

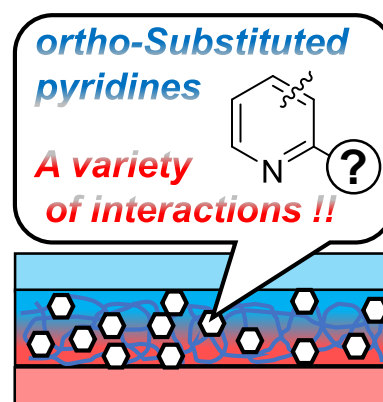
# Accounts of Materials & Surface Research

## *ortho*-Substituted pyridyl groups in adaptable adhesive materials

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Adhesive materials have been used in industrial manufacturing, contributing to not only energy saving but also development of creative products. Adhesive materials are now desired to bond dissimilar substrates, which the two surfaces are chemically different from each other. Then, many researchers have studied and developed adaptable adhesive materials for all-around adhesion. Catechol group is one of useful functional groups for this purpose, and it is incorporated into synthetic polymers (biomimetic adhesives). On the other hand, an analogue of catechol, *ortho*-substituted pyridines also have a potential for adaptation to surfaces of dissimilar substrates. In this paper, a series of *ortho*-substituted pyridines was introduced, especially about their unique chemical properties and their application. We have applied several kinds of them to adaptable photo-adhesive materials, and designed monomers using with photo-initiators. Furthermore, *ortho*-Hydroxypyridine and *ortho*-mercaptopyridine are convenient for syntheses in air, due to their tautomerization reactions. These chemicals could be named as “*mimic molecular structure*”. Photo-adhesion of dissimilar substrates is then performed by using our materials incorporating *ortho*-substituted pyridyl groups. The results are summarized, and roles of these functional groups in the adhesive layer are discussed.



**Keyword:** *ortho*-Substituted pyridyl group, Adaptable adhesive materials, Dissimilar substrate, Mimic molecular structure, Photo-adhesion

Masahiro Furutani obtained his PhD under the guidance of Professor Kazuaki Kudo in 2013 at The University of Tokyo. He joined the research group of Professor Koji Arimitsu at Tokyo University of Science in 2014 as an assistant professor. After spending six years working on the latent reagents for UV curing and photo-patterning, he moved to Fukui prefecture where he was born, and now is an associate professor at Department of Chemistry and Biology, National Institute of Technology, Fukui College. His current research interests are in developing and researching polymer materials for sustainable society.



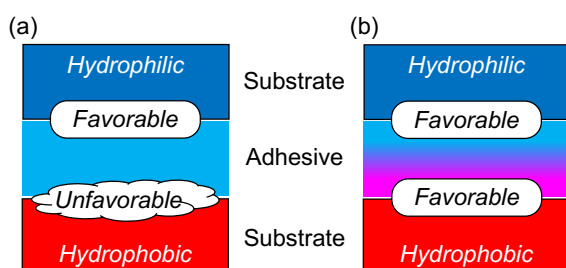
# *ortho*-Substituted pyridyl groups in adaptable adhesive materials

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## 1. Adaptable adhesive materials

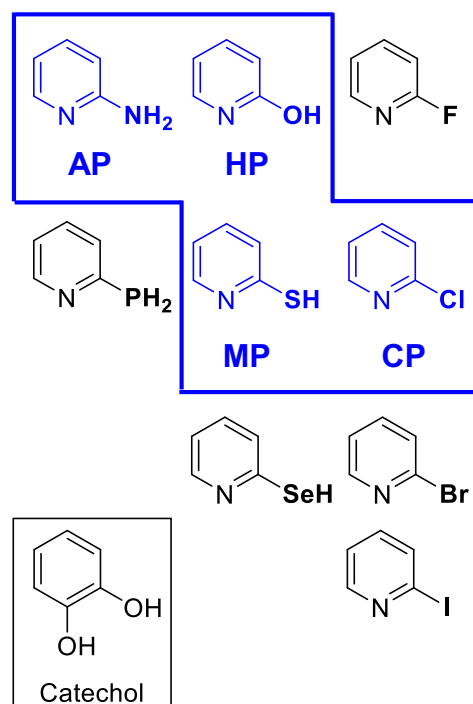
To produce lightweight industrial products made from multi-materials, adhesives which bond dissimilar materials are indispensable. Adherends are glass, ceramics, metals, polymers, woods and so on. It is difficult to bond arbitrary two adherends strongly. For example, when a substrate of hydrophilic surface is bonded to another substrate of hydrophobic surface, a hydrophilic adhesive does not work well (Fig. 1(a)). To overcome this situation, it is required that the adhesive chemically adapts for the hydrophobic surface, by making itself hydrophobic at the surface in some way (Fig. 1(b)).



**Figure 1.** A hydrophilic adhesive to bond a hydrophilic substrate to a hydrophobic one. (a) Not bonding well, and (b) bonding well by adapting for the hydrophobic surface by making itself hydrophobic at the surface.

Adhesive materials could acquire such adaptability, by bearing chemical functional groups which would take various kinds of chemical interactions. Catechol group and its analogues are one of representative functional groups. Catechol group has a benzene ring and two hydroxy groups, and

interacts with a substrate surface through hydrogen bonding,  $\pi$ - $\pi$  interaction, metal coordination, covalent bonding and so on.<sup>1)</sup> Catechol group is found from adhesive proteins of marine lives such as mussels.<sup>2)</sup> Synthetic polymers bearing catechol groups have been studied for strong adhesion between dissimilar substrates.<sup>3-7)</sup> On the other hand, we have featured another analogue of catechol which include heteroatoms except for oxygen atom. We have applied *ortho*-substituted pyridines to fabrication of adaptable adhesive materials.



**Figure 2.** A series of *ortho*-substituted pyridines. Four blue-colored ones (AP, HP, MP and CP) are incorporated into adaptable adhesive materials in our study. The chemical structure of catechol is also shown.

## 2. *ortho*-Substituted pyridines

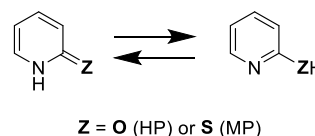
Substituted pyridines consist of a pyridine ring and a heteroatom-containing functional group. A series of *ortho*-substituted pyridines is shown in Fig. 2. A pyridine ring would interact with a substrate surface through hydrogen bonding,  $\pi$ - $\pi$  interaction, and metal coordination. It is known that many kinds of metals such as Co, Rh, Ni, Pd, Cu and Au are coordinated by the pyridine ring.<sup>8-11</sup> Thus, it is expected that substituted pyridines could work as catechol in adaptable adhesives.

A functional group at *ortho*-position has a wide variety, and give the pyridine ring individuality. *ortho*-Aminopyridine (AP) has been studied for development of an anticancer agent<sup>12</sup> or an organocatalyst.<sup>13</sup> Amino group would work as an electron-donating group, and the pyridine-nitrogen atom would have negative charge. Use of *ortho*-phosphinopyridine was limited to metal coordination in organometallic complexes.<sup>8,14</sup>

*ortho*-Hydroxypyridine (HP) was also used as a ligand of an organometallic catalyst for C-C coupling reactions.<sup>15</sup> On the other hand, Zhu *et al.* reported an ionic liquid for CO<sub>2</sub> capture which were prepared with HP and a superbase.<sup>16</sup> *ortho*-Mercaptopyridine (MP) would modify chemically Cu-based metallic surfaces, due to strong sulfur-Cu interactions.<sup>17,18</sup> Furthermore, Lipshutz *et al.* proposed a recyclable coupling reagent (2,2'-dipyridyldithiocarbonate, DPDTC) that was synthesized with MP and triphosgene.<sup>19</sup> *ortho*-Selenopyridine was used as a ligand of Zn complexes for medical applications.<sup>20,21</sup>

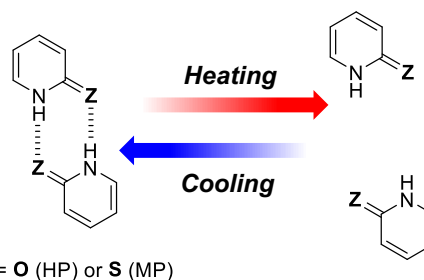
HP and MP have an additional unique character, that is, tautomerization reaction, as shown in Fig. 3. Keto-isomer (for HP) or thione-isomer (for MP) is dominant in the solid

and liquid phases.<sup>22,23</sup> Interestingly, the keto-enol or thione-thiol equilibrium is changed by experimental conditions such as temperature, concentration and solvent.<sup>24-27</sup> In the case of MP, for example, by heating, diluting and/or using a nonpolar solvent, the equilibrium would tend toward the thiol isomers. Similarly, surface polarity of a substrate would also affect the tautomerization reactions in adhesion between dissimilar substrates.



**Figure 3.** Tautomerization reactions of HP and MP.

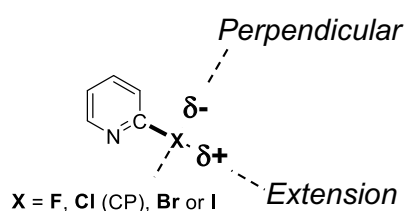
As shown in Fig. 4, keto-isomer (for HP) and thione-isomer (for MP) would form double hydrogen bonds.<sup>28</sup> Hydrogen bonding ability of HP is stronger than that of MP, while polarization of MP is larger than that of HP. In general, binding energy of hydrogen bonds is 5-100 kJ/mol (*cf.* covalent bonds: >300 kJ/mol).<sup>29</sup> These double hydrogen bonds would work as cross-linking points in an adhesive layer, which would ensure high cohesion force of the layer. Furthermore, hydrogen bonds are abolished by heating, and re-formed by cooling to room temperature. This would be applied for thermal dismantlement of an adhesive layer and re-adhesion using the adhesive residue.



**Figure 4.** Formation of a double hydrogen bond between two HPs or MPs.

*ortho*-Fluoropyridines were studied for stabilization of  $\text{Li}^+$  ion in batteries,<sup>30)</sup> or that of an organic electrophilic compound in organic syntheses.<sup>31)</sup> *ortho*-Chloropyridines (CP) were grafted on a carbon nanotube electrode, resulting in enhancement of electroreduction of  $\text{CO}_2$  to  $\text{CO}$ .<sup>32)</sup> Other two *ortho*-halopyridines were used as reagents for introducing a pyridine building block to a synthetic molecule where halide ions were eliminated.<sup>33,34)</sup>

It is also known that a halogen group has a unique charge distribution (Fig. 5).<sup>35)</sup> In a carbon-halogen (C-X) bond, the electron density decreases along the extension of the C-X bond, while the density increases in the direction perpendicular to the bond. For example, a halogen bond (C-Cl...N) can be formed between two CPs, which has one fourth or one fifth of binding energy of a hydrogen bond.<sup>36)</sup> Nevertheless, these halogen bonds could also work as cross-linking points in an adhesive layer, leading to enhancement of the cohesive force.

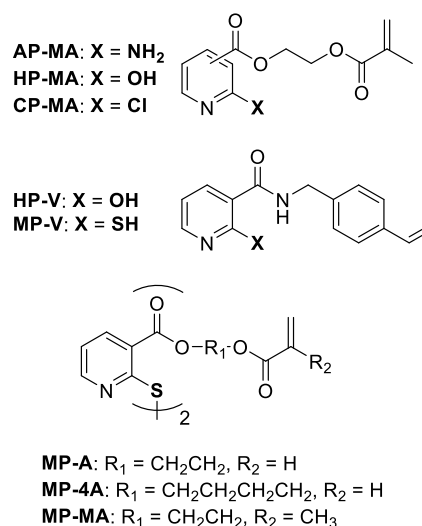


**Figure 5.** Charge distribution around a halogen group (C-X bond).

Utilizing the chemical features of *ortho*-substituted pyridines mentioned above, we have incorporated four kinds of *ortho*-substituted pyridyl groups (AP, HP, MP and CP groups) into adaptable adhesive materials, not only for adhesion between dissimilar substrates, but for thermal dismantlement and reuse of the adhesive residues.

### 3. Synthesis of monomers bearing an *ortho*-substituted pyridyl group

To incorporate *ortho*-substituted pyridyl groups into our photo-adhesive materials, methacrylate (MA), vinyl (V) and acrylate (A) monomers were designed (Fig. 6). Using these monomers with a radical photo-initiator, photo-adhesive materials were prepared.



**Figure 6.** Methacrylate (MA), vinyl (V) and acrylate (A) monomers bearing an *ortho*-substituted pyridyl group, synthesized for our adaptable adhesive materials.

MA and A monomers were synthesized under acidic conditions where thionyl chloride and a corresponding nicotinic acid were starting compounds. V monomers were synthesized under neutral conditions. **AP-MA** was mainly obtained as a sulfate salt.<sup>37)</sup> After UV-Vis spectral measurements of each monomer solution, it was found that HP derivatives (**HP-MA** and **HP-V**) and **MP-V** were mainly present as their keto-isomers,<sup>38-40)</sup> while other derivatives remained their pyridine ring. Monomers were obtained as solid, except for **CP-MA** (obtained as a colorless liquid).<sup>41)</sup>

Three MP derivatives (**MP-A**, **MP-4A** and

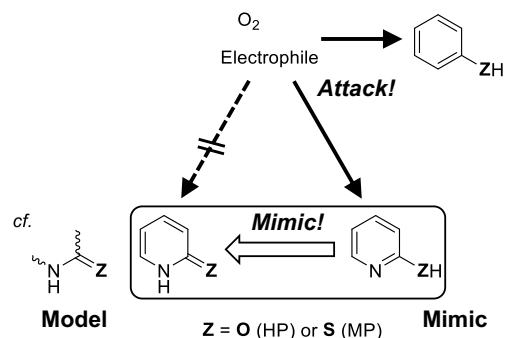
**MP-MA**) were obtained as disulfide dimers.<sup>42-</sup>

<sup>44)</sup> In the synthetic reaction of 2-mercaptopyridine with thionyl chloride, MP groups would be oxidized to dipyridyl disulfides. Fortunately, **MP-4A** and **MP-MA** were purified by recrystallization processes.

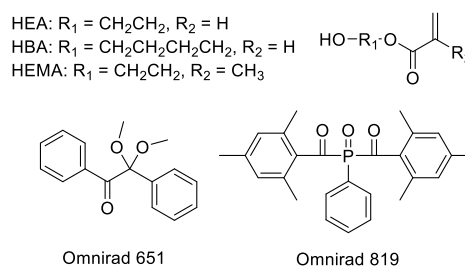
In general, both phenolic hydroxy group and thiol group are not stable under oxidative conditions. On the other hand, HP and MP compounds are relatively stable due to tautomerizing to keto-isomers, which enables to store in a long term, or to synthesize in air under neutral conditions (Fig. 7). This behavior of compounds looks like mimicry of insects. Mimicry consists of (i) a mimic itself, (ii) a model for the mimic, and (iii) those who recognize the mimic. In cases of HP and MP, (i) enol isomer (or thiol isomer), (ii) amide (or thioamide), and (iii) oxygen and other electrophiles. Such chemical structure could be termed “*mimic molecular structure*”.

#### 4. Incorporating *ortho*-substituted pyridyl group into adaptable adhesive materials

Compositions of our adaptable photo-adhesive materials, irradiation conditions,



**Figure 7.** HP and MP as “*mimic molecular structure*”.



**Figure 8.** Chemical structure of comonomers and photo-initiators in our adaptable photo-adhesive materials.

substrates, and the maximum shear stress of each case are summarized in Tables 1 and 2. Chemical structure of comonomers and photo-initiators is shown in Fig. 8. UV irradiation was performed at a wavelength of

**Table 1.** Compositions of our adaptable photo-adhesive materials and irradiation conditions for them.

Compositions				Irradiation conditions	
<i>ortho</i> -Substituted pyridyl group (mol% vs comonomer)	Comonomer	Photo-initiator (wt% vs monomers)	Solvent <sup>b</sup>	Intensity [mW/cm <sup>2</sup> ]	Energy [J/cm <sup>2</sup> ]
Run 1 <b>AP-MA</b> (0.34)	HEMA	Omnirad 651 (2)	-	3.7	1
Run 2 <b>AP-MA</b> (1.0) <sup>a</sup>		Omnirad 819 (2)		50	2
Run 3 <b>HP-MA</b> (26)		Omnirad 651 (2)	THF	2.9	1
Run 4 <b>CP-MA</b>	-		CHCl <sub>3</sub>	50	2
Run 5 <b>HP-V</b> (1.7)	HEMA	Omnirad 651 (10)	-		6
Run 6 <b>MP-V</b> (1.3)			THF		4
Run 7 <b>MP-A</b> (5.0)	HEA	Omnirad 819 (20)	THF	3.7	1
Run 8 <b>MP-4A</b> (1.2)	HBA	Omnirad 651 (2.4)	-		2
Run 9 <b>MP-MA</b> (2.7)	HEMA	Omnirad 651 (2)	CHCl <sub>3</sub>	50	2

<sup>a</sup>Adding equimolar amount of 2-(diethylamino)ethylmethacrylate.

<sup>b</sup>Minimum amount for dissolution of monomers and photo-initiator.

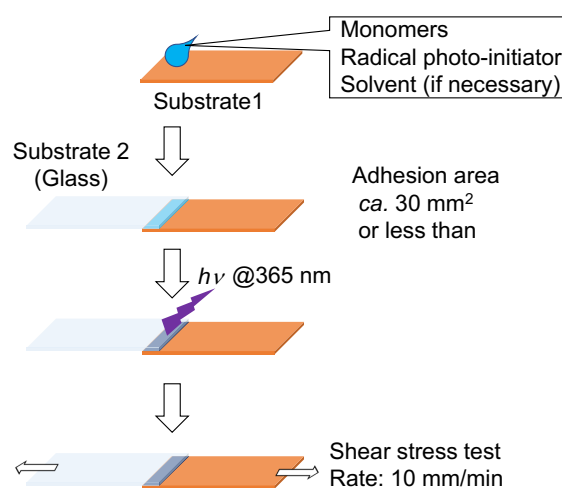
365 nm in all photo-adhesion experiments. The general procedure for fabrication of our photo-adhesive samples is shown in Fig. 9.

**AP-MA** were mixed with HEMA without any solvents (Run 1 and Run 2). Strong adhesion was accomplished, especially toward metallic substrates. X-ray photo-electron spectroscopic (XPS) measurements of the adhesive residue on a glass (Glass) substrate implied that pyridine-nitrogen atom could contribute to interaction.<sup>37)</sup> An organic base, 2-(diethylamino)ethyl methacrylate, was added for anion exchange to *in situ* generate free AP groups in the adhesive layer (Run 2).<sup>45)</sup> Ratio of **AP-MA**, irradiation intensity and energy were also increased. As a result, adaptability of the adhesive layer seemed to be improved.

HP groups would also contribute to strong adhesion toward Glass, metallic and plastic substrates (Run 3 and Run 5).<sup>38,39)</sup> HP groups would favor both Glass and Cu substrates. XPS measurements of the adhesive residues revealed that both nitrogen and oxygen

atoms would contribute to interact with a Cu surface.<sup>38)</sup> In the case of **HP-V** (Run 5), more than 1.9 MPa of shear stresses were recorded about all photo-adhesive samples. This implied that a HP group at PP surface could tautomerize into the enol-isomer, and that it could work as a hydrophobic group.

The results on **CP-MA** (Run 4) implied a tendency of compatibility of CP groups toward substrates, because comonomers



**Figure 9.** The general procedure for fabrication of photo-adhesive samples in our study.

**Table 2.** Results on shear stress measurements of our adaptable photo-adhesive materials. The maximum value is shown for each adhesive sample (unit: MPa, PVC: polyvinylchloride).

ortho-Substituted pyridyl group (mol% vs comonomer)		Substrate used with another Glass substrate						
		Glass	Cu	treated-Cu <sup>b</sup>	SUS	Al	PVC	PP
Run 1	<b>AP-MA</b> (0.34)	11.3	5.4	6.3	4.5	<b>9.9</b>		0.0
Run 2	<b>AP-MA</b> (1.0) <sup>a</sup>	<b>15.5</b>	5.2	6.2	6.8	3.9	<b>4.4</b>	0.3
Run 3	<b>HP-MA</b> (26)	13.1	5.9					
Run 4	<b>CP-MA</b>	6.2	4.1	2.5	1.7	2.7		0.6
Run 5	<b>HP-V</b> (1.7)	9.7	6.6	<b>13.6</b>	<b>14.1</b>	6.0	2.7	<b>1.9</b>
Run 6	<b>MP-V</b> (1.3)	6.0	3.3	2.9		1.2		
Run 7	<b>MP-A</b> (5.0)	1.2	0.8			0.3	1.0	
Run 8	<b>MP-4A</b> (1.2)	2.5	0.9	1.0	1.4	0.6	1.3	0.3
Run 9	<b>MP-MA</b> (2.7)	7.6	<b>12.1</b>					

<sup>a</sup>Adding equimolar amount of 2-(diethylamino)ethylmethacrylate.

<sup>b</sup>Immersing a Cu substrate in conc. nitric acid for 30 s, followed by washing in distilled water.



were not included in the adhesive layer. The order of shear stress was Glass (33°, water contact angle<sup>43)</sup>) > Cu (85°) > Al (71°) > treated-Cu (28°) > SUS (50°) > PP (92°), which was not necessarily consistent with the order of hydrophilicity of substrate surfaces (SUS: stainless, PP: polypropylene). Other factors such as anchor effect could influence the order of adhesive strength.

In the cases of **MP-V** and **MP-A** (Run 6 and Run 7), shear stress values were low overall,<sup>40,42)</sup> probably due to too much amounts of photo-initiators. In Run 8, all-around adhesive property was shown with **MP-4A** and HBA.<sup>43)</sup> By using **MP-MA** with HEMA (Run 9), a Cu substrate was strongly adhered to a Glass substrate, which would result from sulfur-Cu interactions. MP would be present as the oxidized state in cases of Run 7-9. Nevertheless, dipyrindyl disulfide moiety would interact with a Cu surface.<sup>46)</sup>

## 5. Dismantlement and re-adhesion

It was confirmed that adhesive layers fabricated with **CP-MA** (Run 4) and **HP-V** (Run 6) were thermally dismantled. Adhesive layers containing catechol groups would not be dismantled with heat, because catechol groups could cause unexpected side reactions such as oxidization and subsequent polycondensation. A Glass-Glass photo-adhesive sample was heated around 110 °C (Run 4)<sup>41)</sup> or 200 °C (Run 6)<sup>39)</sup> for the dismantlement, which the adhesives would work as hot-melt ones. Furthermore, at least five times of re-adhesion was also accomplished in both cases. Noncovalent cross-linking through halogen or double-hydrogen bonds would enable the thermal dismantlement and re-adhesion. Similar verification is in progress using MP series which include dynamic covalent bonds

instead of noncovalent ones.

## 6. Conclusion

Adhesive properties of *ortho*-substituted pyridyl groups could surpass those of catechol groups. Because, they have a pyridine-nitrogen atom, and the pyridine ring can customize its *ortho*-substituted functional group that a catechol does not have. Adaptable photo-adhesive materials in our study contained AP, HP, MP or CP groups, and showed not only moderate to strong adhesion toward all-around substrates, but also thermal dismantlement and re-adhesion. These *ortho*-substituted pyridyl groups would be incorporated into both side and main chains of cross-linked polymer networks. Furthermore, it was found that HP and MP groups work as “*mimic molecular structure*” which were well tolerated in oxidization. Thus, adaptable adhesive materials using *ortho*-substituted pyridines are one of good candidates for the next generation of adhesive materials in future sustainable society and circular economy.

## 7. Acknowledgement

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