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# **Solvent Driven Performance in Thin Floating-films of PBTTT for Organic Field Effect Transistor: Role of Macroscopic Orientation**

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## Abstract

Considering the advantages of floating films transfer method (FTM), we have investigated the optical and electronic characteristics of PBTTT-C14 thin-films prepared by the static and the dynamic casting on liquid substrate. It has been demonstrated that judicious selection of solvents during FTM switches the casting mode from the static casting (S-FTM) using high boiling point solvent to the dynamic casting (D-FTM) from low boiling point solvent. Although both of the methods provide the edge-on oriented structure of PBTTT-C14 by XRD, the structural and the optical analyses reveal relatively extended  $\pi$ -conjugation length in parallel D-FTM film as compared to that of S-FTM. A high field-effect mobility ( $\mu$ ) of  $0.11 \text{ cm}^2\text{V}^{-1}\text{s}^{-1}$  was exhibited by OFETs fabricated by parallel D-FTM film even without any high temperature post-annealing up to the liquid crystalline phase transition. This observed value of  $\mu$  for parallel D-FTM is 4.7 and 12.8 times higher than the isotropic S-FTM and the perpendicular D-FTM films, respectively.

**Keywords:** casting procedure, static, dynamic, floating film, liquid substrate, anisotropy

## 1. Introduction

Advent of solution processable conjugated polymers (CPs) has witnessed the tremendous growth in their utilization as active semiconducting materials owing to their low cost manufacturing [1–6] and realization of flexible electronics [7-8]. It is well known that morphology of CPs in thin films plays a prominent role in governing the charge transport. Enhancing the device performance of solution processable organic thin film transistors to the level of amorphous silicon is undoubtedly necessary for their practical realization [2]. Thin film morphology of CPs plays a crucial role for achieving the high carrier mobility ( $\mu$ ) which in-turn is highly dependent on the thin film processing conditions [9]. Improving the  $\mu$  using high molecular weight CPs, high boiling point solvents [9,10], dielectric surface treatment [11,12], deposition method [13] and orientation techniques [14] have been widely discussed in the recent past [15]. Although spin coating is most preferred method for the fabrication of thin films of CPs but still existing bottle-necks like high material wastage and surface roughness limits their usage at industrial scale [16]. In the meantime, several new thin film fabrication techniques such as solution flow [17], flow coating [18], solution shearing [19], capillary action [17], slide coating [20] etc. have also been proposed to circumvent problems of the spin coating. Intriguing challenges like simple fabrication process, thickness uniformity and capability for multilayer thin film fabrication are yet to be solved for large scale production of organic electronic devices. One of the plausible solution might be the possibility of large area thin film fabrication on liquid substrates followed by their transfer on the desired solid substrates. Such method are capable of solving both of the existing issues like logical selection of the suitable solvent not only for the preparation of CP solution but also the liquid substrate from which the solid-state thin films are transferred to the desired substrate. At the same time, thickness

and uniformity of the film can be easily controlled by adopting the multi-layer coatings.

In order to provide an amicable solution for problems existing in the spin-coating method, our group has developed a novel thin film fabrication technique, namely floating-film transfer method (FTM). Casting CPs from the low boiling point (BP) solvent like chloroform on an orthogonal liquid substrate, we have demonstrated that there is not only improvement in the device performance for the films prepared by FTM [21,22] but also it has been utilized to achieve molecular orientation for a number of CPs having potential application for the anisotropic optoelectronics [23–25]. Improvement in the field-effect mobility of CPs by spin-coating their thin films from high boiling solvents has also been reported. This was attributed to the slow solvent evaporation leading to thermodynamically favorable molecular arrangement in the thin films, where alkyl chains lie standing with respect to the substrate [9,10,26]. On the other hand, fast solvent evaporation during the formation of thin films leads to kinetically favored structures where alkyl chains lie in the plane of the substrate [10,26]. In the recent past efforts have also been directed towards casting thin films of CPs utilizing high boiling solvent like chlorobenzene (BP 131°C) on liquid substrate in order to enhance the device performance along with the demonstration of multilayer thin film fabrication [27,28]. Enhancement in the device performance was explained by considering the fact that for chlorobenzene solution of CPs, solvent evaporates very slowly giving enough time for the self-organization of macromolecules on the liquid substrate. This process can be being expressed as static mode of FTM (S-FTM).

In contrast, casting CPs with low boiling solvent such as chloroform (BP 61°C) and dichloromethane (BP 40°C), there is a dynamic equilibrium between spreading of the solution on the liquid substrate, formation of the thin film by rapid solvent evaporation and

dragging viscous force offered by the orthogonal liquid substrate. This process can be expressed as dynamic FTM (D-FTM). We have already demonstrated that through the systematic control of casting conditions such as temperature, concentration of solution and viscosity of the liquid substrate, strong uniaxial orientation can be induced in floating-films under D-FTM [24,29]. Recently, other groups have also spotlighted the importance of the casting methodology of conjugated polymers on liquid substrates and their effect on the device performances in various devices like solar cells and phototransistors [23,27,28,30,31]. Owing to the versatile features of floating film, choice of the suitable solvent (high or low BP) during preparation of the solution of CPs is one of decisive steps and plays an important role in the growth mechanism of the thin films. In this study, investigations pertaining to the optical and structural characterization of thin films prepared by both of the S-FTM and D-FTM and their implication on electronic characteristics after the fabrication of the organic field effect transistors (OFETs) have been carried out.

## **2. Experimental Section**

### **2.1 Thin Film Preparation**

A thiophene based CP, poly[2,5-bis(3-tetradecylthiophen-2-yl)thieno[3,2-b]thiophene] (PBTTT-C14) was employed to compare the performance of carrier transport in this study [32]. PBTTT-C14 was purchased from Sigma-Aldrich and used without any further purification. Super dehydrated chlorobenzene and chloroform were purchased from WAKO pure chemicals. PBTTT-C14 solutions were prepared by 1% and 0.5% (wt/wt) in chloroform and chlorobenzene, respectively. These polymer solutions were then heated at 55°C with continuous stirring to prepare the solutions. For S-FTM PBTTT-C14/chlorobenzene solution was dropped on a distilled water filled in a 15 cm diameter petri-dish as liquid substrate, then it was left for several hours to obtain a thin

floating-film as per the literature report [27]. For D-FTM, PBTTT-C14/chloroform solution was dropped on a viscous liquid substrate consisting of ethylene glycol and glycerol mixture as per our earlier publication [24,33]. In this case, floating films were immediately formed on the liquid substrate with spreading the solution. Both of the floating films were transferred on to the desired substrate by stamping.

## **2.2 Thin Film Characterization**

Thickness of floating-film coated on a flat Si substrate was about 25 nm as measured by interference microscopy (Nikon Eclipse LV150). Several pieces of white-glass (2.5 cm x 1.0 cm) were used as the substrates for polarized electronic absorption spectroscopic and optical texture measurements. Polarized spectra were measured by a spectrophotometer, JASCO V570, equipped with a Glan-Thompson prism. Optical textures were taken using Olympus BX50 polarizing optical microscope (POM). Thick films of PBTTT-C14 were prepared by multiple stamping via each FTM (both S-FTM and D-FTM) to get thicker films (about 700-800 nm), which were used for out-of-plane X-ray diffraction (XRD) measured by Rigaku X-ray diffractometer with Cu K $\alpha$  radiation at 20 KV and 20 mA. Si-wafer and white glass surfaces were made hydrophobic using hexamethyldisilazane by a similar manner as per our earlier report [34].

## **2.3 Device Fabrication**

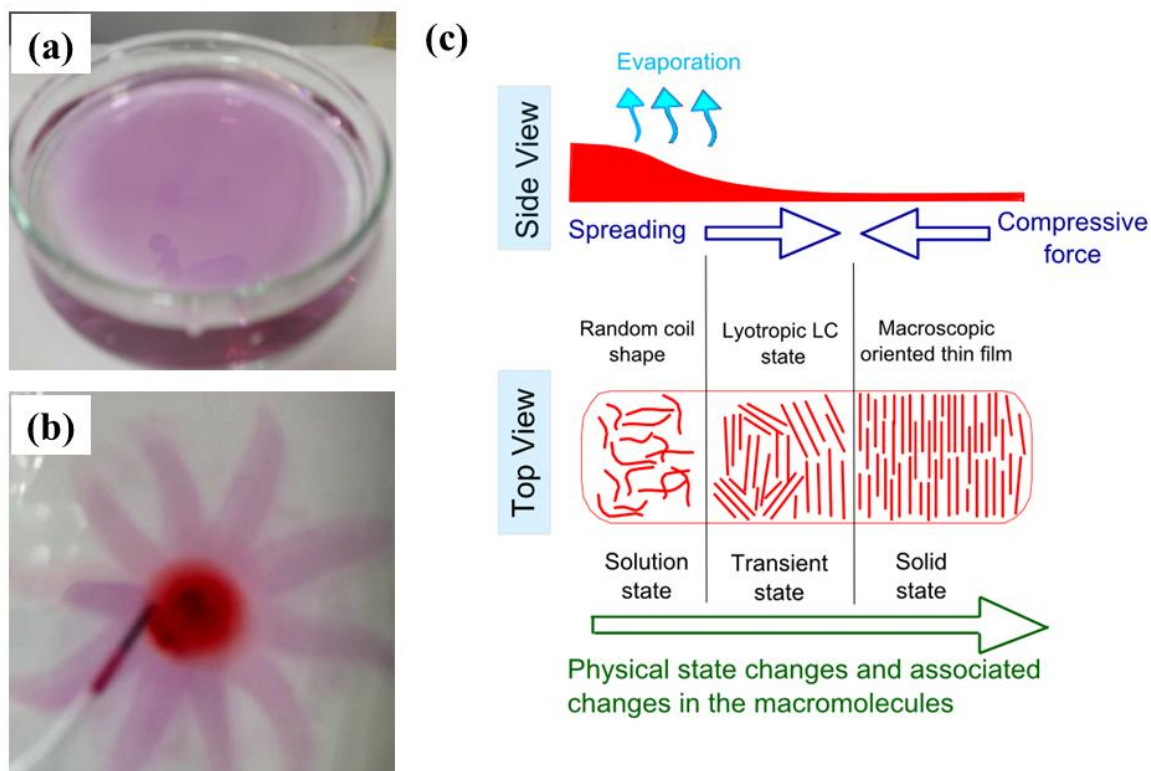
Top contact bottom gate OFETs were fabricated to evaluate carrier transport characteristics for both types of FTM films. Highly doped silicon wafer substrates having 300 nm thick SiO<sub>2</sub> insular were used to spin coat CYTOP<sup>TM</sup> at 3000 rpm on SiO<sub>2</sub> for 120 seconds and annealed at 150°C for 1 hour to increase the surface hydrophobicity. The resultant insulator capacitance was estimated to be about 8 nF/cm<sup>2</sup>. FTM films were then transferred on this hydrophobic surface followed by washing with methanol to remove residual hydrophilic

liquid substrate materials. Gold was utilized for source and drain electrodes to deposit by thermal deposition at a base pressure of  $10^{-6}$  Torr through a nickel shadow mask having channel length ( $L$ ) of 20  $\mu\text{m}$  and channel width ( $W$ ) of 2 mm. To ensure the electrical isolation in OFETs, semiconductor layer was electrically isolated around the device by wiping with cotton bud dipped in chloroform. Electrical parameters were measured with computer-controlled source meter Keithley-2612.

### 3. Results and discussion

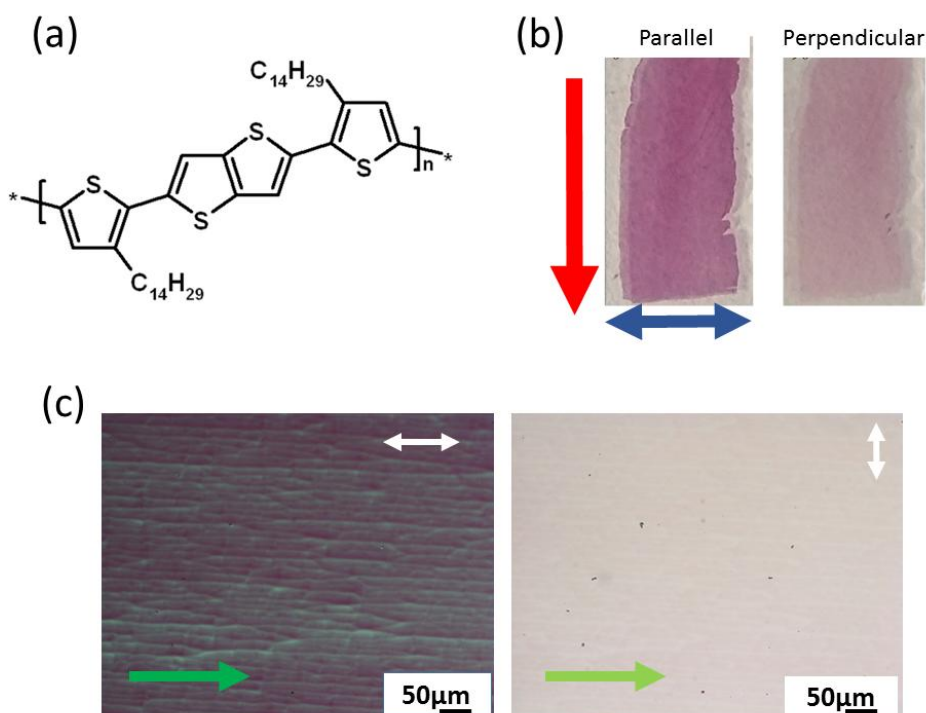
Photographs of the floating-films on liquid substrate are shown in Fig. 1. It can be seen from the Fig.1(a) that for S-FTM with PBTTT-C14/chlorobenzene solution, spreading of the solution takes place throughout the entire surface of the liquid substrate (water) followed by the formation of floating-film after the slow evaporation of chlorobenzene. In contrast, when PBTTT-C14/ $\text{CHCl}_3$  solution was subjected to film casting on liquid substrate consisted of an ethylene glycol/glycerol mixture (3:1) at  $55^\circ\text{C}$ , it spreads in all of the direction in branched form like a star-fish followed by simultaneous solidification leading to the formation of oriented floating-films owing to relatively quick evaporation of chloroform as shown in Fig. 1(b) for D-FTM. In this case, viscosity of the liquid substrate and temperature for casting play important roles for controlling the orientation [29]. It should be noted that the orientation direction in the floating-films can be easily seen by necked eye through a polarizer film. The plausible mechanism for the origin of orientation under D-FTM is schematically shown in the Fig.1(c).





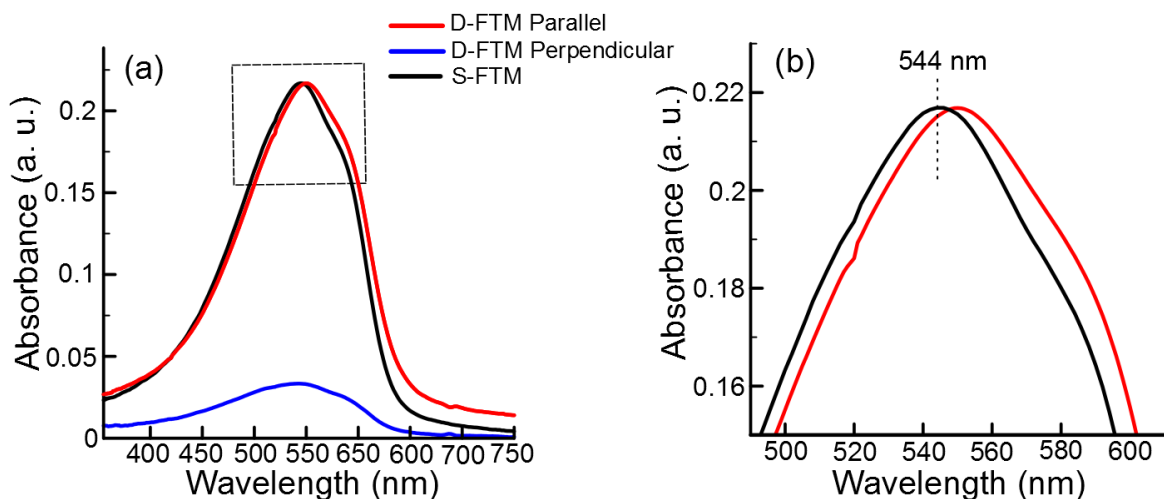
**Fig. 1.** Photograph of the thin films formed at liquid substrate using S-FTM (a) and D-FTM (b), and schematic for possible mechanism for macroscopic orientation in D-FTM (c).

For D-FTM, PBTTT-C14 macromolecules move randomly due to the weak inter-molecular interactions in the solution state. Continuous concentration of the solution on liquid substrate assisted by simultaneous solvent evaporation leads to the short time induction of lyotropic liquid crystalline (LC) phase transition as transient state in the solution before reaching to the solid state [35]. The continuous spreading of the solution on liquid substrate drives the solidifying macromolecules into the same direction as the expansive and the compressive forces assisting towards lyotropic LC phase transition [33,34]. The balance between the evaporation and the spreading speeds in this process specify the length of transient time for the orientation of macromolecules perpendicular to the propagation direction. In contrast, such color change was not observed through the polarized film in the floating-film prepared by S-FTM unlikely to that by D-FTM.



**Fig. 2.** (a) Chemical structure of PBTTT-C14, (b) Photograph of oriented thin films stamped on glass substrate ( $2.5 \text{ cm} \times 1 \text{ cm}$ ) obtained by D-FTM with polarizer rotated parallel and perpendicular to the orientation direction. Floating film was casted from branch of star-fish like floating film, where red arrow represent the film expansion direction and double sided blue arrow represents the orientation direction of main chain and (c) POM images of oriented PBTTT-C14 thin film prepared by D-FTM. Single green arrow represents the direction of oriented PBTTT-C14 films whereas white double arrow represents the polarization of the incident light.

PBTTT-C14 thin films stamped on the glass slide have clearly distinguishable color change when seen through polarized film due to the macroscopic orientation of the polymeric assemblies in D-FTM as shown in the Fig. 2 (b). This high orientation of PBTTT-C14 macromolecules in fabricated thin films is further supported by the polarized optical microscopic images as shown in Fig. 2 (c). A perusal of the Fig. 2 (c) reveals a remarkable color change when the polarization angle of incident light was changed from  $0^\circ$  to  $90^\circ$  along with the contrasting color changes of the texture from dark to almost colorless in the POM images.

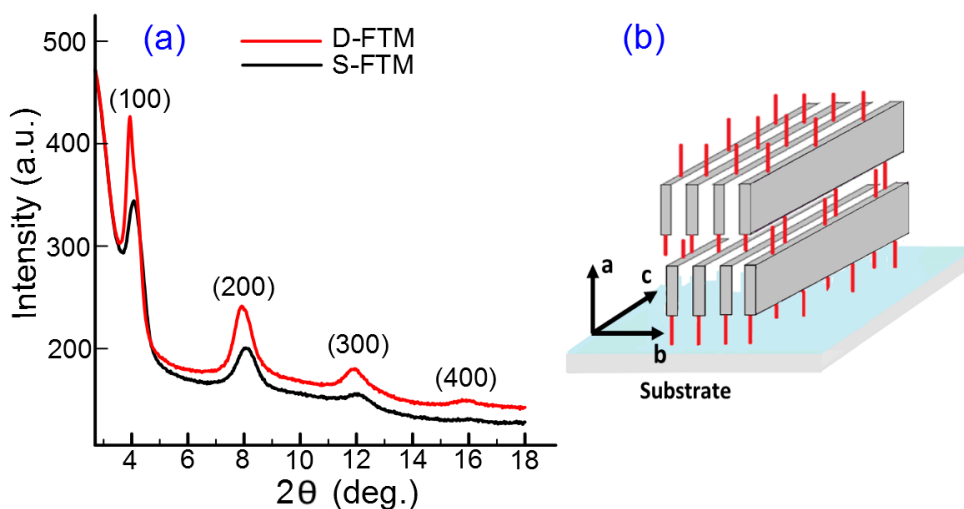


**Fig. 3** (a). Polarized absorption spectra of PBTTT-C14 thin film prepared by S-FTM and D-FTM. (b). Enlarged view of the marked area with square.

In order to visualize this strong optical dichroism of D-FTM films quantitatively, polarized electronic absorption spectroscopic investigations were also carried out which have been shown in the Fig. 3. Strength of molecular orientation was indexed by the quantitative estimation optical dichroic ratio (DR) calculated by the Eq. (1) which was found to be 6.5. It is important to note that such a high DR value for the PBTTT-C14 has been only achieved at elevated temperatures ( $> 100^{\circ}\text{C}$ ) by other research groups through mechanical rubbing and film compression on ionic liquid substrates by utilizing formation of their LC mesophases [30,36].

$$DR = \frac{\text{Maximum Absorption}_{\text{parallel at } (\lambda_{\text{max}}_{\parallel})}}{\text{Absorption}_{\text{perpendicular at } (\lambda_{\text{max}}_{\parallel})}} \quad (1)$$

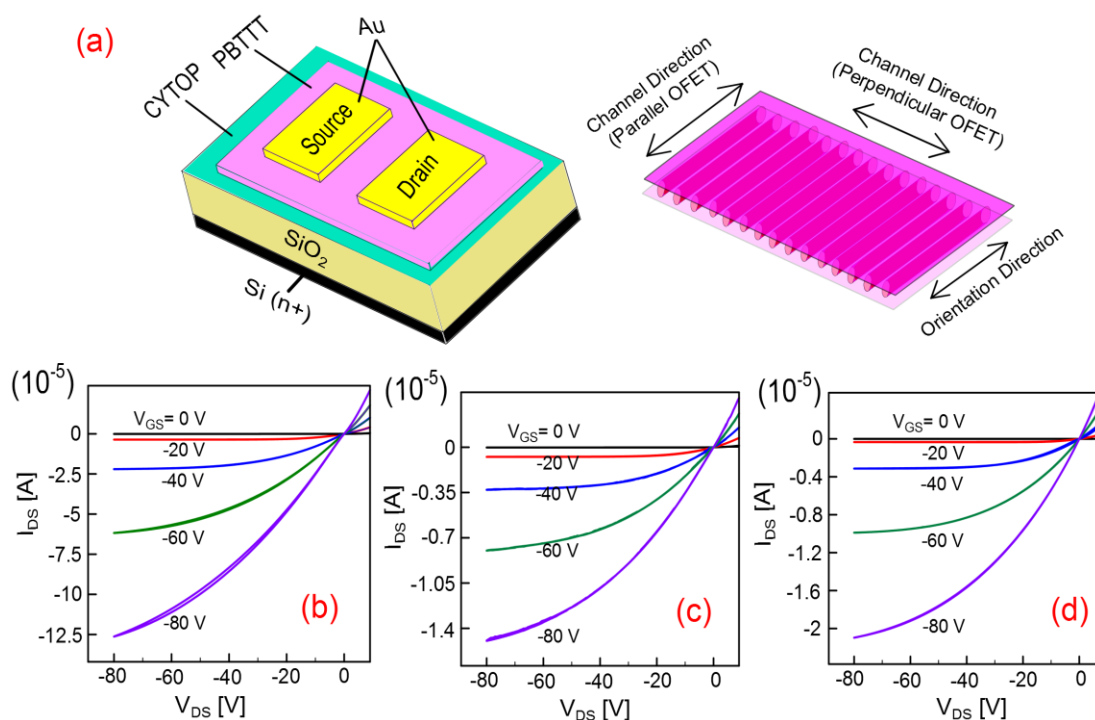
Considering the differences between the electronic absorption spectra of D-FTM (Parallel) and S-FTM films, there was a slightly red-shift in the main absorption peak along with the pronounced vibronic shoulders appearing at 544 nm and 600 nm, respectively [24,37]. The red shift represents the increased effective  $\pi$ -conjugation length in D-FTM film and pronounced shoulder indicates the improvement in the  $\pi$ -orbital delocalization [12].



**Fig. 4.** Out-of-plane XRD pattern of the PBTTC14 films prepared by S-FTM (black) and D-FTM (red) (a) and the schematic representation of edge-on orientation of the polymeric backbone in the films with their unit cell axis (b).

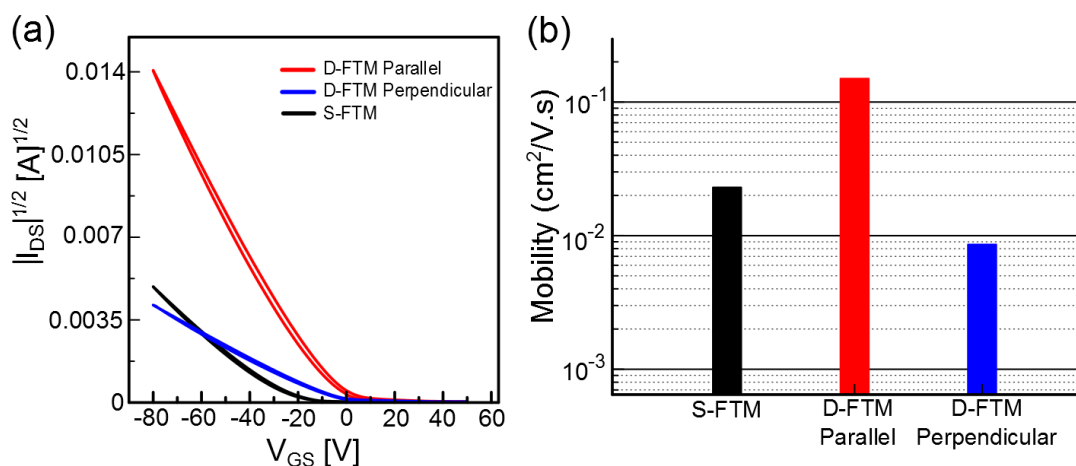
Out of plane X-ray diffraction patterns of the films prepared by both of the S-FTM and D-FTM are shown in the Fig. 4. The preferential orientation of the backbone when a-axis lies (alky-chain direction) out of the plane (Edge-on orientation) is preferred due to high in-plane transport as both the b-axis ( $\pi$ - $\pi$  stacking direction) and c-axis (main-chain direction) lies in the same plane. This edge-on orientation having facile in-plane charge transport is highly beneficial for fabricating in-plane organic electronic devices like OFETs [14,38]. On the other hand opposite face-on orientation is preferred where b-axis lies out of the plane for devices with out of plane vertical charge transport such as solar cells and light emitting diodes. X-ray diffraction patterns of the films prepared by both of the S-FTM and D-FTM are compared in Fig. 4. A series of diffraction peaks corresponding to the lamellar d-spacing (a-axis) is calculated from the average of the peak positions and was found to be 22.2 Å, indicating the edge-on structure perpendicular to the substrate as schematically shown in Fig. 4 (b). The peak intensities are more pronounced up to 4th order in the D-FTM as compared to that of S-FTM film. At the same time, 100 peak in D-FTM is relatively

intense and sharp as compared to S-FTM representing that films prepared by D-FTM are more crystalline with relatively larger grain size [30,39]. Interestingly, there is a peak-shift of the lamella d-spacing for D-FTM films towards the lower angle as compared to those in S-FTM films indicating that the lamella-layer structure is highly stretched along with the edge-on orientation on the substrate. It is worth to mention that such a well-defined and sharp diffraction peaks and lamella d-spacing in the PBTTT-C14 films have been only reported by the thermal annealing up to the thermotropic LC phase transition temperature of about 180°C [14, 18]. This result represents that the developed D-FTM provides the growth of films with well-oriented domains without high temperature thermal treatment. The in-plane orientation in the domain should be discussed with the in-plane GIXD measurements, which is still under investigation.



**Fig. 5.** Device configuration of the fabricated OFET (a) and output OFET characteristics of PBTTT-C14 films prepared by D-FTM parallel (b), D-FTM perpendicular (c) and S-FTM (d).

OFETs were fabricated in the three different device configurations in order to delineate the implication of nature of fabricated thin films on the charge transport properties. Two types of parallel and perpendicular OFETs for D-FTM were fabricated due to the observation of high optical anisotropy while third type of device was fabricated utilizing isotropic films prepared by S-FTM. Results obtained after the measurement of  $I$ - $V$  characteristics for the respective OFETs are shown in the Fig. 5. All of the OFETs under investigation exhibited well known p-type transport characteristics. Relatively large drain currents were observed for the OFETs fabricated with D-FTM films in parallel orientation compared to that prepared by both of the S-FTM and perpendicular D-FTM films. These results are in well agreement considering the fact that  $\pi$ -conjugated backbones are highly aligned in single direction (D-FTM parallel) with respect to source and drain leading to the high drain currents as compared to films of S-FTM films owing to the random macromolecular arrangement in the channel region and one with conjugated backbone aligned in perpendicular direction of the channel [24,40].



**Fig. 6.** (a) Transfer characteristics of PBTTT-C14 films prepared with different methods ( $V_{DS} = -80$  V) and (b) Variation of saturation field-effect mobility of the OFETs fabricated with different PBTTT-C14 films.

Transfer characteristics of the fabricated FETs are shown in the Fig. 6 and parameters like field effect mobility ( $\mu$ ) and threshold voltage ( $V_{th}$ ) were calculated at the saturation regime in the transfer characteristics using the Equation (2).

$$I_{DS} = \frac{W}{2L} \mu C_i (V_{GS} - V_{th})^2 \quad (2)$$

The calculated  $\mu$  for S-FTM, D-FTM parallel and D-FTM perpendicular were found to be  $2.3 \times 10^{-2}$ ,  $1.1 \times 10^{-2}$  and  $8.6 \times 10^{-3}$   $\text{cm}^2/\text{V.s}$  respectively (Fig. 6 (b)). Mobility in parallel oriented films of D-FTM is found to be 4.7 times higher as compared to the isotropic films of S-FTM and 12.8 times higher than perpendicular D-FTM films. This provides a clear evidence of highly anisotropic transport in D-FTM owing to the one directional alignment of the main chains. This observed anisotropy in the oriented PBTTT-C14 films is much higher as compared to the previous report where the same film was subjected to the orientation by high temperature rubbing [36]. It is also worth to note here that such a high  $\mu$  in PBTTT-C14 films has only been achieved by spin coating with high boiling solvents followed by its annealing above to their thermotropic LC phase transition temperatures [30,32,39]. Herein once again we would like to emphasize that D-FTM utilized in this work not only offers a simple and quick preparation of oriented thin films but also leads to enhanced device performance of the polymeric OFETs.

**Table 1. Summary of device characteristics in OFET fabricated through S-FTM and D-FTM**

		Mobility ( $\text{cm}^2/\text{V.s}$ )	$V_{th}$ (V)	On/off ratio	Optical Anisotropy	Mobility Anisotropy
<b>S-FTM</b>		$2.3 \times 10^{-2}$	-30	$7.4 \times 10^4$	-	-
<b>D-FTM</b>	Parallel	$1.1 \times 10^{-1}$	-13	$2.8 \times 10^5$	6.5	12.8
	Perpendicular	$8.6 \times 10^{-3}$	-10	$1.2 \times 10^4$		

## 4. Conclusion

We have investigated the differences in the optical and electronic characteristics of floating-films of PBTTT-C14 prepared by static mode (S-FTM) and dynamic mode (D-FTM) of casting. Macroscopic orientation can be easily seen in the D-FTM films of PBTTT-C14 having a fairly good molecular orientation leading to the attainment of a high optical DR of 6.5. In contrast, isotropic floating-films were obtained when S-FTM was implemented for thin film fabrication. Absorption spectroscopic results support the increase in  $\pi$ -conjugation length in parallel D-FTM film as compared to S-FTM Film. Out-of-plane XRD pattern indicated that D-FTM films promote the more stretched lamella-layer structure with the edge-on orientation as compared to S-FTM films. Field-effect mobility estimated after OFET fabrication utilizing thin films fabricated by various method follows the order parallel D-FTM > S-FTM > perpendicular D-FTM. Interestingly, a high  $\mu$  of 0.11 cm<sup>2</sup>/V.s was found for the thin films of PBTTT-C14 fabricated by parallel D-FTM without any post-thermal annealing to its thermotropic LC temperature. It has been demonstrated that unlikely to known spin-coating and drop casting method preparation of thin films of CPs utilizing low boiling point solvents (D-FTM) serves as better strategy of film fabrication as compared to high boiling point solvent (S-FTM) especially for planer devices like OFETs.

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