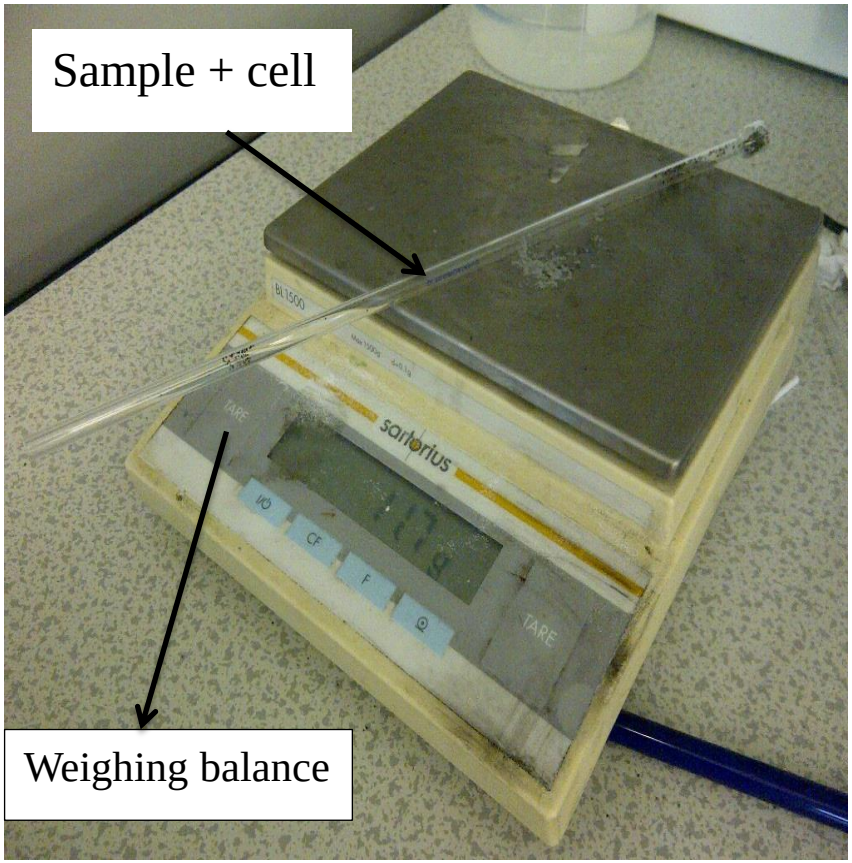


Fig. 20: Analytical weighing balance

PROPERTIES FOR NITROGEN ADSORPTION MEASEUREMET

Different properties of membrane that could be measured with Gas Adsorption

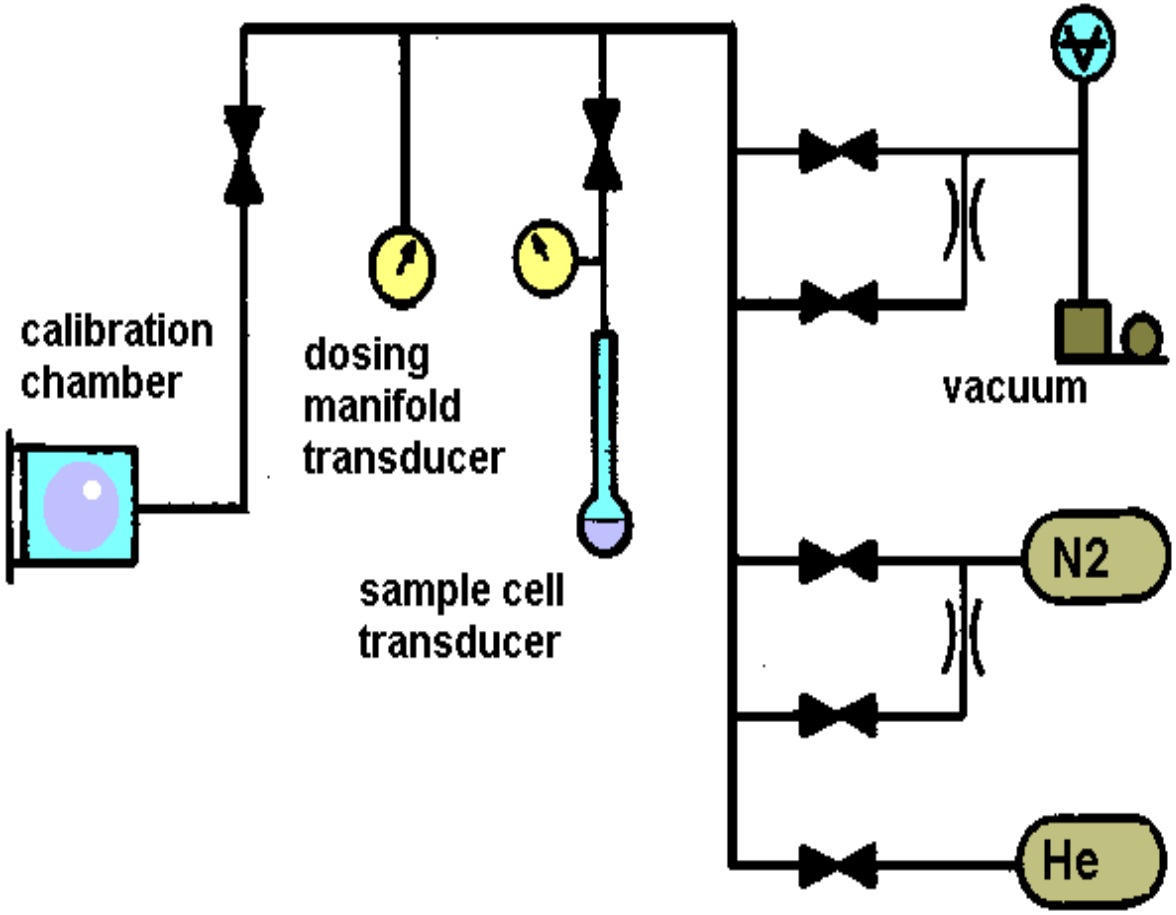


Fig. 21: Typical vacuum apparatus

- Size and Shape
- Surface area
- Pore size
- Pore volume
- Porosity
- Density

The degassing temperature was set at 65 ^{o}C for 2 hrs prior to the sample analysis. The liquid nitrogen temperature was 77 K.

Nitrogen Adsorption Results for the silica membranes at 77 K

BET Curves for 1st and 2nd dip-coated membrane

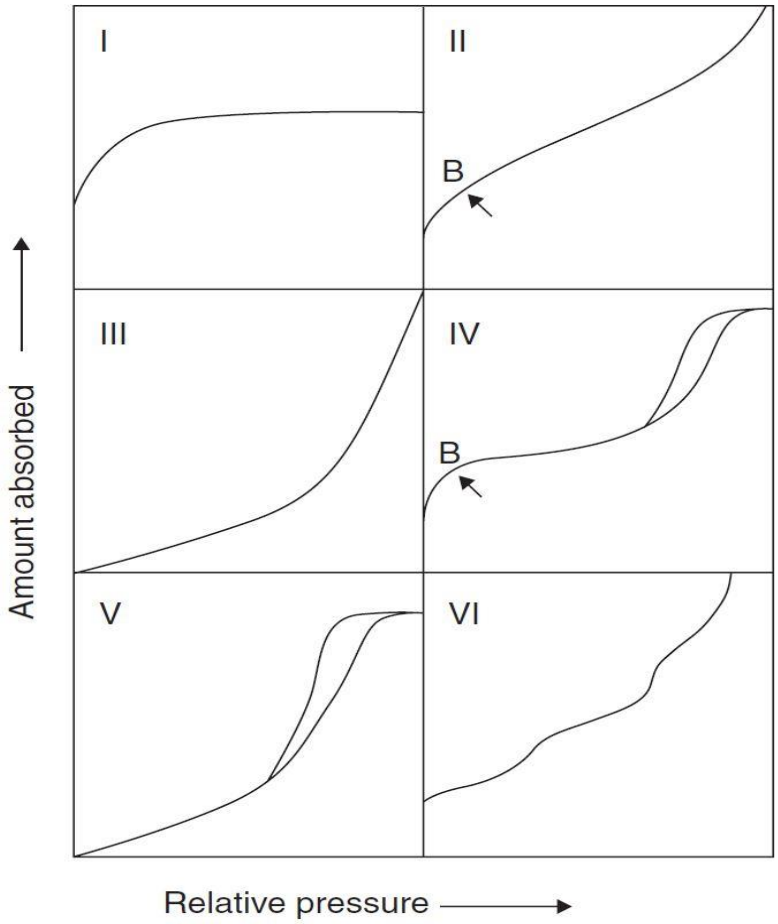


Fig.22: Classification of adsorption isotherm

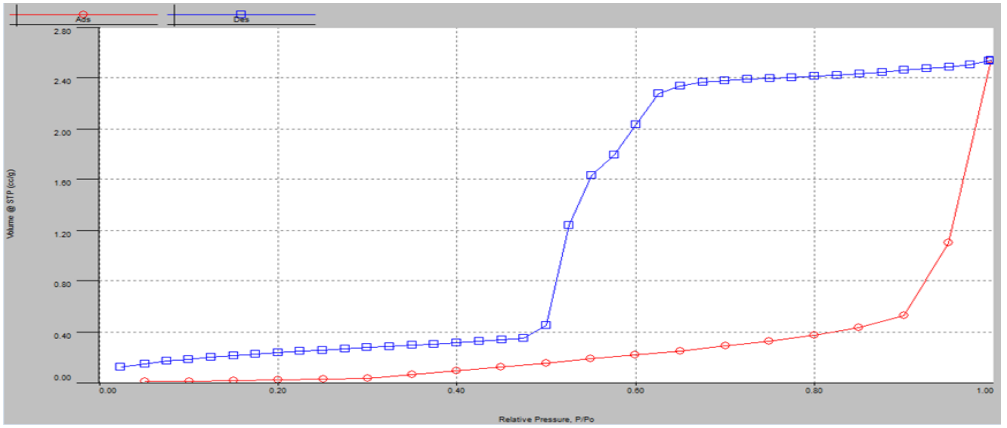


Fig. 22a: BET curve for 1st dip-coated membrane

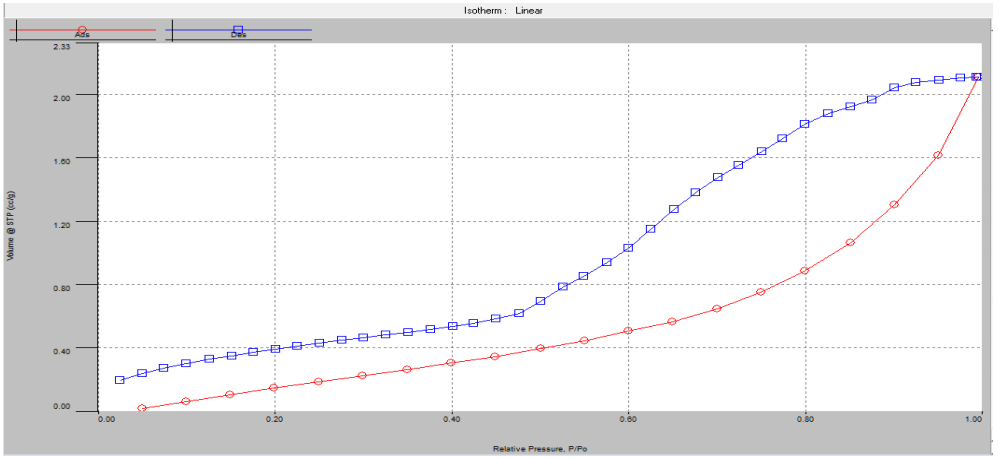


Fig. 23b: BET curve for 2nd dip-coated membrane

Nitrogen Adsorption Results for the silica membranes at 77 K

BJH Curves for 1st and 2nd dip-coated membrane

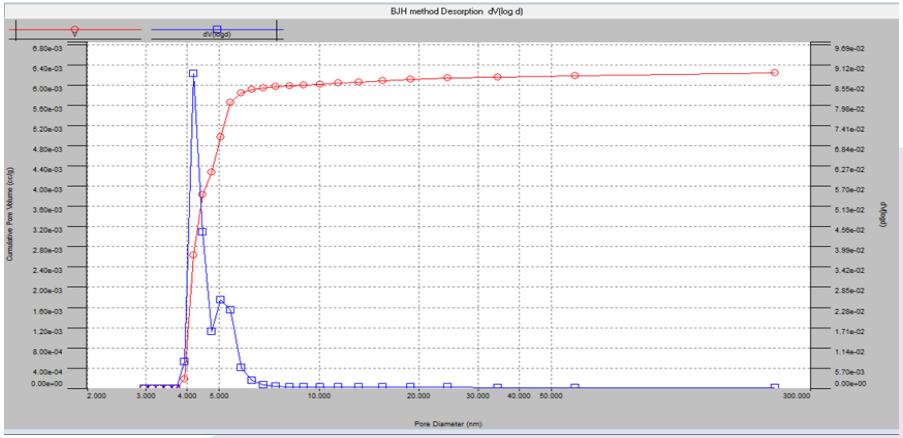


Fig. 24a: BJH curve for 1st dip-coated membrane

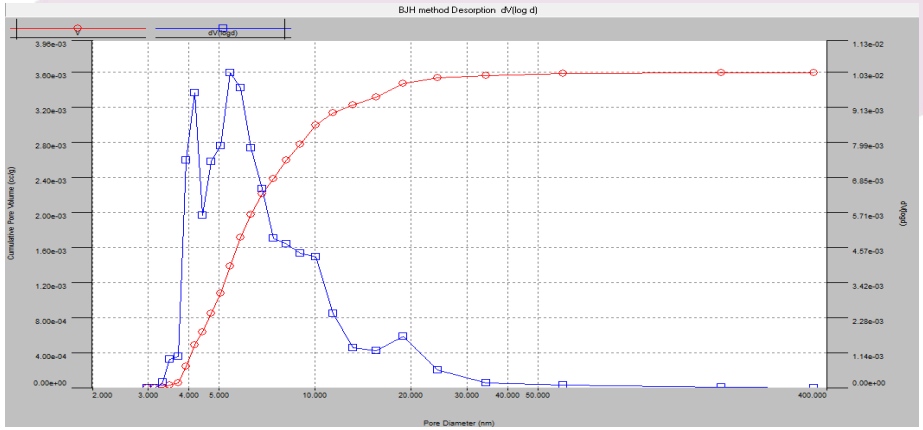


Fig 24b: BJH curve for 2nd dip-coated membrane

Sample	BET Surface area(m^2/g)	BJH Pore diameter(nm)	Pore Volume (cc/g)
1st dipping	0.253	4.184	0.006
2nd dipping	1.497	4.180	0.004

Table 1: BET and BJH of the membranes

The BET surface area of the dip-coated silica membrane showed a type IV isotherm characteristic of mesoporous structure with hysteresis. The BJH curves of the silica membrane was in accordance with mesoporous classification

GC-MS Results

Catalysts	Retention time (min)	Peak Area ()
Amberlyst 15	1.521	149055537.3
	4.169	1761909.18
	7.738	4826559.45
	9.218	113978453.4
Amberlyst 16	1.503	119782347.8
	4.143	9286786.17
	8.731	283366480.1
	10.502	23075482.13
Amberlyst 36	1.527	138638054.1
	3.628	98634990
	7.975	97944520.42
	8.726	121332143.6
	10.490	14091694.59
Dowex 50W8x	1.523	142522149.5
	3.627	78276693.15

Table 2: Retention time and peak area of catalysts.

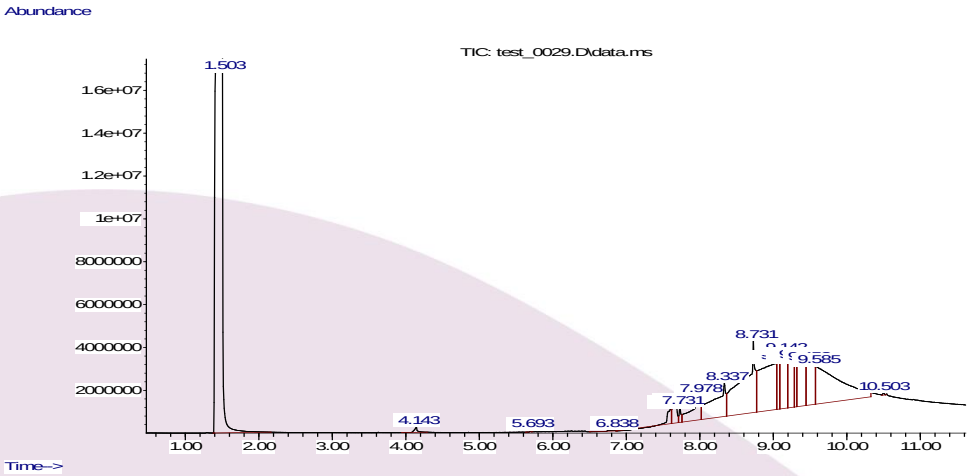


Fig. 25: Ion chromatogram of the esterification product

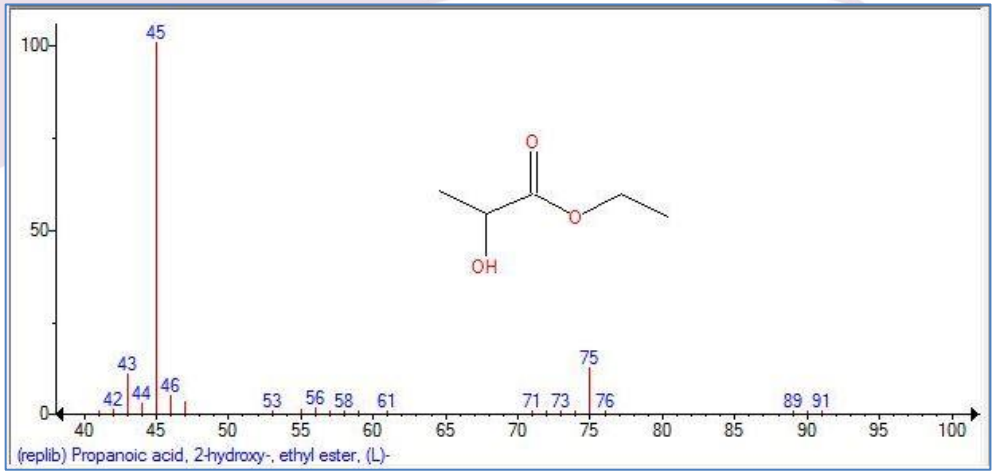


Fig. 26: NIST spectra for ethyl lactate

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SEM/EDXA Results

SEM/EDXA surface image of the membrane

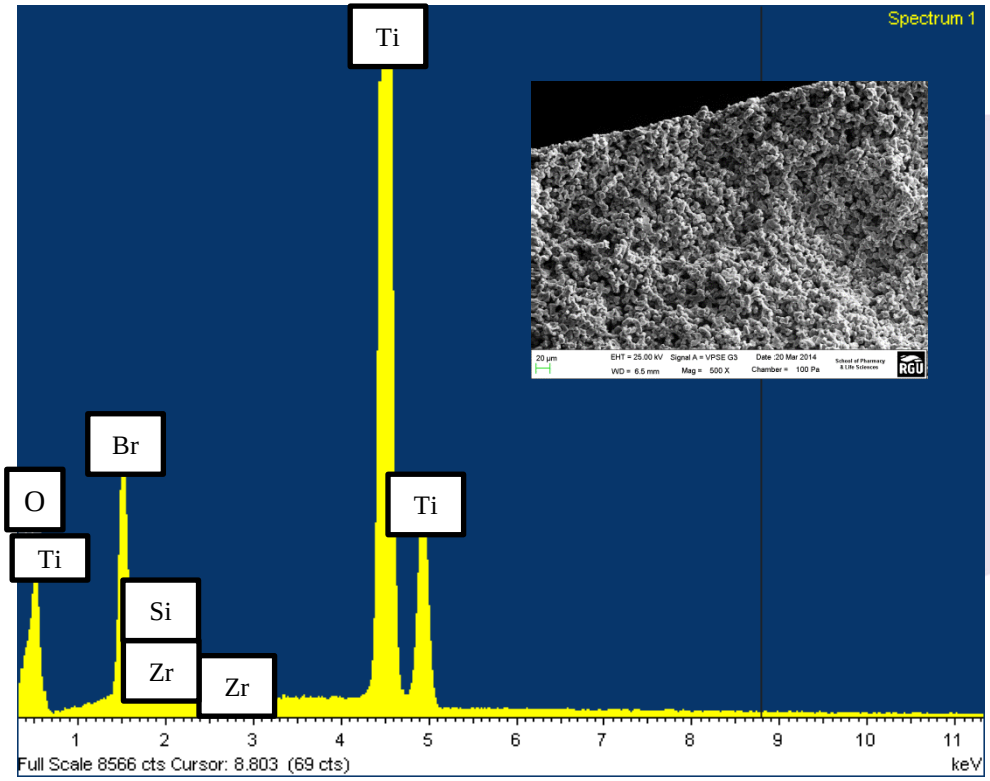


Fig. 27: EDXA spectra of the membrane



Fig 28: SEM/EDXA instrument

The membrane showed a defect-free surface with no evidence of crack on the surface of the silica membrane

PERMEATION TEST RESULTS

Relationship between gas flow rate and gauge pressure

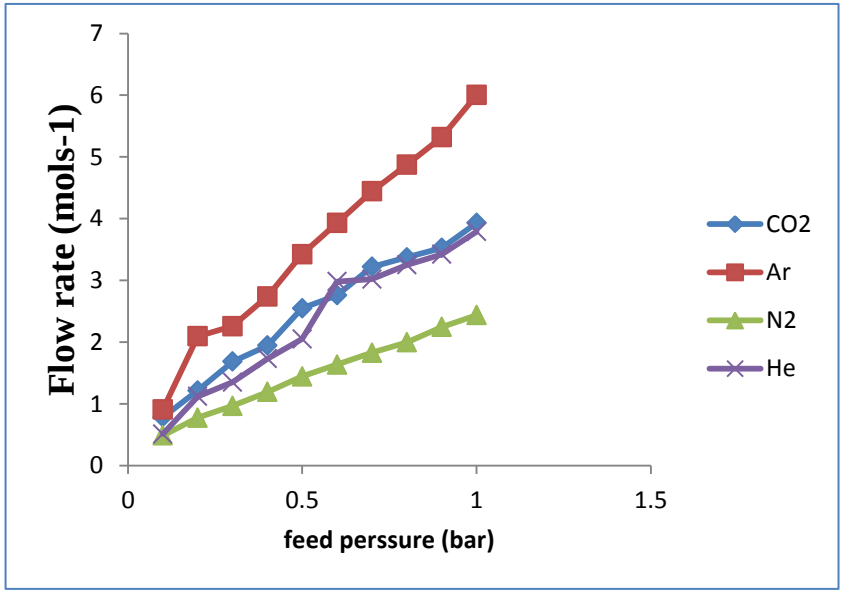


Fig. 29: Flow rate relation with pressure

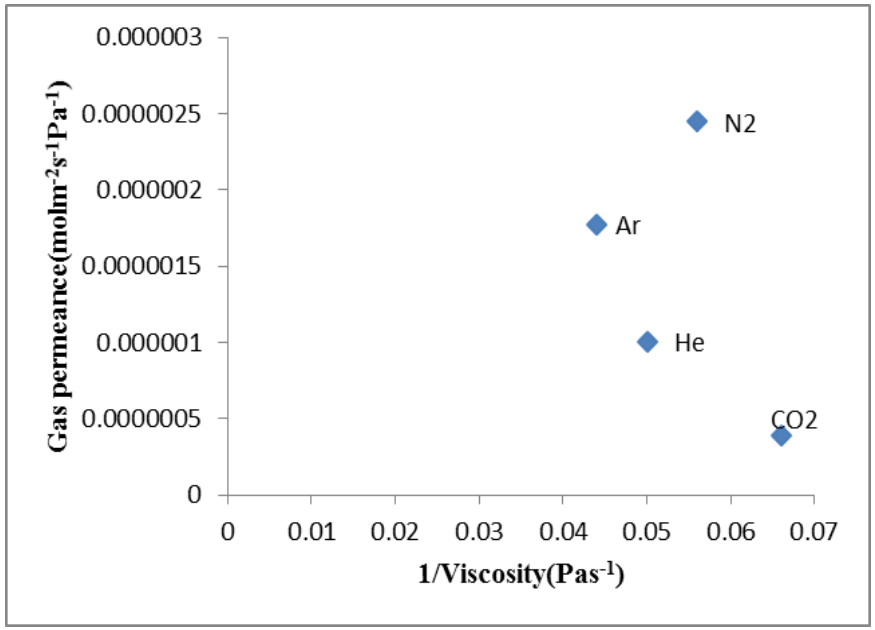


Fig. 30: Permeance relation with viscosity

The gas flow rate exhibited an increase with gauge pressure indicating Knudsen flow mechanism. The order of the gas kinetic diameter from the highest is given as: N_2 > Ar > CO_2 > He.

The gas permeance relationship with viscosity was in agreement with the viscous flow mechanism.

CONCLUSION

- The evaluation and characterization of composite mesoporous membrane for lactic acid Esterification was successfully carried out using different methods including nitrogen adsorption, FTIR, GC-MS and SEM/EDXA.
- Helium gas with the higher permeation rate was used as one of the carrier gas for the analysis of esterification product.
- The SEM surface image of the membrane showed a defect-free surface.
- The NIST mass spectra of the esterification reaction product exhibited the structure of ethyl lactate on the spectra.
- The BET surface area of the composite membrane showed a type IV isotherm with hysteresis indicating a mesopourous layer.
- The BJH curve for the composite membrane was in accordance with the mesoporous classification

FURTHER WORK

Further work will be carried out to investigate the following:

- To carry out permeation for the 3rd dip-coated membrane at different temperatures in contrast to the 4th dipping for further analysis of carrier gas with membrane.
- To compare the specific surface area and pore diameter of the ceramic membrane with subsequence coating using Liquid nitrogen adsorption.
- To carry out ethyl lactate production with flat sheet cellulose acetate membrane at different temperatures.
- Quantify the esterification reaction product and correlate the experimental data.

Thanks for your Attention
Question?

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