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SYNTHESIS & CHARACTERIZATION OF 9, 10-DI (P-METHOXY PHENYL)-2-METHYL ANTHRACENE

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ABSTRACT

Polyacenes belong to a class of polycyclic aromatic hydrocarbon (PAH) compounds which are planar sets of linearly fused benzene rings with the general formula $C_{4n}+_2H_{2n}+4$ or $C_2H_4(C_4H_2)m$. The design and synthesis of polymer are based on the ether linkage on the backbone provides a means of increasing the thermal stability and adjusting the length of conjugation. Thus designed and synthesis of highly stable and luminescent-form of derivative of anthracene is the main aim of this research project.

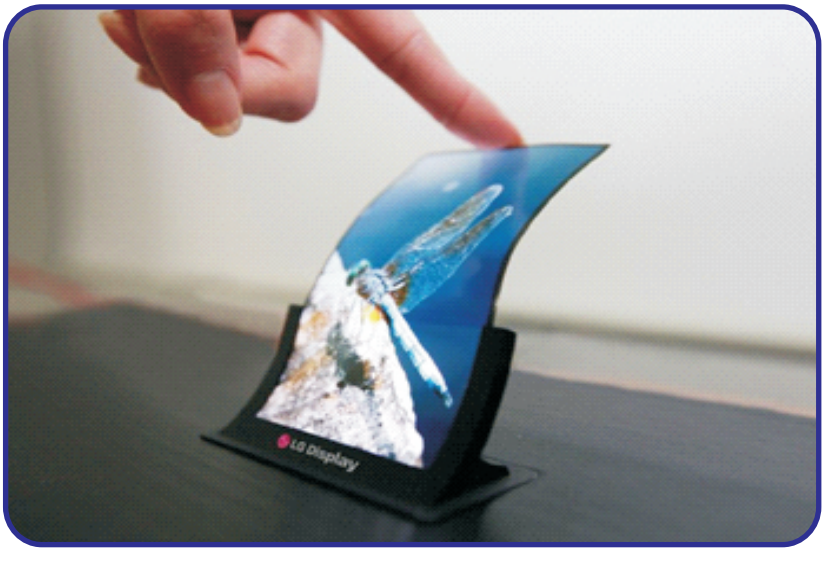
KEYWORDS: Polyacenes, polycyclic aromatic hydrocarbon (PAH).

INTRODUCTION:

Light emitting polymer is a polymer that emits light when a voltage is applied to it. LEP are used to manufacture light weight, ultra thin display for mobiles, TV screen, and computer screen. LEP are plastic materials that convert electrical power into visible light. This is similar to fluorescence of polymers where U.V. radiation is converted into visible light.

Plastic material with metallic and semi-conductor properties are called as “Conducting polymers’’.

E.g. Polyfuran, Polypyrrole, Polyaniline



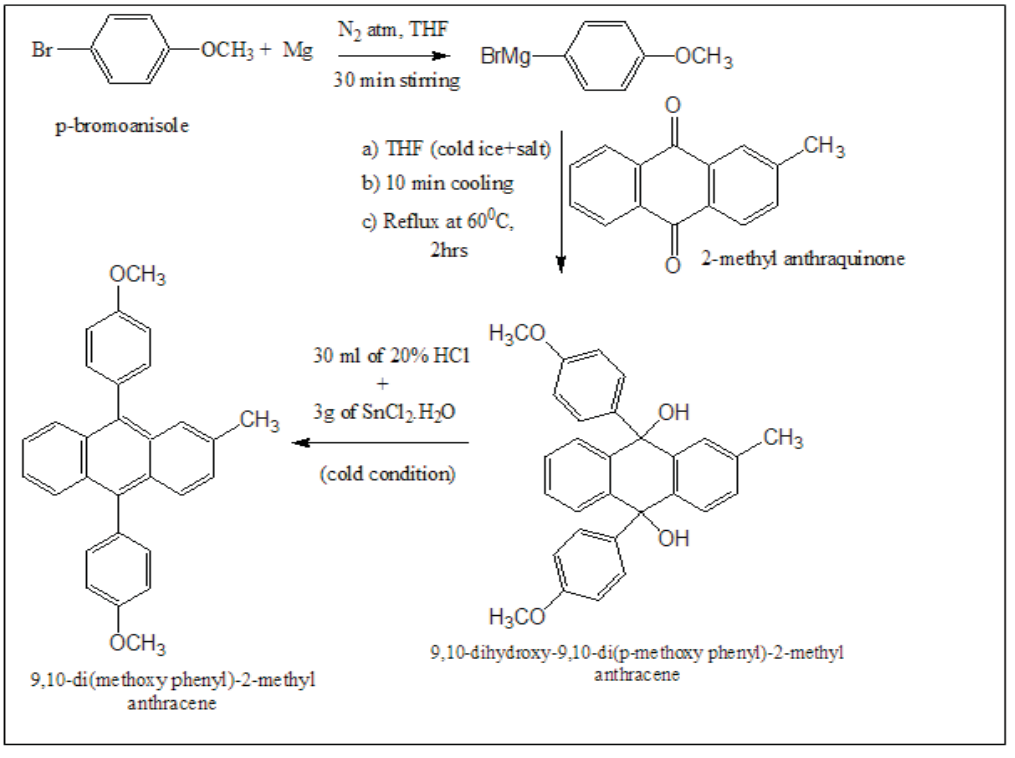
General procedure of synthesis of 9, 10-di (p-methoxy phenyl)-2-methyl anthracene (MDPA):

A solution of 1mmol of p-bromo anisole in THF is added to nitrogen plunged solution of Mg powder (1mmol) in THF at 0-5^{0}C temperature (salt + ice) to synthesize Grignard reagent. The mixture was stirred for 30 minutes.

The suspension of 2-methyl anthraquinone (0.1mmol) in 20ml THF was added drop by drop in a well cooled condition of ice and salt. After completion of addition, cooling was continued for 10 more minutes.

Then reaction mixture was refluxed at 60^{0}C for 2 hours. Then reaction mixture was cooled and mixture of 30ml of 20% HCl and 3g of $SnCl_2.H_2O$ was added drop wise. After the addition, reaction mixture was heated to 60^{0}C with additional 1 hour with stirring.

The precipitate obtained was filtered, washed with water, ethanol and diethyl ether. Product was dried in vacuum.



Reaction scheme : General procedure of synthesis of 9, 10-di (p-methoxy phenyl)-2-methyl anthracene

Molecular formula	:	C29H24O2
Molecular weight	:	404
Weight of crystalline product	:	1.124g
Melting point	:	1980C
Percentage yield	:	61.76%

UV-VISIBLE ABSORPTION:

The absorption of UV- Visible radiations in the CHCl3 (10ppm) solution may occurred due to the transitions of the electrons from lower molecular orbital to higher. From the nature and position of the transitions in the spectra, they were assigned for intra-ligand transitions. The possible intra-ligand transitions are $\sigma\rightarrow\sigma^*$, $\pi\rightarrow\pi^*$, $n\rightarrow\sigma^*$ or $n\rightarrow\pi^*$.The structures of anthracene were characterized through

UV-vis and the spectral data, are listed in Table below;

Table : UV-Visible absorption data for monomers

Compounds	Absorbance	Wavelength (nm)	Assignment
MDPA	0.499	378	π? π*

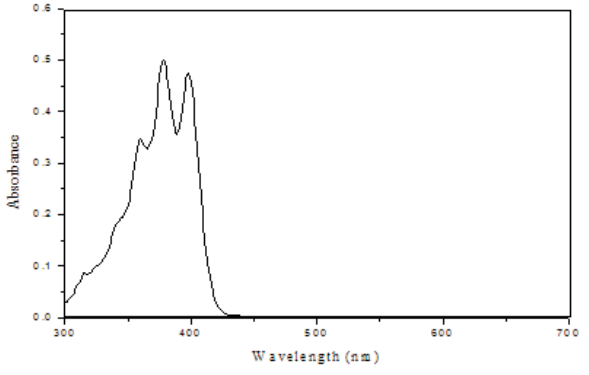


Fig.: UV-Visible Spectra of 9, 10- di (p-methoxy phenyl)-2-methyl anthracene (MDPA)

NUCLEAR MAGNETIC RESONANCE SPECTROSCOPY (NMR):

The ^{1}H NMR spectra were recorded on a Bruker AMX-300 spectrometer in $CDCl_3$/DMSO and chemical shifts were reported in δ ppm. The tetra methyl silane (TMS) was used as an internal standard. The signals were obtained in the range 1.00-8.50 δ ppm. The spectral data of the compounds are summarized in the following tables according to their signals and respective δ ppm values. The signals assigned in the tables were compared with the spectral data for the similar compounds reported in literature.

Table : NMR Spectral Data of 9, 10-di (p-methoxy phenyl) anthracene (MDPA):

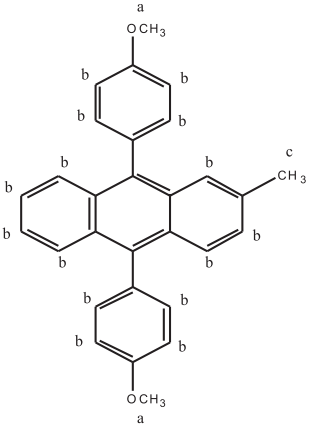
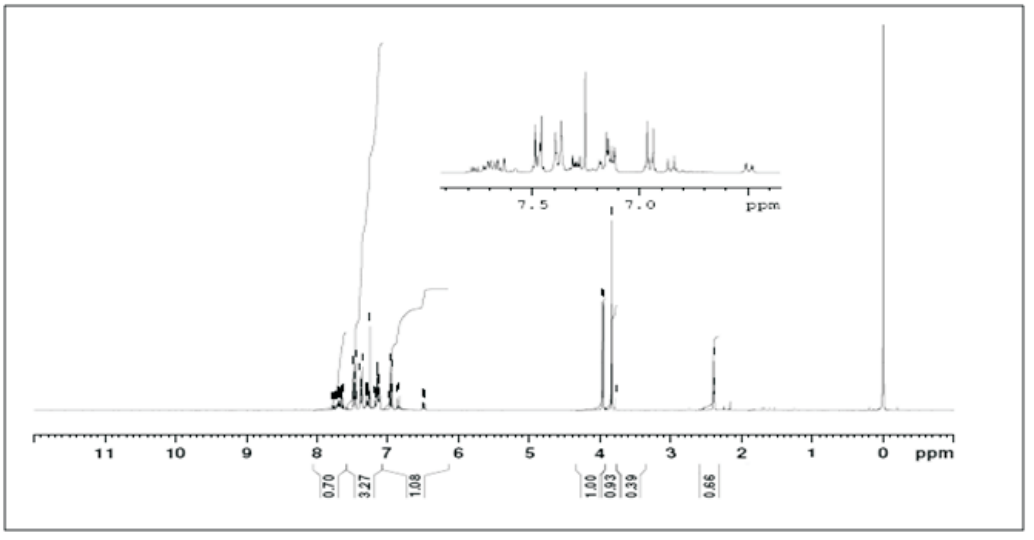
Signals	Nature of signal	δ ppm	Assignment	Structure
a	Singlet	3.957	6H, - OCH_3	
b	Multiplet	6.475-7.784	15H, Ar-H	
c	Singlet	2.398	3H, -CH_3	

Fig.: NMR Spectra of 9, 10- di (p-methoxy phenyl)-2-methyl anthracene (MDPA)



THERMAL STUDY:

The thermogram (TG) of the polymers has been recorded in flowing nitrogen atmosphere at the heating rate of 10°C /min on approximately 10 mg samples.

The TG of diphenyl anthracene derivatives was thermally more stable to varying degree. The thermogram shows the gradual loss in weight due to decomposition by fragmentation with increasing temperature. The decomposition process was started in the temperature range 30-270°C and completed around 1000°C this indicated that the compounds were thermally stable.

The thermogram of MDPA showed three decomposition steps. The first step was observed in the range 30-300 °C, which was accompanied by a weight loss of 27.018%. This may be attributed to the loss of one phenyl ring and one molecule of $–OCH_3$. The second decomposition step was observed in the range 300-410°C, which was accompanied by a weight loss of 21.82%. This may be attributed to the loss of one phenyl ring and one molecule of $–OCH_3$. The third step of decomposition process occurred in the range 410-1000°C which was accompanied by weight loss of 44.532%. This may be attributed to the loss of one molecules of $–C_{15}H_{10}$.

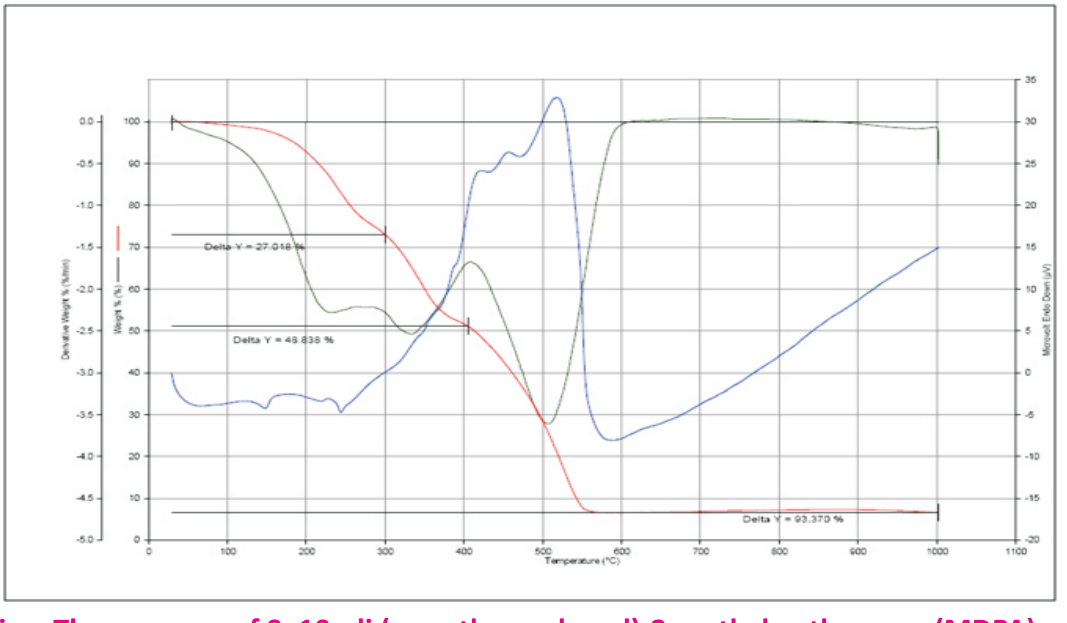


Fig. : Thermogram of 9, 10- di (p-methoxy phenyl)-2-methyl anthracene (MDPA)

CONCLUSIONS:

1)The result of the work carried out showed that derivatives of blue light emitting diphenyl anthracene can be used as monomer for preparation of light emitting polymers.

2)Monomer is easily soluble in organic solvents like DMSO, CDCl3, and THF as well as soluble in common organic solvents.

3)The TG of polymers showed that they were thermally stable to varying degree. The thermogram showed the gradual loss in weight due to decomposition by fragmentation with increasing temperature.

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