

## Shape-Selective Catalysis for Synthesis of High-Value Chemicals from Aromatics in Coal Liquids

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### INTRODUCTION

Liquids derived from coals contain numerous aromatic compounds. Many of the one- to four-ring aromatic and polar compounds can be converted into valuable chemicals. Economic analysis of the viability of liquefaction (and related conversion processes) may well produce a different result if some of the aromatics and phenolics are used for making high-value chemicals and some of the liquids for making high-quality fuels such as thermally stable aviation fuels. To make effective use of aromatics in coal liquids, we are studying shape-selective catalytic conversion of multi-ring compounds. The products of such reactions are intermediates for making value-added chemicals, monomers of advanced polymer materials, or components of advanced jet fuels (Song and Schobert, 1993, 1995).

Two broad strategic approaches can be used for making chemicals and materials from coals (Schobert, 1984, 1990, 1995). The first is the indirect approach: conversion of coals to liquids, followed by transformation of compounds in the liquids into value-added products. The second is direct conversion of coals to materials and chemicals. Both approaches are being explored in this laboratory (Song and Schobert, 1993, 1996).

In this paper, we will give an account of our recent work on 1) shape-selective catalysis which demonstrates that high-value chemicals can be obtained from aromatic compounds by catalytic conversion over certain zeolites; and 2) catalytic graphitization of anthracites, which reveals that using some metal compounds promotes graphitization at lower temperatures and may lead to a more efficient process for making graphites from coals.

### POLYMER MATERIALS FROM AROMATIC MONOMERS

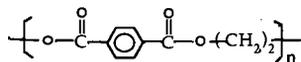
Recent years have witnessed significant growth of existing aromatic polymer materials and rapid development of advanced aromatic polymer materials such as engineering plastics, polyester fibers, polyimides, and liquid crystalline polymers (LCPs) (Beland, 1991, Song and Schobert, 1993). Consequently, there is greater demand for monomers based on aromatic and phenolic compounds. Such compounds can be made from coal-derived liquids and some refinery streams such as light cycle oils.

Scheme 1 shows the structures of some polyester materials, including thermoplastic polyethylene terephthalate (PET), polyethylene naphthalate (PEN), polybutylene terephthalate (PBT), polybutylene naphthalate (PBN), polycarbonate (PC), polyphenylene oxide (PPO), and thermotropic LCPs. PET has found widespread applications in bottles, films, and tapes. PET consumption in the United States reached 1.4 Mt in 1994, with over half being used for soft-drink or custom bottles (MP-News, 1995). Compared to PET, PEN provides five times better oxygen barrier and four times better moisture barrier, as well as 50% greater modulus and 29°C higher thermal resistance temperature. PBT is one of the major engineering plastics. PBN outperforms PBT in chemical and thermal resistance as well as tensile strength (Teijin, 1992). PEN and PBN show great commercial potential. Their markets are expected to grow rapidly. Polycarbonates have found wide-spread applications and are the second most widely used engineering plastics. PPO has an exceptionally high softening point (210°C) and is used for automotive parts, appliance, business machines, computer and electrical equipments. Of all the resins, thermoplastic polyesters grew fastest in 1994 (C&EN-News, 1993, 1994, 1995a).

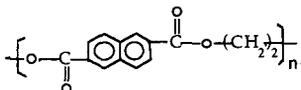
Among the LCPs, Celanese's Vectra is made from three monomers: 6-hydroxy-2-naphthoic acid, 4-hydroxybenzoic acid, and terephthalic acid. Vectra's tensile strength is about tenfold greater than regular engineering plastics such as PC. Its heat deflection temperature is also fairly high, up to 240°C. Moreover, Vectra's linear expansion is similar to that of metal. Amoco's Xydar is synthesized from *p,p'*-biphenol (or 4,4'-biphenol), *p*-hydroxybenzoic acid, and terephthalic acid (NRC, 1990). Xydar's heat deflection temperature is the highest among all the thermoplastic engineering plastics, about 350°C. Its heat-resistance is comparable to high-temperature heat-resistant polyimides. Xydar was originally developed for use in cookware; it can be used at 240°C for one hundred thousand hours (LCP-News, 1988). The global market for LCPs is about 4,500 t, about half in the Asia-Pacific market, 40% in the U.S., and 10% in Europe (MP-News, 1995). Despite their cost, LCPs are enjoying 25% annual growth worldwide and are forecast to maintain that level through the decade. Growth is 20% in the U.S. and Europe, 30% in Asia. Most LCPs are made from naphthalene-based and biphenyl-based monomers, as shown in Scheme 1. More information on these materials may be found elsewhere (Seymour, 1987; Beland, 1991; Allcock and Lampe, 1990; NRC, 1990; Song and Schobert, 1993; Relles, 1994).

**Scheme 1. Structures of Some Important Aromatic Polymer Materials**

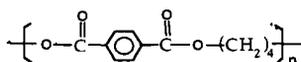
**Thermoplastic Polyesters**



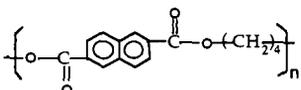
Polyethylene Terephthalate (PET) / Dacron, Mylar



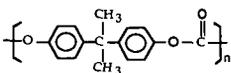
Polyethylene Naphthalate (PEN) / Teijin, Amoco



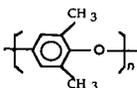
Polybutylene Terephthalate (PBT) / Valox, Celanex



Polybutylene Naphthalate (PBN) / Teijin's PBN Resin

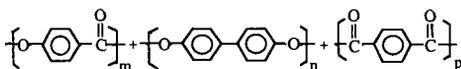


Polycarbonate (PC) / GE's Lexan, Dow's Calibre

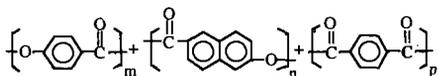


Polyphenylene Oxide (PPO) / GE's PPO, Noryl

**Thermotropic Polyester LCPs**

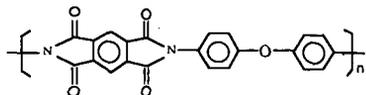


LCP / Amoco's Xydar; Sumitomo's Ekonc

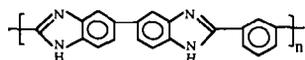


LCP / Hoechst Celanese's Vectra

**High-Temp Heat-Resistant Polymers**



Polyimide / Du Pont's Kapton



Polybenzimidazole / Celanese's Celazole

## AROMATIC AND PHENOLIC CHEMICALS FROM COAL LIQUIDS

Detailed analysis of liquids from several pilot plants of coal liquefaction has clearly shown that phenol, naphthalene, phenanthrene, pyrene, biphenyl, BTX (benzene, toluene, xylene) and their derivatives are present in relatively high concentrations in various fractions (Wright et al., 1986; Lai et al., 1992). Analysis of liquefaction and pyrolysis products in the laboratory indicates the same trends (Caroline et al., 1993; Huang et al., 1993; Song et al., 1993). Many of the one- to four-ring aromatic and polar compounds in coal-derived liquids can be converted into valuable chemicals.

**Phenolic Compounds from Coal Liquids.** Phenol can be separated from coal liquids by liquid-phase extraction. Phenol is one of the top twenty organic chemicals and is commercially produced in a multi-step process involving benzene isopropylation, oxidation of isopropylbenzene, and separation of phenol. Phenol can be used as-is for making phenol resins or converted to monomers such as bisphenol A and 2,6-xyleneol for making aromatic polymers and engineering plastics. Phenol can also be converted to  $\epsilon$ -caprolactam, which ranks third in phenol utilization in USA and Western Europe (Weissermel and Arpe, 1993). Production of synthetic phenol has increased significantly in the past decade in USA, from 1311 to 1838 kt during 1984-1994 (C&EN-News, 1995a). The amounts of phenol produced from coal tar and waste water in USA were 20 and 21 kt in 1972 and 1989, respectively, and they are much smaller compared to synthetic phenol. Synthetic phenol production in Japan is also increasing rapidly, from 308 to 670 kt during 1987-94 (Weissermel and Arpe, 1993; C&EN-News, 1995b). Western Europe produced 1488 kt in 1991, with 98.1% synthetic phenol and 1.9% phenol from coal tar and waste water (Weissermel and Arpe, 1993). It appears that the market for phenol is large enough for considering the phenolic compounds from coal liquefaction.

Naphtha fractions of coal liquefaction products generally contain 1.5-3.5 wt% oxygen, due to the presence of mainly phenolic compounds (Yoshida et al., 1991). Phenols are more abundant in the oils from coal hydrogenation using dispersed Mo catalyst and water (Saini and Song, 1994; Song and Saini, 1995). Separation of phenolics from coal-derived oils can not only produce useful chemicals, but also eliminate the need for down-stream hydrodeoxygenation which consumes the costly hydrogen to produce useless water byproduct. Therefore, such an operation also contributes to improving the economics of coal liquefaction. According to the recent reports by Yoshida and co-workers at Hokkaido National Industrial Research Institute in Japan and by Kodera and Ukegawa of National Institute for Resources and Environment in Japan, phenolic compounds (mainly phenol and cresols) can be obtained from naphtha distillates of coal liquefaction products by surfactant-mediated (Yoshida et al., 1991) or methanol-mediated (Kodera et al., 1990, 1993) aqueous extraction.

**Catechol from Coal Liquids.** Catechol (1,2-dihydroxybenzene) has some industrial applications such as therapeutic agent, tanning agent, anticorrosion agent, anti-UV agent, antioxidant for rubber, polyolefins and polyurethanes, intermediate for synthesis of agrochemicals, and intermediate for perfumes, cosmetics, and aromas (Phone-Poulenc, 1991). A new synthetic process developed in Italy produces 10,000 t/y of catechol and hydroquinone by hydroxylation of phenol with  $H_2O_2$  using titanium silicate catalyst (Notari, 1991). On the other hand, catechol is one of the major products detected in flash pyrolysis-GC-MS of subbituminous coals and lignites (Song et al., 1992; 1993; Saini et al., 1992). It may be possible to obtain catechol-rich liquids from low-rank coals through some new processing methods.

**Aromatic Compounds from Coal Liquids.** The use of aromatics in coal liquids for making value-added chemicals requires the starting material to be reasonably pure. This requirement adds a challenge for chemists and engineers, since coal liquids contain dozens or even hundreds of components. Dealkylation or dehydrogenation of oils from coal can significantly simplify the composition, leading to simple separation of individual components by distillation.

Nomura and co-workers at Osaka University in Japan have reported making aromatic chemicals from coal through catalytic dehydrogenation and dealkylation of liquefaction products (Ida et al., 1991; Nomura et al., 1995). In particular, dehydrogenation can simplify the liquid composition, because two or more isomers (e.g., 2- and 6-methyltetralin) become one compound (e.g., 2-methylnaphthalene) upon dehydrogenation. In the presence of a carbon-supported palladium catalyst, dehydrogenation of heavy naphtha (bp ca. 200-220°C) and light oil (bp ca. 220-240°C) from several coal-derived liquids is effective for producing naphthalene and methylnaphthalene, respectively (Nomura et al., 1995). Sato and co-workers at National Institute for Resources and Environment reported that non-catalytic hydrodealkylation of middle distillates (bp 180-450°C) from coal liquefaction produces mainly unsubstituted and methylsubstituted 1- to 3-ring aromatic chemicals including benzene, toluene, indene, naphthalene, methylnaphthalene, biphenyl, acenaphthylene, fluorene, and phenanthrene (Sato et al., 1992, 1994). They have also shown that catalytic cracking of the middle distillates (bp 200-260°C) from coal liquefaction produces the oils that consist mainly of alkyl naphthalenes; hydrodealkylation of such oils can produce high-purity naphthalene and methylnaphthalene as chemicals (Sato et al., 1994).

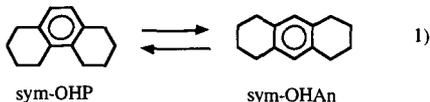
## SHAPE-SELECTIVE SYNTHESIS OF HIGH-VALUE CHEMICALS

Our attention on chemicals has focused on shape-selective catalytic synthesis of value-added chemicals from polycyclic aromatic compounds that are rich in coal liquids (and in some refinery streams such as LCO). We are studying ring-shift isomerization of phenanthrene derivatives to anthracene derivatives, shape-selective alkylation of naphthalene, conformational

isomerization of *cis*-decahydronaphthalene, shape-selective hydrogenation of naphthalene, and shape-selective isopropylation of biphenyl, as described below.

**Ring-Shift Isomerization.** Phenanthrene and its derivatives are rich in various coal-derived liquids, but their industrial use is still very limited (Kurata, 1986, Song and Schobert, 1992). On the other hand, anthracene and its derivatives have found wide industrial applications (Song and Schobert, 1993). Some catalysts selectively promote the transformation of *sym*-octahydrophenanthrene (*sym*-OHP) to *sym*-octahydroanthracene (*sym*-OHAn), which we call ring-shift isomerization (Song and Moffatt, 1993, 1994). This reaction is in distinct contrast to, and mechanistically different from, the well-known ring-contraction isomerization of tetralin (Hooper et al., 1979; Franz and Camaioni, 1980), *sym*-OHP (Cronauer et al., 1979), and *sym*-OHAn (Collin et al., 1985); ring-contraction results in methylindane-type products.

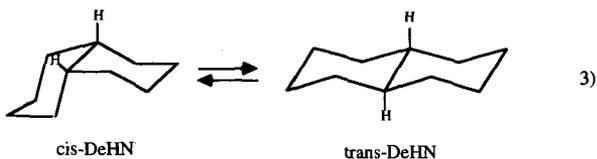
Under mild conditions, some zeolites can afford over 90% selectivity to *sym*-OHAn with high conversion of *sym*-OHP (Song and Moffatt, 1994; Lai et al., 1995). This could provide a cheap route to anthracene and its derivatives, which are valuable chemicals in demand, from phenanthrene, which is rich in liquids from coal. Possible applications of *sym*-OHAn include the manufacturing of anthracene (for dyestuffs), anthraquinone (pulping agent), and pyromellitic dianhydride (the monomer for polyimides such as Du Pont's Kapton) (Song and Schobert, 1993).



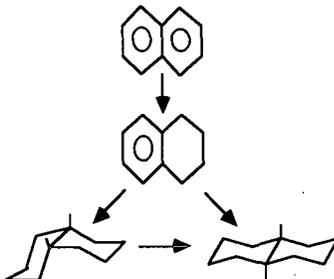
**Shape-Selective Alkylation of Naphthalene.** Naphthalene and its derivatives are rich in oils from bituminous coals. Shape-selective alkylation over some molecular sieve catalysts can produce 2,6-dialkyl substituted naphthalene (2,6-DAN). 2,6-DAN is needed now as the feedstock for monomers for making advanced polyester materials such as polyethylene naphthalate (PEN, Scheme 1), polybutylene naphthalate (PBN), and liquid crystalline polymers (LCP). By using some shape-selective catalysts, regioselective alkylation of naphthalene can be achieved with over 65% selectivity to 2,6-DAN by using isopropanol (Song and Kirby, 1993, 1994) or propylene as the alkylating agent (Schmitz and Song, 1994, 1995). Partially dealuminated proton-form mordenite can be used as shape-selective catalyst for isopropylation of naphthalene (Katayama et al., 1991; Schmitz and Song, 1994, 1995). We also found some simple and effective methods for enhancing the shape selectivity (Schmitz and Song, 1994, 1996b).

**Shape-selective Alkylation of Biphenyl.** Biphenyl and its derivatives are present in coal-derived liquids, although at concentrations lower than those of naphthalene derivatives. Shape-selective alkylation of biphenyl can produce 4,4'-dialkyl substituted biphenyl (4,4'-DAB), the starting material for monomer of some LCP materials represented by Xydar (Scheme 1). Partially dealuminated proton-form mordenite can be used as shape-selective catalyst for isopropylation of biphenyl (Lee et al., 1989; Sugi et al., 1991, 1994; Schmitz and Song, 1995). Dealumination of some commercial mordenites by acid treatment first increases then decreases their activity, but increases their selectivity toward 4,4'-DAB in isopropylation with propylene (Schmitz and Song, 1995). More recently, we have found a simple method using an additive to inhibit deactivation of the partially dealuminated mordenite catalysts without losing activity and selectivity (Schmitz and Song, 1996c).

**Conformational Isomerization.** Commercial decalins obtained from naphthalene hydrogenation are almost equimolar mixtures of *cis*-decalin and *trans*-decalin. Recently we have found that *cis*-decalin isomerizes to *trans*-decalin, as shown in equation 1, at low temperatures (250°C) over some catalysts (Song and Moffatt, 1993, 1994). This reaction would require a temperature of about 450°C in the absence of a catalyst (Song et al., 1992). It is possible to achieve over 90% conversion with 95% selectivity with some zeolites at 200°C (Lai and Song, 1995). *trans*-Decalin has substantially higher thermal stability at temperatures above 400 °C (Song et al., 1992). Possible applications are high-temperature heat-transfer fluids and advanced thermally stable jet fuels, which can be used both as heat sinks and as fuels for high-Mach aircraft (Coleman et al., 1993; Song et al., 1993, 1994).



**Shape-selective Naphthalene Hydrogenation.** Complete hydrogenation of naphthalene in conventional processes produces mixtures of *cis*- and *trans*-decalin. The motivation for selective naphthalene hydrogenation comes from our accidental finding on zeolite-catalyzed isomerization of *cis*-decalin and from the need to tailor the formation of desired isomers from two-ring compounds (Song and Moffatt, 1994). In our previous studies on naphthalene hydrogenation, certain catalysts show higher selectivity towards *cis*-decalin or *trans*-decalin (Song and Grainne, 1993; Lin and Song, 1995; Schmitz et al., 1995; Reddy and Song, 1995). More recently, we found that zeolite-supported catalysts selectively promote the formation of *cis*-decalin or *trans*-decalin (Schmitz et al., 1995, 1996), as shown below.



Now we can produce *cis*-decalin, with over 80% selectivity (or over 80% *trans*-decalin) at 100% conversion by using some zeolite-supported catalysts at 200°C (Schmitz et al., 1995, 1996). *cis*-Decalin may have potential industrial application as the starting material for making sebacic acid. Sebacic acid can be used for manufacturing Nylon 6,10 and softeners (Weissermel and Arpe, 1993).

There is also an industrial need for selective production of tetralin, a hydrogen-donor solvent, from naphthalene. Partial passivation of some zeolite-supported noble metal catalysts by sulfur can make them highly selective for the production of tetralin during metal-catalyzed hydrogenation of naphthalene at low temperatures (Song and Schmitz, 1996).

#### CATALYTIC GRAPHITIZATION OF ANTHRACITES

We are also exploring the direct synthesis of high-value carbon materials—graphites—from coals. Both non-catalytic and catalytic graphitization of anthracites has been studied. The main objectives of catalytic graphitization are to further improve the quality of the graphitic product, and to reduce the temperature of graphitization. A reduction in graphitization temperature from typical present-day practice ( $\approx 2900^\circ\text{C}$ ) to, say,  $2000^\circ\text{C}$  could translate into enormous energy savings for the graphite industry. The product quality parameter of interest is the apparent crystalline height,  $L_c$ , which is the thickness of the “stack” of aromatic planes.

Four Pennsylvania anthracites are being evaluated as feedstocks. The catalysts under investigation are iron, cobalt, and nickel naphthenates, used at various loadings. Preliminary results demonstrate that using some metal compounds significantly promotes the graphitization at lower temperatures. For example, West Cameron anthracite graphitized at  $2000^\circ\text{C}$  with added nickel naphthenate shows an interlayer spacing (d-spacing) of 0.3365 nm, approaching the value of 0.3354 nm characteristic of perfectly crystalline graphite. An unanticipated finding is that, for most anthracites and graphitization reaction conditions, the nature of the metal in the naphthenate salt is relatively unimportant in terms of improved d-spacings or  $L_c$  values. Often when a family of compounds is evaluated for catalytic activity there is a progressive change in activity among the family, depending on the metallic element in the catalyst. The metal naphthenate catalyzed graphitization of anthracites is unusual in this respect. We have not yet elucidated the reasons, but it appears that metal carbides may be intermediates. The carbides decompose at the extreme temperatures of graphitization. We hypothesize that the thermal decomposition of the carbides liberates carbon in a highly reactive form, this reactive carbon then interacting with non-graphitic carbon or with heteroatoms in the anthracite to facilitate the graphitization.

Catalytic graphitization is presently being tested by standard industrial procedures for the production of molded graphite articles. Two anthracites, one which graphitizes well without added catalyst and a second which shows significant benefit of catalyst, are being tested. Control experiments with sponge coke are being run in parallel. Nickel naphthenate is the catalyst; experiments without added catalyst will also be run. The anthracites and the petroleum coke were mixed with coal tar pitch and isostatically molded into test billets, using standard industrial mixing and molding methods. At the time of writing, the molded billets are in the baking cycle in an industrial car bottom furnace. Upon completion of baking, half the samples will be graphitized in an induction furnace at a standard industrial temperature ( $2850\text{--}2900^\circ$ ) and the other half will be graphitized at  $\approx 2500^\circ\text{C}$ . The full range of industrial characterization tests, such as density, electrical resistivity, and coefficient of thermal expansion, will be run on all graphitized samples, and the results will be compared with standard commercial products.

## CONCLUDING REMARKS

Shape-selective catalysis is an effective approach for making high-value chemicals from aromatic components in coal liquids and some refinery streams such as light cycle oils (LCO). Catalytic graphitization is promising for producing graphites from anthracites at substantially lower temperatures compared to current practice. Expansion of the non-fuel uses of hydrocarbon resources, particularly coals, is desirable, because coal will also become more important as source of both energy and chemical feedstocks in the next century. From the viewpoints of the resource conservation and effective utilization, many of the components in coals as well as in petroleum should be converted to, or used as value-added chemicals, polymers, and carbon materials.

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