



## REVIEW

### Model Compounds Used in Research of Coal Conversion and Utilization

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Advances in the research of coal chemistry by the model compounds were reviewed. With the development of technology, the molecular-level information of coal was increasingly demanded in studying effective utilization of coal. Comparing with the disorderly and unsystematic organic-groups in the coal, the known functional group in the coal-related model compounds is easier to be traced and measured. As a kind of simplified approach, the model compounds help researchers to understand the details in the processes of coal-related reactions. Unlike the direct investigation on coal, the indagation through coal-related model compounds not only exposes the micro-level variation in typical structures of coal, but also finds out the optimum conditions and influential factors which controlling the whole reaction process. Simultaneously, varieties of possible reaction paths were raised and discussed. Notably, some external additives affect the magnitude and rate of the coal-related model compounds reactions significantly. In the future, the further studies in virtue of coal-related model compounds will continually contribute to the coal industry.

**Keywords:** Coal model compounds, Pyrolysis, Liquefaction, Sulfur, Nitrogen, Low-temperature oxidation.

## INTRODUCTION

In order to study features of some substances with very complicated compositions, scientists attempt to test them through a variety of means. However, people are easily disturbed in the traditional methods since these materials have extremely intricate structures. Therefore, the model compounds were adopted as the surrogates in the research areas on complex organic macromolecules. As early as the 20<sup>th</sup> century, the model compound was used in the chemical field on petroleum and lignin<sup>1,2</sup>. Similarly, as a kind of organic mineral, the coal includes a wide range of elements and components. Meanwhile, different coal has unique structure-characteristics because it received multiple effects in the coal-forming process<sup>3</sup>. As the result, some issues of coal conversion and utilization, such as coal clean, liquefaction, pyrolysis and oxidation, are gotten into complicated situations. In this paper, we review the progresses about what the consequents obtained from the studies by the coal-related model compounds (CMCs) since the eighties of the last century and also forecast the values and trends of the coal-related model compounds research in times to come.

**Pyrolysis:** The pyrolysis is a committed step in some processes during the coal conversion, for instance, the coal

gasification, liquefaction, coking and combustion. Among the mentioned operations, no one can achieve success without this important procedure. Although extensive work has been done in the coal pyrolysis area, many particular questions still remain unknown<sup>4</sup>.

Previously, Ikeda and Mackie<sup>5</sup> adopted the pyridine and 2-picoline as the model compounds for studying the thermal decomposition of coal in 1995. Under the reaction conditions of temperature between 1060 to 1240 K and pressures range from 8.0 to 13.3 KPa, the products and kinetics were obtained by the GC/MS. They find that in the reaction of pyridine, the cyanoacetylene and hydrogen cyanide are the primary and secondary products, respectively. In addition, the nitrogen atom in the condensed ring products of 2-picoline is considered as a significant role for the formation of soot in the thermal decomposition of coal<sup>5</sup>.

To supply the additional observation into the complicated changes during the pyrolysis of coal, Arenillas *et al.*<sup>6</sup> proposed the model compounds (Fig. 1) and synthetic coal as objects of study.

This method successfully avoids the probable interactions between different surface groups and cross-links occurring in the pyrolysis process. The measurement *via* the joint applications of FTIR/TG/MS helps to explain the variation of

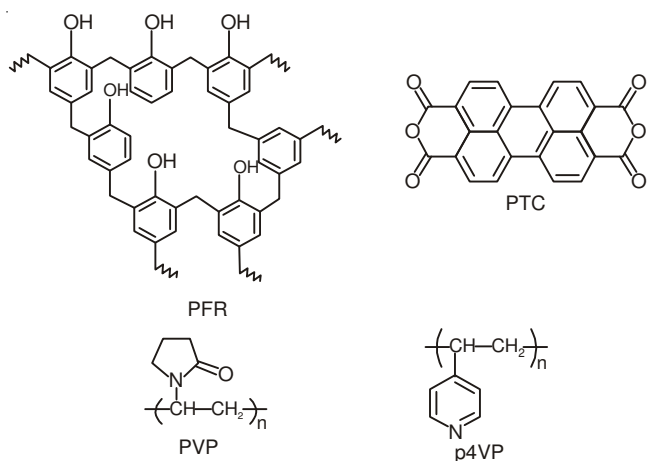


Fig. 1. Model compounds used for simulating the coal pyrolysis

composition and structure in the model compound and synthetic coal, with a correspondence between the absorption and functional groups. Based on the DTG curve of PTC (500-650 °C), the emission of CO and CO<sub>2</sub> is stem from the breaking of anhydride groups. By comparison, even the temperature only reaches 300 °C, the CO, CO<sub>2</sub> and moisture which may originate from the ether, methylol or formaldehyde is still caught in the thermal cracking of PFR. During reaction processes of p4VP, the six-member ring does not crack between the 350-500 °C. However, the H<sub>2</sub> and H<sub>2</sub>O are detected, which demonstrate that the transformation from the polymer into monomers may be the major activity. In contrast, the rupture of five-member ring in the PVP not just contributes to the generation of CO and CO<sub>2</sub>, is also responsible for the producing of HCN and NO.

Analogously, in 2008, the pyridine and pyrrole were employed as the model compounds for researching their

emission characteristics in the thermal decomposition process by Wang *et al.*<sup>7</sup> The pyridine and pyrrole were mixed and then pyrolyzed under 873-1473 K. Their products were tested by the gas chromatography and infrared gas analyzer. The results suggest that the thermal stability of the pyridine is better than that of the pyrrole. Hydrogen cyanide, hydrogen and even small amounts of methane were detected among the pyrolysis products of model compounds.

As another important aspect, the pyrolysis paths of coal also cannot be ignored. Through analyzing the thermodynamics and kinetics of the model compounds (anisole) in the isomerization reaction of pyrolysis by the density functional theory of quantum chemistry, Zhao *et al.*<sup>8</sup> state that there are two parallel reaction paths in the generation of a critical intermediate *i.e.*, cyclohexadienones. And the homolysis of -O-CH<sub>3</sub>- in the anisole is rate determining and its activation energy is 285.90 kJ/mol (1000 K). Additionally, ten transition states, seven intermediate structure and three possible reaction pathways in the pyrolysis isomerization of anisole are revealed in the Fig. 2.

Jia *et al.*<sup>9</sup> verify that the C-C bond at the side chain of N-butyl is most likely to break by the analyzing the initial pyrolysis of coal model-compound (butylantraquinone). Using the calculation of DFT / B3LYP from the Gaussian-03 program, the thermodynamic parameters of the reaction path and energy difference of the frontier orbital under different reaction temperature were calculated. Among three isomers of butylantraquinone, the  $\gamma$  side chain has the highest reactivity. The size of the aromatic ring rarely affects the C-C bond in alkyl side chains.

**Liquefaction:** The liquefaction is a kind of promising coal-conversion technology, which provides a variety of derivative products for chemical industry<sup>10</sup>. Therefore, we should elaborately probe into the liquefaction process as much as

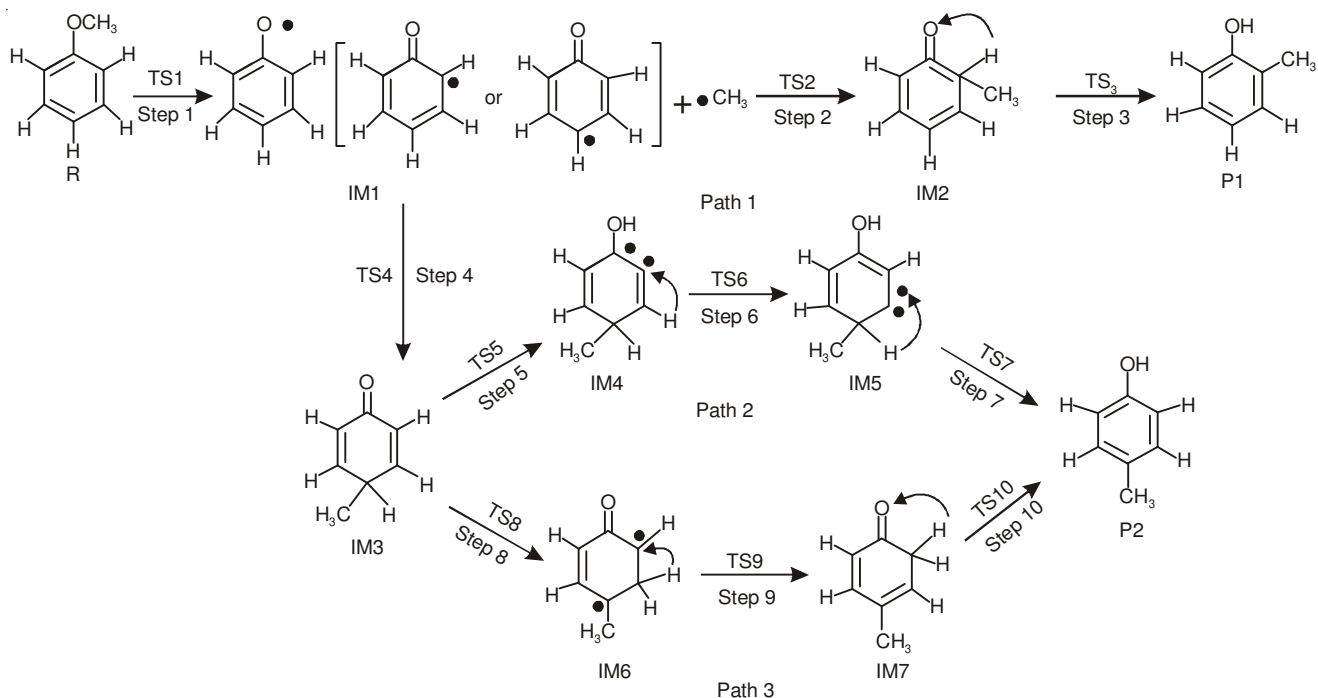


Fig. 2. Pyrolysis paths of the anisole

possible, *albeit* with plenty of relative conclusions at present. As early as 1980s, the dibenzyl, dibenzyl ether, benzyl phenyl sulfide, acetophenone and benzaldehyde were employed as the substitute in researching of the hydrogen transfer reactions. Through testing the hydrogen donating capabilities and the catalytic effect of SRC residue of these coal-related model compounds, it is found that the depolymerization of hetero-functional linkages is happen at first in the bonds cracking of coal liquefaction. And then the alkyl linkages are broken. The increase of hydrogen pressure promotes the cracking of bonds notably, which favourable for the initial stage of coal liquefaction. Furthermore, the type of donor solvent and catalytic solids influence these reactions distinctly<sup>11</sup>.

Yang *et al.*<sup>12</sup> chose the diphenyl methane, diphenyl oxide, diphenyl ether, phenyl benzyl ether and dibenzyl ether as model compounds on study of coal liquefaction. These model compounds were put into the tetralin solvent with 5-6MPa N<sub>2</sub> and their pyrolysis kinetics and reaction mechanism was studied. In this study, the diphenyl-methane and diphenyl ether are remain relatively stable under 435 °C/2 h conditions, which suggest that the breaking of C-C and C-O is more complex in the actual process of coal liquefaction. More importantly, the toluene generated by further deoxidized of benzaldehyde may be a very important way in the coal liquefaction process.

Wei *et al.*<sup>13</sup> summarized the hydrogen transfer in model compounds of coal liquefaction. Many studies have shown that the molecular hydrogen accelerated the thermolysis, hydrogenation and hydrocracking of model compounds. By contrast, the hydrogen-donating solvents are counterproductive in these reactions. It is worth noting that the sulfides catalyze radical hydrogen transfer to model compounds, whereas other metallic catalysts (*e.g.* Fe, Pd and Ni) do not. Meanwhile, the hydrocracking of model compounds is also closely related to the adsorption strength on the catalyst surface. Chang *et al.*<sup>14</sup> also measured the thermodynamic properties of some coal-related model compounds of coal-liquids (bicyclohexyl, quinoline tetralin, *etc.*). After the calculation and comparison of the parameters like the vapor pressure, molar volume, excess volume, excess enthalpy and vapor-liquid equilibrium, they believe that the excess volume is a representative data which clearly reflecting the binary interaction of these coal-related model compounds<sup>14</sup>. Sugano *et al.*<sup>15</sup> adopted the coal-related model compounds related the coal liquid for investigating the hydrogenolysis reactions of coal-liquefaction residue (CLR) *via* red mud and sulfur catalysts. They insist that the compounds with the skeleton structure are redound to the upgrading of coal-liquefaction residue.

Recently, the SO<sub>4</sub><sup>2-</sup>/ZrO<sub>2</sub> solid acid is used as the novel catalyst for the hydrocracked in coal liquefaction. To directly address the catalytic mechanism and behavior of SO<sub>4</sub><sup>2-</sup>/ZrO<sub>2</sub> solid acid in liquefaction process, Wang *et al.*<sup>16,17</sup> apply the Shenghua coal and some coal-related model compounds (involved the diphenylmethane, dibenzyl and phenyl ethyl ether) as the experimental samples to implement hydro-liquefaction. The outcome suggests that conversion efficiency is closely link with the factors, for instance, the strength, amount and nature of acid sites on the catalyst surface. Besides, it is notable that the intermediate of carbenium ion is the critical ingredient for the SO<sub>4</sub><sup>2-</sup>/ZrO<sub>2</sub> solid acid in the catalytic process,

instead of the radicals intermediate in conventional catalysts. Also the catalytic mechanism of SO<sub>4</sub><sup>2-</sup>/ZrO<sub>2</sub> in the hydro-cracking, isomerization, alkylation and hydrogenation/dehydrogenation of coal-related model compounds was proposed and discussed, combination with the kinetics parameters and products during the reactions.

**Clean:** As the main associated-elements, the sulfur and nitrogen in the coal are commonly existed. The distribution of organic sulfur and nitrogen in coal are closely affected by the chemical structure of coal. It is significant to research their emission disciplines in the process of coal utilization due to the fact that products of N and S polluted the environment after the pyrolysis or combustion of coal. Thus, seeking effective desulfurization and denitrification methods is an important challenge in fields of coal clean. It is well accepted that the deprivation of N or S in coal is a complex chemical processing, which affecting by many factors<sup>18</sup>. Undoubtedly, taking advantage of the coal-related model compounds can help people recognize the migration of N or S more accurately.

In order to create a coal-like condition, five kinds of S-containing coal-related model compounds were infused into a charcoal and carried out pyrolysis under a nitrogen atmosphere. According to the analysis of some gas products (such as H<sub>2</sub>S, COS, SO<sub>2</sub>, CS<sub>2</sub> and hydrocarbons, *etc.*), it can be learned that the sulfur radical, a kind of active intermediate, plays a significant role during these reactions. And the S-compounds are the origin of sulfur-containing gases<sup>19</sup>.

Apart from the laboratory testing, simulation is another salutary method. Using the naphthyl benzyl sulfide as the model compound, its pyrolytic process was calculated by the density functional theory in order to study the thermal stability of thioether bond in the coal. The reaction paths are shown in Fig. 3.

The analysis of bond lengths and bond orders shows that the weak bond between the C and S in the benzyl methylene is first break in the pyrolysis. Besides, the micro-structural parameters and thermodynamic functions demonstrate that the stability of the chemical bonds can be sequenced as follows: Path 1 < Path 3 < Path7 < Path 2 < Path 5 < Pat h 4 ≈ Path 6. Meantime, the elevated temperature is conducive to the thermal cracking reaction of naphthyl benzyl sulfide<sup>20</sup>. According to the theory of molecular structure and thermodynamics of coal, Yi *et al.*<sup>21</sup> calculated the theoretical decomposition-voltage for electrolytic desulfurization of the model compound contained organic sulfur under the standard state. By means of the analysis on the possibility of electrochemical removal of organic sulfur in the coal, they assert that the non-aromatic S is prone to be removal by the electrolytic oxidation, whereas the organic sulfur in the form of thiophene ring is rather steadier, especially with the increase of fused ring. Thereby, suitable catalyst and modified electrode are eximious choices for improving the degradation and deprivation of the organic sulfur in coal.

In terms of N-containing coal-related model compounds, in order to investigate the N release in the entrained-flow gasification of coal more detail, the coal-related model compound pyridine was mixed with the diesel oil for simulating the N in coal molecular. In the light of the axial and radial concentration distribution of gas products (HCN, NH<sub>3</sub>, NO

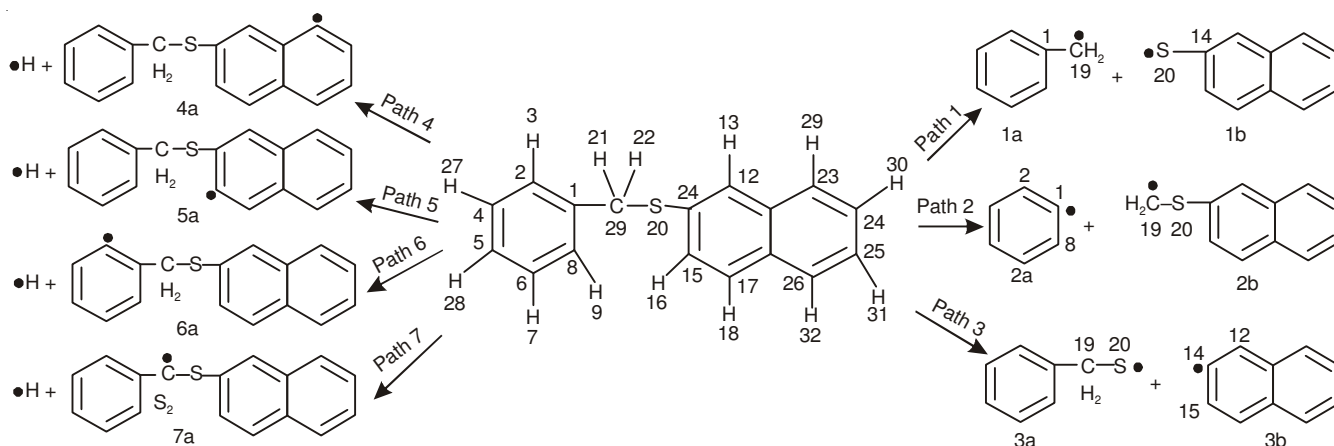


Fig. 3. Thermal decomposition paths of the naphthyl benzyl sulfide

and  $N_2$ ) which record by an opposed multi-burner (OMB) gasifier, the data show that the order of emission concentration is as follows:  $N_2 > HCN > NH_3 > NO$ . Besides, it is noteworthy that the  $O_2$ /fuel ratio exerts a staple influence in the generation of N-products. The NO and  $N_2$  are growing with this ratio increase, while the HCN and  $NH_3$  will achieve the maximum point at the ratio of 1.3. No matter extreme high (1.7) or low (0.9) value may impede the emission of HCN and  $NH_3$ <sup>22</sup>. Moreover, the pyridine blended with diesel were placed in the entrained flow gasification for the purpose of surveying the  $NH_3$  generation rate from the outlet of gasification chamber under different C/O ratio. There is a maximum generation-rate of  $NH_3$  when the C/O ratio within range of 1105 to 1197  $m^3/kg$ . Obviously, excessively high or low C/O ratio will hinder the producing of  $NH_3$ . At the same time, the lower  $NH_3$  generation-rate will appear when the N content is too high<sup>23</sup>.

Zhao *et al.*<sup>24</sup> put the carbazole in coking conditions with 650 °C and 12MPa and selected its reaction product as the nitrogen-containing compound-model. The N emission of this model compound under the pyrolysis and combustion processes in a fixed bed reactor was researched. Na, Ca and Fe were used as the catalyst in this process. The outcome shows that the nitrogen mainly remains in the semi-coke when the pyrolysis temperature is less than 900 °C. Sodium promotes carbocoal nitrogen to convert to volatile nitrogen. By comparison, Ca and Fe have little effect. Under the high combustion-temperature, the catalyst is propitious to inhibit the release of NO.

Another study discussed the NO emission during coal gasification by N-containing model compounds (carbazole char) too. Through the reaction in a quartz tube fixed bed reactor with different concentration of  $O_2$ , they propose that the  $O_2$  concentration is a critical factor affecting the NO emission in gasification. Moderate  $O_2$  concentration will dramatically decline the NO emission, whereas excessively high or low  $O_2$  concentration boosts the NO emission. The additives of iron catalysts [ $Fe(CH_3COO)_2$ ,  $FeCl_2$  and  $FeCl_3$ ] promote the NO emission. In contrast, the calcium compounds [ $Ca(CH_3COO)_2$  and  $CaCl_2$ ] have different effects, which mainly depends upon the variation of the reaction conditions<sup>25</sup>.

**Oxidation:** It is well known that the low-temperature oxidation lead to the spontaneous combustion of coal, which

not only causes huge losses in the process of coal mining and transportation, but also damages the coal quality and reduces its utilization value<sup>26</sup>. The conventional approaches always rely on the measurement of thermodynamic parameters to reflect and explain the reaction mechanism during the low-temperature of coal. Even the process of spontaneous combustion of coal was well-known and some factors such as moisture, particle size, mineral content were considered as critical elements influencing the low-temperature oxidation of coal<sup>27,28</sup> and some possible changes in the functional group of coal are speculated. However, the detail mechanism for self-heating of coal was still unknown until now. And the molecular-level observations should be conducted in order to understand this process more accurately. Therefore, the coal-related model compounds are emerged as the subject for replacing the complex molecular structure of coal.

Some studies suggest that the content of some active groups like -COOH and -C=O will increase after the low-temperature oxidation<sup>29</sup>. Similarly, the coal-related model compounds of polymeric were oxidized under air atmosphere from 30 to 500 °C by Cetinkaya *et al.*<sup>30</sup>. Combined with the results from the test of FTIR spectroscopy and scanning calorimetry system (DSC), it was clear that the carbonyl groups turned to destroy at the point of 208 °C.

As we know, mechanism study is the basis of the prevention and control technology on self-ignite of coal. Based on the quantum chemical calculations, investigation showed that the oxygen molecule will adsorb to the active structures of coal at the initial stage of self-heating of coal (Fig. 4). During this process, new chemical bonds were generated and the adsorption energy was increased with the number of oxygen molecules involved in these reactions. The heat evolved by the adsorption at the surface side-chains of coal is more than the heat released from the benzene adsorption, which means that the contribution of the side chain adsorption is greater than that of the benzene ring<sup>31</sup>. Further studies also illustrated that the oxygen molecules adsorbed on the surface of coal belong to the multilayer adsorption. Analogically, the atom transfer and energy change of bonds when the P-containing and S-containing side groups adsorbed oxygen were estimated<sup>32,33</sup>.

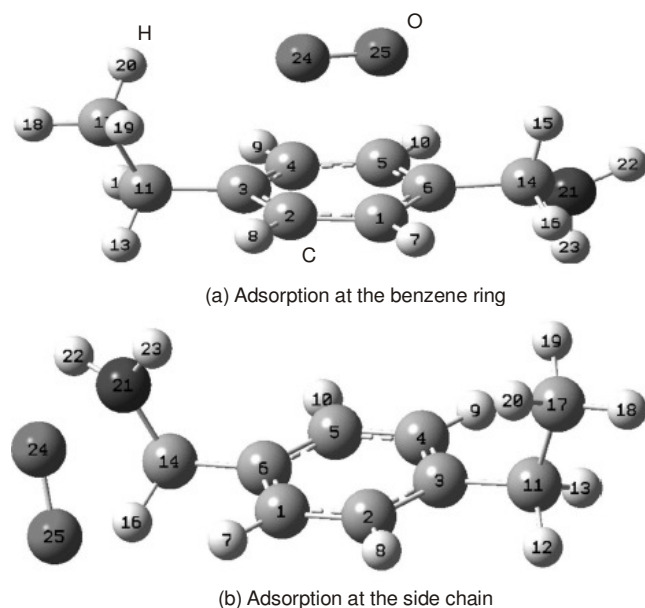


Fig. 4. Equilibrium geometries of the single oxygen molecule adsorbed at coal

In order to verify the theoretical calculations, seven kinds of model compounds (anisole, benzyl alcohol, diphenyl methanol, diphenyl methane, 1-phenylpropanol, phenetidine, phenylacetaldehyde) were used in the low-temperature-oxidation experiments of reactive functional groups in coal. Combined with the analysis of the oxidation products, changes in infrared spectra and data from the GC/MS, their oxidation path and intensity were studied. The results show that the oxidation of the active structure in coal-related model

compounds is a kind of multistep reaction, which includes the chemical adsorption, initial reaction and further decomposition. Similar with the low-temperature oxidation of coal, in these model compounds, the active groups (methyl, hydroxyl and carbonyl groups *etc.*) at the side chain or bridge bond will be gradually oxidized and then generate CO and CO<sub>2</sub>. For example, intermediates like carbon monoxide, carbon dioxide, phenols, benzaldehyde, diphenylmethanone, phenyl benzoate, 2-benzyl phenols and 2-phenolic diphenylmethanone *etc.*, were detected in products of diphenyl methane during the low-temperature oxidation process. The brief reaction paths can be seen in the Fig. 5. In addition, some phosphides also be believed as the inhibitors at the oxidizing of surface-functional-groups in the coal *via* the data before and after the loading of P-containing additives. These studies help people to understand the complicated processes during the spontaneous combustion of coal<sup>34-38</sup>.

**Summary:** Over the last decades, a plethora of achievements on coal chemistry was acquired by using the coal-related model compounds. The detail information on a series of coal chemical processes was revealed. Particularly, the changes in chemical bonds and reaction products of coal-related model compounds under different conditions indicated the major reaction path and optimal efficiency for the coal pyrolysis, liquefaction, clean and oxidation, *etc.* Aside from the mentioned aspect, some effects of additives in processes of coal conversion were shown clearly with the employment of model compounds. There is no doubt that the research methods with coal-related model compounds are highly suit for the development of coal conversion and utilization. The exploration of coal-related model compounds will move forward with the refinement and precision on coal-related reactions. No unexpectedly, the coal-

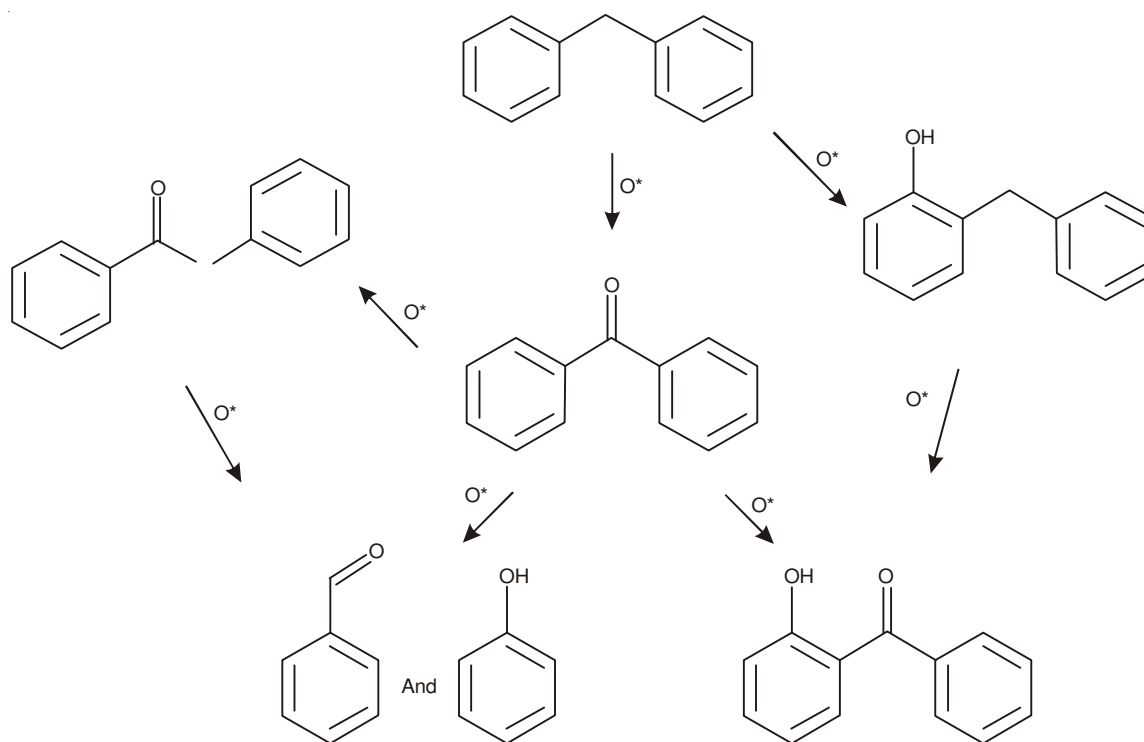


Fig. 5. Simple low-temperature-oxidation reaction pathway of diphenyl methane

related model compounds will bring about a profound impact in the further studies of coal industries.

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