ABSTRACT: Vacuum-processed diphenylbis(3-(pyridine-2-yl)phenyl)silane (2PTPS), diphenylbis(3-(pyridine-3-yl)phenyl)silane (3PTPS), and diphenylbis-(3-(pyridine-4-yl)phenyl)silane (4PTPS) have been used as electron-transporting host materials combined with tris(4-carbazoyl-9-ylphenyl)amine (TCTA) as the hole-transporting host, which induce balanced charge carrier transport for high-efficiency phosphorescent organic light-emitting diodes. The 4PTPS-based organic light-emitting diodes with tris[2-phenylpyridinato-C2,N]iridium(III) [Ir(ppy)3] dopant showed highest current efficiency and external quantum efficiency of 53.54 cd/A and 15.61%, compared to 2PTPS (40.75 cd/A, 11.84%) and 3PTPS (29.35 cd/A, 8.54%). These results were attributed to the well-aligned structure with preferential horizontal orientation of the emitting material layer by the diffraction intensity distribution as a function of azimuthal angle in two-dimensional grazing incidence X-ray diffraction analysis. The molecular orientation of TCTA:4PTPS material with a narrow azimuthal intensity distribution had better priority to the horizontal direction than the other TCTA:2PTPS and TCTA:3PTPS materials, which is related to the charge transport as well as the device efficiency. We found that the preferential horizontal orientation of the co-host material with a balanced charge carrier was not affected by Ir(ppy)3 dopant with a homoleptic structure and bis-[2-(4,6-difluorophenyl)pyridinato-N,C](picolinato)iridium [Firpic] dopant with a heteroleptic structure in the co-host/dopant system.

INTRODUCTION

The organic light-emitting diodes (OLEDs) are attracting much attention because of their advantages, such as self-emissive, flexible, and low power consumption for next-generation display and lighting applications, and various studies have been carried out to improving the device efficiency by the development of both host and dopant materials. The co-host (or mixed host) materials composed with hole-transporting (HT) and electron-transporting (ET) materials have been investigated due to their advantages of high efficiency by balanced charge carriers, low driving voltage, and controllable carrier mobility.1−6 Blue emitting phosphorescent OLEDs (PhOLEDs) with single host material have shown lower external quantum efficiency (EQE) than green, red, and orange PhOLEDs over 30%, but it reported a high efficiency of 29.5% by using mCP:B3PYPM:PM as the co-host and bis-[2-(4,6-difluorophenyl)pyridinato-N,C](picolinato)iridium [Firpic] dopant for horizontal orientation of the transition dipole moment in 2014.7 The synthesized universal ET host materials based on tetraphenylsilane with pyridine moieties produced high EQEs in bis(2-phenylbenzo-thiazolato-N,C)iridium(acetylacetonate) [Bt,Ir(acac)]-based orange-red (~35.5%), tris[2-phenylpyridinato-C2,N]-iridium(III) [Ir(ppy)3]-based green (~29%), and Firpic-based blue (~20.9%) due to high triplet energy (E_T) and electron mobility (μ_e) in solution-processed PhOLEDs. The ET host material can align the charge-carrier balance and broaden the electron-hole recombination zone by using the HT/ET co-host material in the emitting material layer (EML).8−11 Recently, some research groups suggested that the investigation of molecular orientation is known as one of the key factors for high device efficiency by improving the outcoupling efficiency. It has been theoretically estimated that the preferential molecular orientation of the emitters with a horizontal orientation can increase the EQE by up to 45%, which is higher by about 25−30% than that achieved by isotropically oriented molecules.11−13 One of the studies on organic molecular orientation of tris(4-carbazoyl-9-ylphenyl)amine (TCTA) with HT property showed different hole mobility and molecular orientation by different processes.
such as the face-to-face $\pi-\pi$ stacking in vacuum process and randomly oriented molecules in solution process. However, most studies on the molecular orientation of the emitting materials in conventional PhOLEDs have only been discussed about the dopant orientation with heteroleptic or homoleptic structure, different linker kinds, and linker positions, which are identified using indirect analysis methods on device characteristics. Kim and co-workers have investigated the molecular orientations of the Ir complexes/Firpic combination with preferred horizontal orientation in the co-host material. In this group, the effects of intermolecular interaction between host and dopant molecules on the emitting dipole orientation (EDO) of dopants are studied by comparison between emitting dipole distribution and intermolecular binding energies. The relationships between the molecular structure of Ir complexes and their EDO, which have different substituted positions of functional groups, such as strong electron-withdrawing trifluoromethyl group, elongated propyl group, planar, and charge conjugated phenyl group, were also studied. The transition dipole moments of the films were analyzed from the angle-dependent photoluminescence (PL) spectra. The Brütting group has investigated various molecular orientations; for example, three phosphorescent heteroleptic Ir complex dopants with different orientations of benzopyranopyridinone (bppo) and 2-phenylpyridinate (ppy) with isotropic dipole orientation, and acetylacetonate (acac) with relatively horizontal dipole orientation to the substrate can control the alignment of the emitting molecules to improving device efficiency. The horizontal orientation of fluorescent emitter Coumarin 6 depends on increase in the glass transition temperature due to decrease in the molecular surface mobility during deposition. The different orientations in the evaporated and solution-processed four different iridium complexes doped host materials were analyzed by the angular dependent PL analysis or dipole moments calculation using variable-angle spectroscopic ellipsometry. In another study on molecular orientation, the dipole–dipole potential of seven Ir complex phosphorescent emitters was strongly correlated with the transition dipole orientation using the spectral radiant intensity based on experimental and simulated data. The orientation emitters with various dopant ratios of 0–50 wt % and different dopant molecule structures in host/dopant system have been investigated by X-ray diffraction analysis.

In our previous works, we first reported the relationship between device efficiency and molecular orientation on the nondoping fluorescence OLEDs of different molecular orientations with two linker types through direct-analysis methods using two-dimensional grazing incidence X-ray diffraction (2D-GIXD) analysis. In thermally activated delayed fluorescence OLEDs, we found that the molecular orientation of the emitting materials had different horizontal and random orientations as a function of linker positions of para and meta, which showed 12.5 times difference of the device efficiency. This improved device efficiency was confirmed by the enhanced out-coupling efficiency of a well-aligned stacking structure in the EML with a horizontal molecular orientation to in-plane and out-of-plane analysis using 2D-GIXD.

In this study, we investigated the relationship between the vacuum-processed PhOLED properties and the molecular orientations of $n$PTPS ($n = 2, 3, 4$) as host materials using 2D-GIXD analysis in high-efficiency PHOLEDs. The nPTPS ET host materials have many advantages, such as good morphological stability and high ET property for balanced charge-carrier transport by pyridyl-substituted linker in solution-processed PhOLED, as reported in the literature. In particular, we analyzed the 2D-GIXD patterns and the out-of-plane spectra of the co-host and co-host/dopant molecules, as well as the azimuthal intensity profiles for a plot of orientation distributions of molecules. The research on preferential orientation of the host material has been discussed for the first time using 2D-GIXD analysis, and is believed provide new evidence for identifying the cause of device efficiency.

![Chemical structures and triplet-energy values of (a) 2PTPS, 3PTPS, and 4PTPS electron-transporting host materials and (b) Ir(ppy)$_3$, and Firpic dopant materials. (c) Energy-band diagram of PHOLEDs.](image)
RESULTS AND DISCUSSION

Figure 1a,b show the chemical structures of the nPTPS composed of tetrahedral silicon and two meta-pyridine substituents, as well as two dopant materials. To obtain high device efficiency, the energy levels of each layer were considered with a balanced charge carrier, and Figure 1c shows the energy-band diagram of the fabricated PhOLEDs. The near-edge X-ray absorption fine structure (NEXAFS) spectra of the TCTA:nPTPS (n = 2, 3, and 4) films in Figure S1 are assigned to the \( \pi^* (C=C) \) orbital at 284.5 eV, the \( \sigma^* (C=H) \) orbital mixed with Rydberg orbitals around (287–291) eV, and \( \sigma^* (C=H) \) or \( \sigma^* (C=C) \) orbitals in the energy range (292–307) eV (see the Supporting Information). The tilting angle \( \alpha \) between the C=C double bond in the conjugated planes and the substrate surface with a photon beam size (0.1 mm × 0.3 mm) were determined. The inset in Figure S1 is the average tilt angle of approximately (55 ± 5)\(^\circ\).

The \( \pi^* \) resonance intensity of the TCTA:nPTPS (n = 2, 3, and 4) films shows similar values with increase in the incidence angle \( (\theta) \). The crystallinity as the C=C/C=H ratio of the films is insignificant because all films have randomly oriented distributions.

Figure 2 shows the 2D-GIXD patterns for vacuum-processed co-host (TCTA:nPTPS) films on SiO\(_2\)/Si substrate, respectively. In the 2D-GIXD patterns, \( q_{xy} \) or \( q_x \) means an in-plane or an out-of-plane component of the scattering vector, \( q \), respectively. Figure 2a–c show that all of the 2D-GIXD patterns for the TCTA:nPTPS films consistently present a weak and broad diffraction peak because vacuum-processed molecules usually tend to be randomly oriented on the substrate due to the weak molecular interaction. Figure 2d shows the out-of-plane intensity profiles, in which a broad diffraction peak is evident at \( q_z = 1.43 \) Å\(^{-1}\), induced by vacuum-processed planar TCTA molecules with an amorphous structure. The planar packing distance \( (d = 2\pi/q_x) \) of all the vacuum-processed TCTA:nPTPS is estimated to be 4.4 Å. It is similar to the face-to-face \( \pi-\pi \) stacking distance \( (d \sim 3.8 \) Å) because of the loosely packed and rough structure of the vacuum-deposited TCTA molecules with a weak intermolecular interaction. Additionally, all of the 2D-GIXD patterns of the TCTA:nPTPS films show anisotropic intensity distributions. In particular, relatively strong diffraction intensity is distributed along the out-of-plane direction, which implies that the planar packing of the TCTA:nPTPS molecules is preferentially distributed along the out-of-plane direction. To compare the planar packing distributions of the TCTA:nPTPS films, Figure 2e shows the diffraction intensity distributions as a function of azimuthal angle. All of the TCTA:nPTPS molecules...
present a maximum intensity at \( \chi = 0 \), indicating a flat-on orientation. Vacuum-processed TCTA:4PTPS film shows the narrowest azimuthal intensity distribution, whereas vacuum-processed TCTA:2PTPS and TCTA:3PTPS present relatively broad azimuthal intensity distributions. This implies that the TCTA:4PTPS film has more flat-on orientation compared to...
TCTA:2PTPS and TCTA:3PTPS, as described in Figure 2f,g. In general, the flat-on orientation along the out-of-plane directions induces a fast charge transport due to the formation of delocalized states by the overlapping of π-orbitals. Therefore, the vacuum-processed TCTA:4PTPS shows priority in the horizontal orientation, compared to TCTA:2PTPS and TCTA:3PTPS.

Figures 3 and 4 shows the 2D-GIXD for vacuum-processed TCTA:nPTPS:Ir(ppy)$_3$ and TCTA:4PTPS:Firpic films on SiO$_2$/Si substrate, respectively. All of the 2D-GIXD patterns for vacuum-processed TCTA:nPTPS:Ir(ppy)$_3$ and TCTA:4PTPS:Firpic also similarly exhibit anisotropic ring pattern at $q_z = 1.43$ Å$^{-1}$, which is the same as discussed in Figure 2 and literature. In the out-of-plane intensity profiles for TCTA:nPTPS:Ir(ppy)$_3$ (Figure 3e), all films show a weak diffraction peak at $q_z = 0.75$ Å$^{-1}$, which originates from the diffraction of the (220) plane of Ir(ppy)$_3$. The TCTA:4PTPS:Firpic films show the same feature as the nondoped and Ir(ppy)$_3$-doped TCTA:nPTPS because the small doping concentration of 5 wt % does not affect the peak, which originates from the fully amorphous TCTA (Figure 4e).

Figures 3f and 4f show the azimuthal intensity plot of the TCTA:nPTPS:Ir(ppy)$_3$ or TCTA:4PTPS:Firpic films. The full width at half maximum for azimuthal intensity distributions of TCTA:4PTPS, TCTA:4PTPS:Ir(ppy)$_3$, and TCTA:4PTPS:Firpic are estimated as 99.4, 71.6, and 89° by Gaussian model fitting, respectively. The co-host/dopant molecules of TCTA:4PTPS:Ir(ppy)$_3$ and TCTA:4PTPS:Firpic show a slightly increased flat-on orientation, compared with the host/host molecule of TCTA:4PTPS film. The enhanced flat-on orientation in the co-host/dopant molecule probably arises from the slight increase in the intermolecular interaction by the addition of a dopant because the linear binding geometry of the co-host/dopant molecule with a larger binding energy more strongly causes the building blocks to align horizontally along the substrate than in the host/host or dopant/dopant system.

Similar to the TCTA:4PTPS film, the TCTA:4PTPS:Ir(ppy)$_3$ and TCTA:4PTPS:Firpic films showed the strongest and the narrowest intensity distributions near $\chi = 0^\circ$, implying a higher flat-on orientation than Ir(ppy)$_3$ or Firpic-doped TCTA:2PTPS and TCTA:3PTPS films. The results of the 2D-GIXD analysis show that the predominant features of the molecular orientation in the host/host (TCTA:nPTPS) and/or co-host/dopant (TCTA:nPTPS:Ir(ppy)$_3$, or TCTA:4PTPS:Firpic) systems are not affected by the homoleptic Ir(ppy)$_3$, or heteroleptic Firpic dopant with an almost isotropic emitting dipole orientation, as described in Figures 3f,g or 4f,g. It can be proposed that the orientation of the host material determines the preferential orientation in the EML.

To compare the orientation ratio between nPTPS molecules, the parameters of the crystal orientation factor to a set of hkl planes were calculated using the equation $\langle \cos^2 \phi \rangle_{hkl}$ for a set of hkl planes and Herman’s orientation function $f_2$ and Tables 1, 2, and 3 summarize the parameters. The orientation parameter $\langle \cos^2 \phi \rangle_{hkl}$ with azimuthal angle $\phi$ has the value of unity, and the parameter $f_2$ of Herman’s orientation function is used to describe the degree of orientation. The $f_2$ values of the emitting materials in co-host or co-host/dopant systems are close to 0.1, whereas the films with 4PTPS host have relatively higher values than 2PTPS or 3PTPS. This means that the orientation of 4PTPS is mostly parallel to the reference direction because $f_2$ is unity when the normal of the reflection plane is parallel to the reference direction, whereas the value of $f_2$ is zero. However, it is perpendicular to the reference direction.

The vacuum-processed Ir(ppy)$_3$-doped devices show similar current–density curves and high luminance of 23 630, 11 040, and 22 950 cd/m$^2$ in 2PTPS, 3PTPS, and 4PTPS emitters, respectively, as shown in Figure 5a,b. Among them, the current efficiency (CE) and EQE of the Ir(ppy)$_3$-doped 4PTPS device shows the highest values of 53.54 cd/A and 15.61%, respectively, compared with the devices with 2PTPS (40.75 cd/A, 11.84%) and 3PTPS (29.35 cd/A, 8.54%), as shown in Figure 5c,d. The highest efficiencies of the TCTA:4PTPS:Ir(ppy)$_3$ device can be explained by the following effects: (i) as calculated in literature, the solution-processed nPTPS host materials have a higher $\mu_2$ of $\sim 10^{-5}$ cm$^2$/V s in the order of 4PTPS $<$ 3PTPS $<$ 2PTPS than the 2,2’,2”-(1,3,5-benzinetriyl)-tris(1-phenyl-1-H-benzimidazole) (TPBi) electron-transporting layer (ETL) with $\mu_2$ of $\sim 10^{-3}$ cm$^2$/V s, but it has different device structure compared with our vacuum-processed device. In Ir(ppy)$_3$-doped devices, the higher $E_T$ of 2PTPS (2.82 eV), 3PTPS (2.82 eV), and 4PTPS (2.90 eV) host materials than Ir(ppy)$_3$ ($\sim 2.4$ eV) dopant, nPTPS can prevent reverse energy transfer from the dopant to the host. (ii) From the energy level of the highest occupied molecular orbital (HOMO) and the lowest unoccupied molecular orbital of 2PTPS ($\sim 6.47$, $\sim 2.4$ eV), 3PTPS ($\sim 6.50$, $\sim 2.30$ eV), and 4PTPS ($\sim 6.55$, $\sim 2.27$ eV), the proper exciton recombination of 4PTPS might be explained as the hole-blocking effect due to the deep HOMO level of 4PTPS, which effectively confines carriers within the emitting layer. (iii) The preferential orientation of the 4PTPS host material has a higher flat-on orientation with strong and narrow intensity distributions in the azimuthal intensity plot compared with 2PTPS and 3PTPS host materials, as shown in Figures 2–4.

To confirm the preferential host orientation, the vacuum-processed Firpic-doped devices with a heteroleptic structure were investigated and shown to have similar tendency as the Ir(ppy)$_3$-doped devices with a homoleptic structure, as shown in Figure 6. The device with TCTA:4PTPS:Firpic showed similar current–density curves and high luminance, CE, and EQE (7874 cd/m$^2$, 11.08 cd/A, and 5.95%) compared with 2PTPS (5835 cd/m$^2$, 6.2 cd/A, and 3.15%) and 3PTPS (4130 cd/m$^2$, 6.52 cd/A, and 3.41%) devices. The featured device performance in TCTA:4PTPS:Firpic is believed to be due to the priority

#### Table 1. Orientation Parameters of TCTA:Films for the Host/Host System

| Crystal orientation factor | 2PTPS | 3PTPS | 4PTPS |
|-----------------------------|-------|-------|-------|
| $\cos^2 \phi$               | 0.378 | 0.381 | 0.396 |
| $f_2$                       | 0.067 | 0.071 | 0.095 |

#### Table 2. Orientation Parameters of TCTA:nPTPS:Ir(ppy)$_3$ Films for the Co-Host/Dopant System

| Crystal orientation factor | 2PTPS | 3PTPS | 4PTPS |
|-----------------------------|-------|-------|-------|
| $\cos^2 \phi$               | 0.376 | 0.384 | 0.388 |
| $f_2$                       | 0.065 | 0.076 | 0.082 |

#### Table 3. Orientation Parameters of TCTA:nPTPS:Firpic Films for the Co-Host/Dopant System

| Crystal orientation factor | 2PTPS | 3PTPS | 4PTPS |
|-----------------------------|-------|-------|-------|
| $\cos^2 \phi$               | 0.376 | 0.372 | 0.382 |
| $f_2$                       | 0.064 | 0.058 | 0.073 |
of the host molecular orientation to the horizontal direction, and we found that the preferential orientation was not affected by Firpic or Ir(ppy)$_3$ dopants.

**CONCLUSIONS**

By comparing the 2D-GIXD analysis and device characteristics, the preferential host orientation to the horizontal direction in the emitting material can be one of the main factors for...
improving device efficiency, and, in particular, priority of the host material was not affected by the dopant materials with a homoleptic or a heteroleptic structure. The Ir(ppy)$_3$- and Firpic-doped TCTA:4PTPS devices with a preferential horizontal orientation showed highest CE and EQE due to balanced hole and electron recombination in the EML with the well-aligned stacking structure in the horizontal direction, compared with 2PTPS and 3PTPS materials with a relatively random orientation. This correlation between the preferential orientation of charge-balanced HT-host/ET-host material and device characteristics can contribute to device efficiency and useful molecule design of emitting materials.

**EXPERIMENTAL DETAILS**

**Materials.** nPTPS ($n = 2, 3$, and $4$) were synthesized in Gyeongsang National University, and detailed synthetic procedures are given in the literature.\(^8\) We used Ir(ppy)$_3$ and Firpic dopants with sublimed grade ($\sim 99.7\%$) from Ossila. The 4,4′-cyclohexylidenebis[4,4-bis(4-methylphenyl)benzenamine] (TAPC), TCTA, and 2,2′-((1,3,5-benzenetriyl)-tris(1-phenyl-1-H-benzimidazole) (TPBi) of sublimed grade ($\sim 99.5\%$) were purchased from Lumtec Co.

**Microstructure Analysis and Device Characterization.** Near-edge X-ray absorption fine structure (NEXAFS) and 2D-GIXD measurements were, respectively, carried out using the 4D and 9A U-SAXS beamline of the Pohang Light Source (PLS-II) in the Pohang Accelerator Laboratory. Detailed X-ray measurement procedures are given in the literature.\(^12,25\) Electrical and optical characteristics of PhOLEDs were measured by the Keithley 236 source meter unit and CS-1000 Microstructure Analysis and Device Characterization.

**Device Fabrication.** The indium tin oxide-coated glass substrates were used as anode, cleaned with deionized water, acetone, and isopropanol alcohol for 15 min using an ultrasonic bath, and dried in the oven for 1 day. Before device fabrication, the substrate was treated under O$_2$ plasma for 60 s. In our vacuum process, molbdenium oxide (MoO$_3$, 5 nm) and TAPC (70 nm) were used as hole-injection and -transporting materials. Ir(ppy)$_3$ and Firpic dopants were relatively co-evaporated with 5 wt % ratio during the EML deposition. TPBi (30 nm), 8-hydroxyquinolatolithium (Liq, 1 nm), and aluminum (Al, 120 nm) were continuously evaporated and used as ETL, interlayer, and cathode, respectively.

**ASSOCIATED CONTENT**

Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acsomega.8b01358.

Comparison of crystallinity changes of the emitting TCTA:nPTPS ($n = 2, 3$, and $4$) materials by the NEXAFS spectra (PDF)

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**NOTES**

The authors declare no competing financial interest.

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