IR spectroscopic and photoluminescence studies of plasma polymerized organic thin films based on tea tree oil

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Abstract

Plasma-assisted synthesis of transparent, environment friendly, lightweight, flexible and stable organic thin films from naturally occurring precursors were emerging as potential candidates for organic semiconductor industry. In the present study, tea tree (*Malaleuca alternifolia*) oil based polymer thin films were deposited on glass and silicon substrate by using radio frequency plasma polymerization technique. The polymer thin films were characterized with atomic force microscopy (AFM) and Fourier transform infrared (FTIR) spectroscopy techniques. AFM images indicate the formation of homogenous film on the substrate surface. FTIR spectra gives bands related to methyl and methylene groups which confirms the formation of chain branching in the polymer films. Relatively intense infrared (IR) bands obtained from films deposited on glass substrate reveals that glass substrate is more favourable for the growth of polymers than silicon substrate. Optical band gap of the polymerized thin film on glass substrate was estimated using Tauc plot which gives a value of 3.19 eV, indicating the semiconducting nature of the material. Photoluminescence (PL) emission in the yellow region were observed from both the samples and its CIE colour coordinates also matches with yellow emission. Broad visible emission observed in the wavelength range 465–695 nm indicates the presence of multichromophores in the polymer film. Samples also gives IR emission in the wavelength range of 850–1090 nm, upon excitation with a wavelength of 785 nm contributed to polaronic transitions.

Keywords Rf plasma polymerization · Tea tree oil · Thin film · Photoluminescence · Infrared emission

1 Introduction

Polymer based thin films are important materials for the fabrication of next generation flexible electronic devices [1], sensors [2], organic light emitting devices [3], organic field effect transistors [4] and organic photovoltaic devices [5] as insulating, conducting or semiconducting layers in electronic devices [1, 6–7]. Modification of polymer structure with functional groups and organic moieties changes its physical and chemical properties which consequently provides better optical and electronic responses [8]. Recently, researchers attention is focussed on the development of low cost electronic grade eco-friendly, natural

oil based polymer films for flexible electronic devices [9]. But the development of natural oil based polymers for flexible electronic components is a challenging task because natural oil contains several organic functional moieties. Nevertheless, organic polymer thin films can be deposited on different substrates by using methods such as spin coating [10], dip coating [11], evaporation [12] and solvent casting [13]. Radio frequency (rf) plasma polymerization is a versatile technique used for the fabrication of smooth and highly cross linked natural oil based polymer films on substrates like glass, silicon, quartz etc.[14].

Bazaka et al. [15] used rf plasma polymerization for the deposition of polyterpenol thin films based on *Melaleuca*

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alternifolia oil and studied its optoelectronic and chemical properties. Optical properties of thin films derived from cineole fabricated using rf plasma polymerization method are reported by Easton et al. [16]. Transparent, thermally-stable and biocompatible organic cis- β -ocimene based thin films with an optical band gap of 2.85 eV synthesized by rf plasma polymerization is suitable for inter layer dielectric applications in flexible electronic devices [17]. The rf plasma polymerization based synthesis of smooth and defect free terpinen-4-ol thin films with band gap of 2.67 eV is reported previously [18]. Polyterpenol thin films can be used as an insulating layer in organic field effect transistors [19]. Modifications of chains of polyphenylene vinylene thin films give bright yellow fluorescence at 551 nm [8].

Tea tree oil (*Melaleuca alternifolia* oil) is used as flavouring fragrances and in antimicrobial systems [20, 21]. Investigations of photoluminescence properties of polymers derived from tea tree oil are important since these materials can be used for biofriendly organic LED applications. In the present communication, we are reporting the deposition of polymer thin films on glass and silicon substrates by rf plasma polymerization technique using tea tree oil as monomer and examined its structural and photophysical properties.

2 Materials and methods

2.1 Preparation of tea tree oil thin films on glass and silicon substrate

Thin films used for the present investigation were deposited by rf plasma polymerization with double distilled tea tree (*Malaleuca alternifolia*) oil precursor using a home-made experimental set up [22, 23]. Rf plasma polymerization experimental set up consists of a deposition cell made up of borosilicate glass tube of length 50 cm and diameter of 5 cm. This tube was connected to an evacuation system (rotary pump), rf source (20 W) and monomer injection chamber. Capacitively coupled two aluminium foils wrapped around the glass tube separated at a distance of 5 cm were connected to the rf voltage source which act as electrodes. For the deposition of polymer films, microscopic glass slides (Labtech) and silicon wafer (p-type with {111} orientation) were used as substrates. Glass substrates were chemically cleaned using dil:HNO₃ and distilled water followed by ultrasonication. Later these substrates were again cleaned with organic solvents such as trichloro ethylene, ethanol, acetone and isopropyl alcohol sequentially. Silicon substrates were flushed with air and cleaned using distilled water, trichloro ethylene, ethanol, acetone and isopropyl alcohol independently. Substrates for the

film deposition were placed inside the glass tube exactly under the space separated by the aluminium electrodes. The chamber was evacuated to a pressure of 0.01 mbar before injection of the monomer precursor. A glow discharge of plasma appears between the electrodes when an rf frequency of 7–13 MHz was employed with current in the range of 60–80 mA. After monomer injection, the current was maintained to 64 mA and pressure inside the chamber was 0.028 mbar. The monomer flow rate was controlled using the needle valve of the deposition chamber. Thin films were deposited on glass (T10 G) and silicon substrates (T10 Si) for a time period of 10 min.

2.2 Characterization techniques

FTIR spectra of the polymer samples were recorded using PerkinElmer FTIR Spectrometer Spectrum Two in ATR mode in the range of 400–4000 cm⁻¹. AFM of the samples were obtained using Multi Mode SPM with Nanoscope IIIa controller acquired from Digital/Veeco Instruments Inc, in tapping mode. Thickness of the film was examined using J. A. Woolam Co. Inc EC-400 ellipsometer and analysed using complete ease software and for fitting the ellipsometry data, wavelength dependent measurements were carried out. The thicknesses of the films were also measured using Dektak 6M stylus profiler. The UV–visible absorption spectra of the samples were recorded with JASCO V 570 UV–Vis spectrophotometer. Photoluminescence spectra of the films were obtained using Via Reflex Raman Spectrometer (Renishaw, UK, Model No. M-9836-3991-01-A), by exciting the samples with radiations of wavelength 405 and 785 nm and analysed with WiRE 3.4 software.

3 Results and discussion

3.1 FTIR analysis

FTIR spectra of thin films synthesized with rf plasma polymerization of tea tree oil on glass (T10 G) and silicon (T10 Si) substrates are given in Fig. 1a, b. The constituents of tea tree oil are terpinolene, 1,8 cineole, α -terpinene, γ -terpinene, *p*-cymene, terpinen-4-ol, α -terpineol, limonene, sabinene, aromadendrene, δ -cadinene, globulol, viridiflorol and α -pinene [21]. The detailed assignments of bands are given in Table 1. Spectral pattern of both the samples are different especially in terms of its intensity. The most intense IR band is observed at 880 cm⁻¹ in T10 G contributed to C–H deformation. However, band related to C–H deformation is relatively weak in T10 Si which appeared at 817 cm⁻¹ [16]. The bending of C–H is observed at 1017 cm⁻¹ as a shoulder peak in T10 G and 1042 cm⁻¹ in T10 Si. Stretching mode of C–C is seen at

Fig. 1 FTIR spectra of rf plasma polymerized tea tree oil thin films on **a** glass (T10 G) and **b** silicon (T10 Si) substrate

Table 1 FTIR spectral data of rf plasma polymerized tea tree oil thin films on glass (T10 G) and silicon (T10 Si) substrate

Wavenumber (cm ⁻¹)		Band assignments
T10 G	T10 Si	
438 m	431 m	C–C–C deformation
	609 m	C–H deformation
756 m		C–H deformation
880 vs	817 w	C–H deformation
1017 vw	1042 s	δ (C–H)
	1221 m	ν (C–C)
1378 m	1377 s	δ_s (C–H)
1450 w	1446 w	δ (C–H)
1642 w	1611 w	ν (C=C)
1708 s	1706 vs	ν (C=O)
2964 m	2930 s	ν_a (C–H)
3373 m	3373 s	ν (O–H)

w: weak, m: medium, s: strong, vs: very strong, vw: very weak, vvw: very very weak, sh: shoulder

1221 cm⁻¹ as a moderately intense band, which is absent in T10 G [16]. The symmetric bending mode of C–H is observed around 1377 cm⁻¹ in both the samples indicates the presence of sp³ hybridized methyl groups in the polymer films [15]. The C–H stretching vibrations observed at 1450 cm⁻¹ (T10 G) and 1446 cm⁻¹ (T10 Si) are related to sp² hybridized methylene groups in the plasma polymerized thin film [15]. The band in this region also gives the signatures of the retention of aromatic rings in the polymer backbone [16, 24]. Vibrational bands corresponding to C=C are seen around 1642 cm⁻¹ in T10 G, while the same is at 1611 cm⁻¹ in T10 Si. IR band contributed to ketonic functional group (C=O) is observed in T10 Si as an intense band at 1706 cm⁻¹ and at 1708 cm⁻¹ in T10 G [16]. Presence of C=C and C=O in the FTIR spectra indicate the existence of

multichromophores in the polymer film deposited on glass as well on silicon substrates. Asymmetric stretching vibrations of C–H band at 2964 cm⁻¹ in T10 G is also observed to be shifted to lower wavelength region at 2930 cm⁻¹ in T10 Si [16]. The band observed at this region also indicates the presence of methylene group in the polymer films [15]. The appearance of C–H bands at 1377 cm⁻¹ (T10 G and T10 Si), 2964 cm⁻¹ (T10 G) and 2930 cm⁻¹ (T10 Si) indicate the presence of hydrogen bonded benzene rings as well as chain branching in polymerised samples under study [15]. The presence of saturated and unsaturated carbon bonds and aromatic rings in the FTIR spectra confirms that rf plasma polymerized tea tree oil films deposited on glass and silicon substrate are hydrocarbon rich polymers.

It is interesting to note that a shift in the deformation and stretching bands of C–H groups to lower wavenumber region is observed in the sample grown on silicon substrate (T10 Si). This indicates that there exist a slight decrease in bond lengths of these functional groups in T10 Si with respect to T10 G. Formation of additional peaks (1221 cm⁻¹) and shift in wave numbers of certain functional groups (817, 1042, 1611 and 2930 cm⁻¹) in T10 Si gives the strong indication of the difference in structural pattern with respect to sample grown on glass substrate (T10 G).

Chemical reactions occurring under plasma conditions are complex and the polymerization process is initiated due to the formation of free radicals in the glow discharge plasma [25]. The input power applied into the reaction chamber is basically responsible for the creation and maintenance of plasma. Fragmentation of the monomer molecules are initiated by the assistance of plasma which leads to the formation of free radicals. The highly reactive free radicals thus formed are collide with remaining monomers results in dissociation of bonds in them or can create excited species and will trigger the chemical

reactions. The initiation, propagation and termination of free radicals thus generated will leads to the deposition of polymer molecules on substrates [25, 26]. The presence of saturated and unsaturated carbons, and aromatic functional group in the FTIR spectra (Fig. 1) confirms the formation of polymer thin films on glass and silicon substrates.

3.2 Thickness and surface morphology analysis

The thickness of plasma polymerized thin films made of tea tree oil, on glass (T10 G) and silicon (T10 Si) substrates are measured by using spectroscopic ellipsometric technique. Information obtained from ellipsometric study is fitted with an optical model for the polymer film by considering the surface roughness layer, polymer film and substrate. The optical model chosen for fitting the experimental data for T10 G was “glass with transparent film mode” at an angle of incidence 53° . Similarly, optical model chosen for fitting T10 Si was “Si with transparent film mode” at an angle of incidence 85° . Thickness of the polymer films obtained from the ellipsometric study for T10 G and T10 Si are 2006 and 1553 nm respectively. The thickness of T10 G and T10 Si are also measured using stylus profiler are comparable with that of obtained from the ellipsometric study.

AFM images of thin films made up of rf plasma polymerized tea tree oil thin films on glass and silicon substrates are shown in Fig. 2. The root mean square roughness (Rq) of T10 G is ~ 5.95 nm which is measured from an area of $1 \mu\text{m}^2$. But, the film on the silicon substrate (T10 Si) possess a roughness (Rq) value of 0.253 nm from an area of $5 \mu\text{m}^2$. Polymer films deposited on glass substrate exhibit higher thickness and

roughness with respect to the silicon substrate. These types of differences are expected due to the variations in surface energy as well as lattice mismatch of the substrate [27, 28]. Glass is an amorphous substrate and silicon is a crystalline substrate, both of them are having different surface energies and there exist lattice mismatch with each other. Since polymers are amorphous in nature, glass substrates may favour the growth of amorphous materials like polymer which in turn help the deposition of thicker films over its surface [29]. AFM images confirmed that the rf plasma polymerized thin films on glass and silicon substrates are homogeneous.

3.3 UV–Vis absorption studies

Optical absorption spectrum of rf plasma polymerized tea tree oil thin film on glass substrate shows absorption in the UV region at 332 nm (Fig. 3a) due to the the presence of carbonyl groups which gives π – π^* transitions of aromatic ring [30]. This film also shows broad weak absorption bands at 558 nm (Fig. 3a) contributed to interchain π – π stacking interactions of rings similar to that reported in polythiophene films prepared by spin coating method [31].

The optical band gap of rf plasma polymerized tea tree oil thin films deposited on glass substrate (T10 G) is determined from the UV–Vis absorption data using Tauc plot relation (1) [15].

$$\alpha h\nu = B(h\nu - E_g)^n \quad (1)$$

where ‘ α ’ is the absorption coefficient, ‘ $h\nu$ ’ is the photon energy, ‘ B ’ is a constant which dependent on the length

Fig. 2 AFM images of rf plasma polymerized tea tree oil thin films on **a** glass (T10 G) and **b** silicon (T10 Si) substrate

Fig. 3 **a** UV-Vis absorption spectrum of plasma polymerized tea tree oil thin film on glass substrate (T10 G) and **b** Tauc plot of T10 G

of localized state tails and ' E_g ' is the optical band gap of the material [15]. For polymer thin films the value of ' n ' is chosen as 2 by considering the present sample as an indirect band gap material [15]. Optical band gap of T10 G is determined from the Tauc plot, which gives a value of 3.19 eV, indicating the semiconducting nature of the film (Fig. 3b). Aromatic benzene rings in the FTIR spectrum (Fig. 1a) and weak optical absorption band around 558 nm (Fig. 3a) observed from the sample indicate the presence of isolated conjugation units in the structure of the polymer. This in turn suggests the presence of π electron cloud arising from the aromatic rings of the polymer skeleton which might have contributed to the semiconducting properties of the sample.

3.4 Photoluminescence studies

In order to understand the light emission properties of plasma polymerized tea tree oil thin films on glass and silicon substrates, photoluminescence (PL) measurements are carried out using excitation wavelength (λ_{ext}) such as 325, 405 and 785 nm. PL spectra of rf plasma polymerized tea tree oil thin film deposited on glass (T10 G) and silicon (T10 Si) substrate shows a broad emission band which extends from 465 to 695 nm with a laser excitation wavelength of 325 nm (Fig. 4a). Emission spectra of T10 G and T10 Si show broad visible emission in the yellow region on exciting with the above laser beam. T10 G shows an intense broad emission band around 584 nm and T10 Si shows broad emission at 600 nm. The emission band around 584 nm from T10 G, arises due to the transitions

Fig. 4 **a** PL emission spectra of rf plasma polymerized tea tree oil thin films on glass (T10 G) and silicon (T10 Si) substrates under an excitation wavelength of 325 nm and **b** CIE 1931 diagram of T10 G and T10 Si

from HOMO level to upper polaron states [32]. Emission peak at 600 nm in T10 Si indicates the presence of excimers. This type of excimeric emission may arise in the polymer film due to interchain interactions between longer polymer chains [34]. With respect to T10 G, intensity of PL emission band decreases and peak is redshifted in T10 Si. Experimentally obtained PL data of T10 G and T10 Si are converted into Commission International De l'Eclairage (CIE) 1931 chromaticity diagram using CIE colour coordinate calculator (Fig. 4b). The colour coordinates calculated for T10 G ($x=0.450$, $y=0.474$) and for T10 Si ($x=0.465$, $y=0.467$) are associated with yellow emission.

The PL emission spectra of T10 G and T10 Si with a laser excitation wavelength of 405 nm is shown in Fig. 5a. T10 G shows a broad emission band which extends from 465 to 695 nm with peak centred around 556 nm when excited with a laser beam of wavelength 405 nm. PL spectrum of T10 Si shows a broad spectrum and its peak position is redshifted (581 nm) with respect to the film grown on glass substrate (T10 G). Broad and intense PL emission band at 556 nm in T10 G (Fig. 5a) under an excitation of 405 nm is probably due to the generation of singlet excitons [32]. The emission at 581 nm from T10 Si contributed to the transitions from HOMO level to upper polaron states [32]. The semiconducting properties of these thin films can be attributed to the transition of electrons from highest occupied molecular orbital (HOMO) to the antibonding polaron states [33].

Experimentally collected PL data of T10 G and T10 Si at 405 nm laser excitation are also converted into CIE 1931 chromaticity diagram using CIE colour coordinate calculator (Fig. 5b). The colour coordinates calculated for T10 G ($x=0.392$, $y=0.48$) and for T10 Si ($x=0.422$, $y=0.469$)

are related to yellow emission. Both the PL spectra and CIE colour coordinates obtained for laser excitation wavelength 325 nm (Fig. 4) and 405 nm (Fig. 5) show yellow emission. This indicates that rf plasma polymerized tea tree oil thin films deposited on glass and silicon substrates have good emission at yellow region of the electromagnetic spectrum.

In order to understand the infrared (IR) light emitting capability and nature of species taken part in the emission properties, PL emission spectra of the T10 G and T10 Si is carried out using a laser excitation of 785 nm (Fig. 6). Interestingly, PL emission spectra of rf plasma polymerized films of tea tree oil (T10 G and T10 Si) show intense peaks in the IR region at 880 nm when excited with a wavelength of 785 nm. But T10 Si shows an additional peak at 819 nm followed by a broad band which extends from 850 to 1090 nm with peak at 1005 nm. Moderate intense IR emission bands at 880 and 877 nm with an excitation of 785 nm from T10 G and T10 Si respectively are associated with polaronic transitions from lower energy level to upper level (Fig. 6). The appearance of broad and intense band at 1058 nm from T10 Si may be due intrachain emission from the polymer films [32].

PL emission spectra of T10 G and T10 Si obtained at laser excitation wavelengths of 325, 405 and 785 nm are broad in nature. The broad PL bands in the spectra indicates the presence of various defect states in the polymeric thin films, probably associated with the weak polymerization of monomers [32]. Rf plasma polymerized tea tree oil thin films formed on glass and silicon substrate are having flexible polymer chains, as evidenced from the broad PL emission [35]. The observed decrease in intensity of PL band from the sample on Si

Fig. 5 **a** PL emission spectra of rf plasma polymerized tea tree oil thin films on glass (T10 G) and silicon (T10 Si) substrates under an excitation wavelength of 405 nm and **b** CIE 1931 diagram of T10 G and T10 Si

Fig. 6 PL emission spectra of rf plasma polymerized tea tree oil thin films on glass (T10 G) and silicon (T10 Si) substrates under an excitation wavelength of 785 nm

substrate with respect to glass substrate may be due to the large interchain contributions [36]. With respect to glass substrate, rf plasma polymerized tea tree oil film deposited over silicon substrate (T10 Si) show a slight redshift in its PL emission. This may be due to the decreased surface roughness of the polymer film on the silicon substrate. Our AFM analysis also supports a decrease in surface roughness in T10 Si (0.253 nm) with respect to T10 G (5.95 nm). This in turn suggest that polymer films deposited on the silicon substrate are much smoother than the film deposited on glass substrate.

The origin of PL emissions in the present sample is associated with the conduction of electrons in the lowest unoccupied molecular orbitals (LUMO) and holes from the highest occupied molecular orbitals (HOMO). Intra and interchain excitons are the major photoexcited species of organic polymers [32]. Apart from that, organic polymers are complex systems in which the dominant electronic excitations are solitons, polarons and bipolarons, which might have coupled with structural distortions [37]. In the present case, excitons are generated on the polymer films upon interaction with photons from laser beam. The polaronic excitons (intrachain excitons) may recombine with local deformations in the polymer backbone leading to the radiative recombination in polymeric structures. The transitions between polarons and HOMO levels, and also from the defects present in the film may contribute to PL. PL intensity has strong dependence on the polymer chain length, nature of polymer, defects, impurities and oxygen containing functional groups in polymer films [32]. Multichromophores

in polymer thin films also act as active subunits for the electronic energy transfer, which may also contribute to strong PL emission [38].

FTIR bands at 1642 , 1708 cm^{-1} (T10 G) and 1611 , 1706 cm^{-1} (T10 Si) are corresponds to C=C and C=O, which indicate the existence of multichromophores in plasma polymerized thin film with tea tree oil precursor (Table 1). Electronic energy can migrate through interchain interactions and cause excitations towards chromophores and give rise to strong emission in the visible region. The occurrence of visible PL emission from the present sample in the range 500–600 nm with an excitation of 325 and 405 nm laser excitation indicates the presence of π conjugation in the polymeric structure [3]. From these results, it can be concluded that rf plasma polymerized tea tree oil thin films (T10 G and T10 Si) contains different chromophore units having isolated electron rich conjugated entities, which facilitates the localization of π -electrons. Moreover, this type of electron localization in turn ensures better stability to the polymer films [29].

4 Conclusion

Polymer thin films deposited by rf plasma polymerization process of tea tree oil on glass and silicon substrates are hydrocarbon rich and homogenous. Polymeric chain branching and interchain π - π stacking interactions are seen in the plasma polymerized tea tree oil thin films. Tea tree oil derived polymer thin film on glass substrate possess semiconducting properties, with an optical band gap of 3.19 eV. Broad PL emission in the range of 465–695 nm shows the presence of chromophore units in the polymer film. Polaronic transitions and interchain emissions are also occurring in the films as evidenced by the presence of IR emission on excitation of the sample with 785 nm. The structural and photophysical properties of the rf plasma polymerized tea tree oil films indicates that glass substrate promote better growth of polymer than the silicon substrate. These results predict that, biofriendly rf plasma polymerized tea tree oil thin films can be used as semiconducting layers in electronic devices and in organic light emitting systems.

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Compliance with ethical standards

Conflict of interest No potential conflict of interest was reported by the authors.

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