

(Deemed to be University) (Under Section 3 of UGC Act 1956)

KARPAGAM ACADEMY OF HIGHER EDUCATION

(Deemed to be University Established Under Section 3 of UGC Act 1956)

Coimbatore – 641 021.

SYLLABUS

DEPARTMENT OF CHEMISTRY

Semester-IV

16CHU414A GREEN METHODS IN CHEMISTRY PRACTICAL 3H 1C

Instruction Hours/week:L:0 T:0 P:3 Marks: Internal: 40 External: 60 Total:100

Scope

This lab course deals with the experiments which involve techniques used in green chemistry

Objectives

This course enable the student to

1. Understand the principles and the practical aspects of green chemistry

Practical's

- 1. Preparation and characterization of biodiesel from vegetable oil.
- 2. Extraction of D-limonene from orange peel using liquid CO2 prepared from dry ice.
- 3. Mechano chemical solvent free synthesis of azomethine.
- 4. Solvent free, microwave assisted one pot synthesis of phthalocyanine complex of copper(II).

Suggested Readings

Text Books:

- 1. Anastas, P.T. & Warner, J.K. (2005). *Green Chemistry- Theory and Practical*. Oxford University Press.
- 2. Matlack, A.S. (2001). Introduction to Green Chemistry. Marcel Dekker
- 3. Cann, M.C. & Connely, M.E. (2000). *Real-World cases in Green Chemistry*, American Chemical Society. Washington.

Reference Books

- 1. Ryan, M.A. & Tinnesand, M. (2002). *Introduction to Green Chemistry*. American Chemical Society. Washington.
- 2. Lancaster, M.(2010). *Green Chemistry: An introductory text.* 2nd Edition. RSC publishing.



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LAB MANUAL

Green methods in Chemistry Practical (16CHU414A)

- 1. Preparation and characterization of biodiesel from vegetable oil.
- 2. Extraction of D-limonene from orange peel using liquid CO2 prepared from dry ice.
- 3. Mechano chemical solvent free synthesis of azomethine.
- 4. Solvent free, microwave assisted one pot synthesis of phthalocyanine complex of copper (II).

PREPARATION AND CHARACTERISATION OF BIO DIESEL FROM VEGETABLE OIL Aim:

To prepare and characterized the bio diesel from vegetable oil.

Principle:

Materials required:

- 1. Sunflower oil 6 lit
- 2. NaOH 22.8 g
- 3. Methanol 1.2 lit

4. Beaker &stirrer

Procedure:

- Weighed 6 kg of vegetable oil (Refined sunflower oil) and poured into the reactor for preliminary heating to Temperature of about 60-70 °c.
- In separate container, dissolve 22.8 g of NaOH (3.8 g/lit of oil, got by 3.5 g of stiochiometric equivalent and 0.3 g for neutralizing FFA) in 1.2 lit methanol (200 ml/ lit of oil) add the NaOH slowly. This combined mixture makes sodium ethoxide.
- Add this to the vegetable oil and provide vigorous mixing with the use of stirrer.
- The cloudy looking FFA, called glycerine will sink to the bottom and the methyl ester (a translucent) will remain on the top.
- Let the mixture settled over night and liquid on top will take over to the any remaining soups or salts which could cause engine damage to be removed.
- The glycerine which has sunk to the bottom can be used in the production of cosmetics.

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Calculation:

Calculation of vegetable oil

1. pH =

2. Density:

e = M/V

Where,

 $M-mass \ of the vegetable \ oil$

 $V-volume \ of the vegetable \ oil$

3. Viscosity:

n = ket

Where,

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k- viscometer constant (1.3)
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- e- density of the vegetable oil
- t- time consumption for

4. Specific gravity:

= density of the vegetable oil/

Calculation for bio diesel

1.pH =

2. Density:

e = M/V

Where,

 $M-mass\ of\ the\ bio-diesel$

V- volume of the bio- diesel

3. Viscosity:

n = ket

Where,

k- Viscometer constant (1.3)

e- density of the bio-diesel

t- time consumption for

conventional process

4. Specific gravity:

= density of the bio diesel / density of water

Result:

The bio diesel from vegetable oil is prepared successfully and calculated.

EXTRACTION OF d- LIMONENE FROM ORANGE PEEL

Aim:

To extract d-limonene from orange peel

Principle:

Limonene as two isomers namely d-*limonene* and l-limonene,where d-limonene as a citrus smell. A citrus peels such as orange, pine, conifer as citrus smell. Essential oil found in d-limonene and which as important function in plants repulsing fungi and pest. In fruits essential oil attract fruit eating animals that help dispose the seeds. d-limonene (1-methyl-4-prop-1-ene-zyl-cyclohexane) is an unsaturated hydrocarbon classified as terpene. The room temperature d-limonene exhibit colourless oily liquid with smell of orange. The molecular formula for d-limonene is C10 H6 and the boiling point is 176°c.

Procedure:

- Orange peel can separated into or crushed into the pith.
- A white pith contains negligible amount of orange oil and it is discorded. The outer orange part of the orange peel also contains the oil.
- d-limonene is a stable terpene it is extracted through steam distillation withoutdecomposing. This maintains the structure of d-limonene molecule and contains usual purity.
- Hot steam open the pocket of orange peel allowing volatile orange oil to be released. As the temperature increases, these volatile material vaporized on the cooler surface of condenser. Since limonene is volatile, it can be vaporized at the temperature lower than its boiling point. Crush the liquid collected from the steam distillation is a mixture of water and limonene.
- The extract test with the drop of bromine water. If colour does not change it contains the extract of d-limonene.

Calculation:

Empty beaker weight(a)	=	g	
Oil + beaker weight (b)	=	g	
Orange oil weight (b-a)	=	g	
Final mass of orange oil	=	g	
Result:			
(i) Intial mass of orange Peel(a)		=	
(ii) Final mass of orange oil(b)		=	
(iii) Percentage of yield		=	b/a × 100

SOLVENT FREE SYNTHESIS OF AZO-METHINE (SCHIFF'S BASE)

Aim:

To prepare azo-methine by using solvent free synthetic method.

Principle:

A chief's base are prepared from benzaldehyde and substituted aniline by using microwave irradiation in appropriate time. It is absorbed that a condensation between carbonyl compound and aniline.

Materials required:

- 1. Benzaldehyde
- 2. Aniline
- 3.50 ml beaker
- 4. Ethanol
- 5. Ice water
- 6. Microwave oven

Procedure:

A mixture of aniline and benzaldehyde is taken in a 50 ml beaker and mixed well. This mixture irradiated in microwaveoven at a power of 160 waltz for a specific time. The reaction after completion, the mixture is poured into the ice water. The yellow solid precipitate obtained was filtered, washed, dried and recrystallised from ethanol.

Result:

The yield of prepared Schiff's base under microwave conditions = g