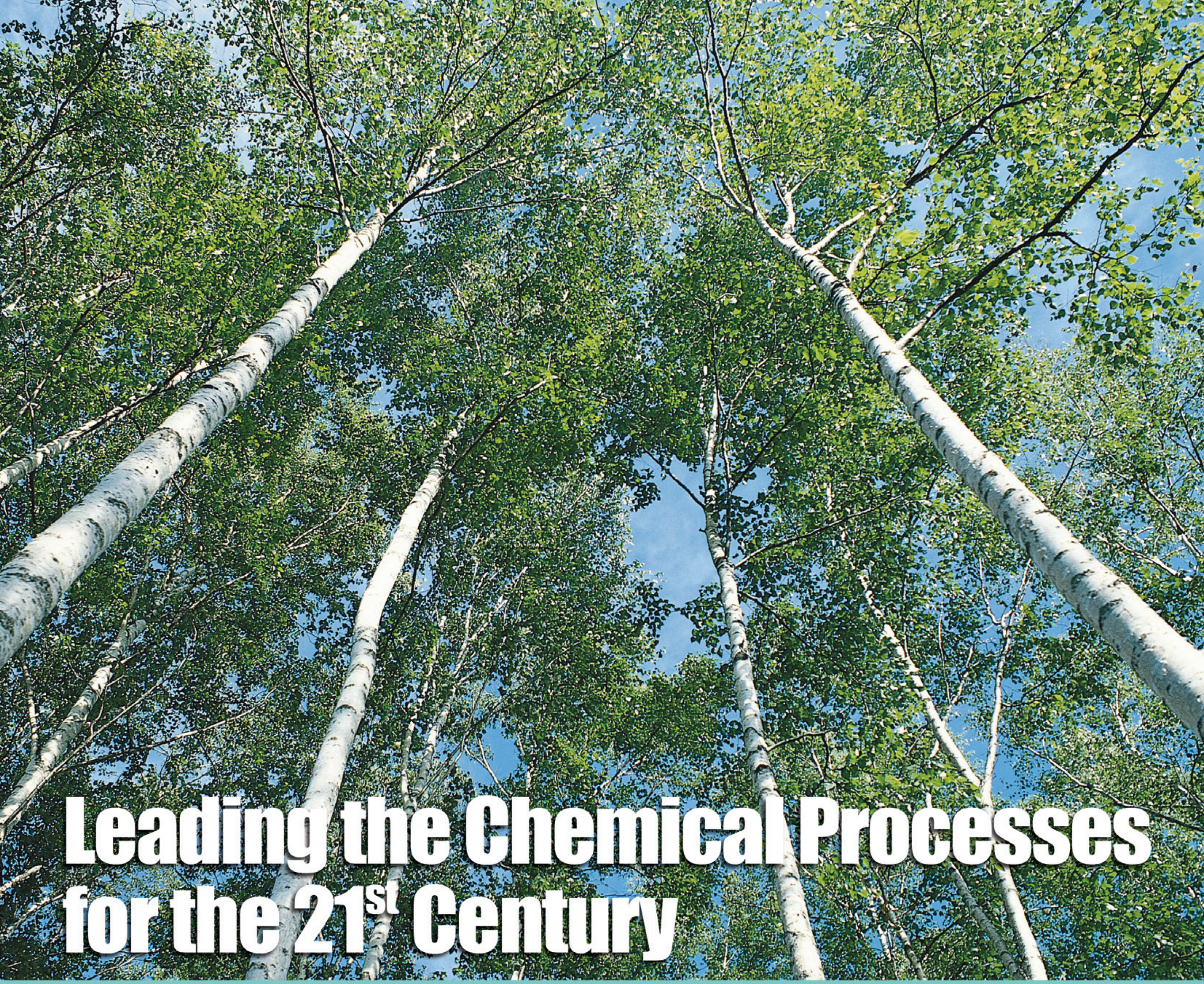
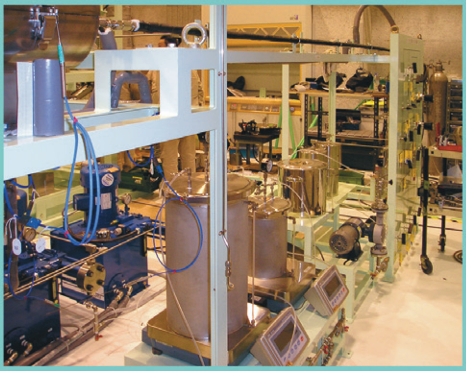


Leading the Chemical Processes for the 21st Century

New Material Circulation Systems for the Future



Leading the Chemical Processes for the 21st Century

New Material Circulation Systems for the Future

Pursuit of Ultimate Efficiency and Compactness for Future Chemical Plants
: Challenges at the Tohoku Center

Masaaki Yamabe
Research Coordinator/Energy & Environment

Chemistry, a key contributor to the prosperous material civilization of the 20th century, will continue to play a crucial role even in the 21st century to provide new materials, devices and processes for a wide variety of promising areas including energy, environment, life sciences, IT and electronics, and nanotechnology.

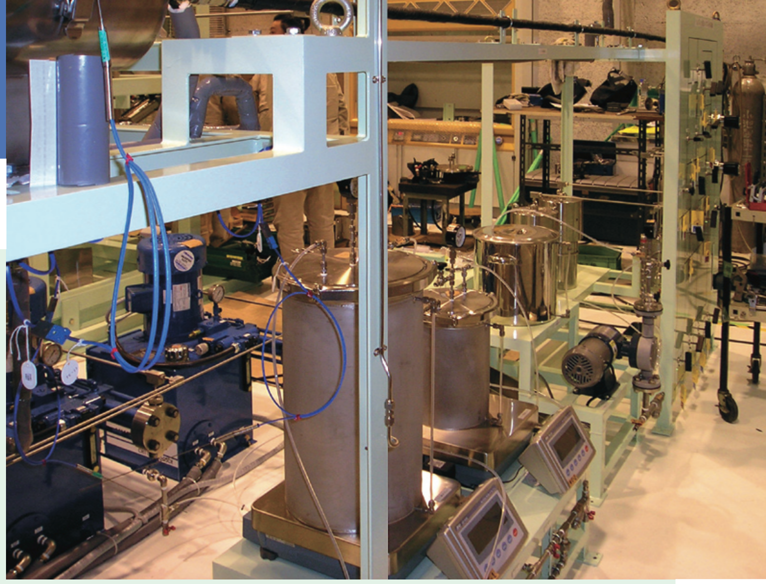
To realize a “sustainable society” is global desire to pursue in the present century. With this standpoint of global environment, “Green Sustainable Chemistry” (GSC) has been proposed to review, revise and reorganize chemical reactions and processes.

AIST has developed its "Second-Phase Research Strategy" in which development of new recyclable materials, and energy-efficient chemical reaction, separation and purification processes is committed as the "Strategic Goal No.2" in the energy and environment field (see AIST's official Web site at http://www.aist.go.jp/aist_ j/information/strategy2_full.pdf).

Through these efforts, AIST has been challenging to create innovative chemical processes and to establish a new material circulation system. The recycle-oriented system is hardly compatible with the currently predominant mass production system with its inherent difficulty in recycling and reuse of products. It requires a radical transformation of the social and economic systems into a decentralized system in which products are manufactured in an actually demanded quantity at locations that actually need them.Key requirements of such a system are minimized environmental load and efficient, highly flexible production processes. This means that the chemical processes as a core of the system should be characterized by compactness with high-speed production and easy control. So far, Japanese chemical industry has been taking a leadership in developing superior technologies for economical mass production system. These experiences will help the country play a leading role again in the development of the "compact chemical processes" technology as a key to the decentralized production system.

AIST Research Activities for Miniaturization of Chemical Plants

Hirokazu Kato
Director, AIST Tohoku



A continuous critical fluid supplier under construction in the OSL laboratory, AIST Tohoku

Sustainable social development based on a recycle-oriented system requires a transformation of industrial structure into a new production system that provides products in a quantity needed at locations that demand them. This would remain an unattainable goal before technically innovative and economically competitive compact chemical processes are realized, along with engineering infrastructures for their commercialization.

The Research Center for Compact Chemical Process in AIST Tohoku is focused on two globally-recognized key tools for the next-generation GSC: supercritical fluids and multifunctional (predominantly inorganic) membrane technology, as well as their synergistic combinations. They are furthermore likely to be combined with the microreactor and other technologies under development in other AIST locations. Creation of new technological and industrial areas in this way, an endeavor comparable to the industrialization of Japan in the late 19th to early 20th century, should contribute to GSC, resulting in minimized energy consumption and waste generation.

Close collaboration with the industry is a prerequisite for successful commercialization of new technologies. AIST Tohoku has established the Green Process Incubation Consortium (GIC) to introduce promising ideas generated in the Ministry of Economy, Trade and Industry and other government agencies, local authorities and universities to over 100 member companies. In addition, specialized consortia, e.g. one for the supercritical fluid technology, and collaborative research groups have also been established, which may eventually result in creating venture businesses. These activities are performed in close cooperation with AIST Collaboration Centers.

AIST's research on GSC has been conducted mainly in the Research Institute for Innovation in Sustainable Chemistry and Tohoku Center (Supercritical Fluid Research Center and Laboratory for Membrane Chemistry) in the first phase. Now the latter has been reorganized to create a new Research Center for Compact Chemical Process at the beginning of the second phase of our research program. The new Center has already started studies for the creation of "environment-friendly chemical industry based on new energy and resource technologies" as stated in our mid-term plan. What the program aims at is illustrated in a scenario shown below.

The purpose of the present issue is to present an overview of new technologies for the "compact chemical processes" under development principally in Tohoku Center, as well as related initiatives in industry-academia-government collaboration. We at AIST all believe that our endeavor contributes to the establishment of innovative material circulation systems for the 21st century.

In the year 20xx...
Big chemical plants with distillation towers and smoke stacks have disappeared.
New clean and compact plants are now in operation
in industrial parks neighboring on residential quarters
without disturbing the environment.
New technologies for exploiting new starting materials,
efficient reaction and separation processes have turned
what once was a typically energy-guzzling industry
into a model energy-saving industry.

Compact Chemical Processes Based on Inorganic Membranes

Fujio Mizukami
Director, Research Center for Compact Chemical Process

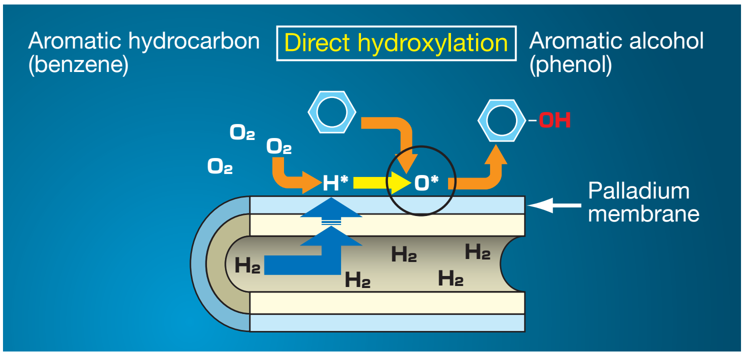


Fig.1 : Phenol synthesis by direct hydroxylation of benzene using a hydrogen-permeable membrane

Membranes as reaction process elements

The energy consumption of the chemical industry accounts for about one third of the total consumption by all the manufacturing industries. It is estimated that more than a half of the energy for the chemical industry is used for separation or concentration of substances. Separation or concentration by membranes is known to require less energy -- about 70% according to an estimation -- than distillation. Membranes may also have additional functions such as transportation, sensing or reaction. Membranes can, therefore, not only minimize the energy consumption of separation and concentration processes in chemical plants, but also have a great potential to provide a simple, cost-effective platform for complicated multistage chemical reactions, leading to reduced energy consumption and substance-related risks including that of exposure to toxic substances or explosion.

Direct synthesis of phenol from benzene

Phenol is a typical commodity chemical used in synthetic resins, plasticizers, intermediates for dyes and pharmaceuticals, and disinfectants, among others. More than 900 thousand tons of phenol is produced annually in Japan. A common industrial production process of phenol consists of three stages: reacting benzene with propylene to produce cumene; oxidizing cumene with air to produce cumene hydroperoxide; and decomposing the hydroperoxide with an acid to produce phenol, with a yield of about 5%, along with acetone as a byproduct. Since each step has a relatively low yield, and additional processes, involving distillation and extraction, are needed to separate and purify the product while recovering unreacted substances, the production process of phenol is rather complicated and energy-consuming.

We have proposed a simple process for direct synthesis of phenol from benzene using a hydrogen-permeable membrane, as shown in Fig. 1. Hydrogen inside the system diffuses across a palladium membrane to the outer surface, where it activates oxygen. The activated oxygen then binds additively to the C=C bond of benzene to produce phenol, with a yield of a little more than 10%. The palladium membrane acts as a catalyst for the reaction and also as a diaphragm to prevent detonation due to the direct contact of hydrogen with oxygen.

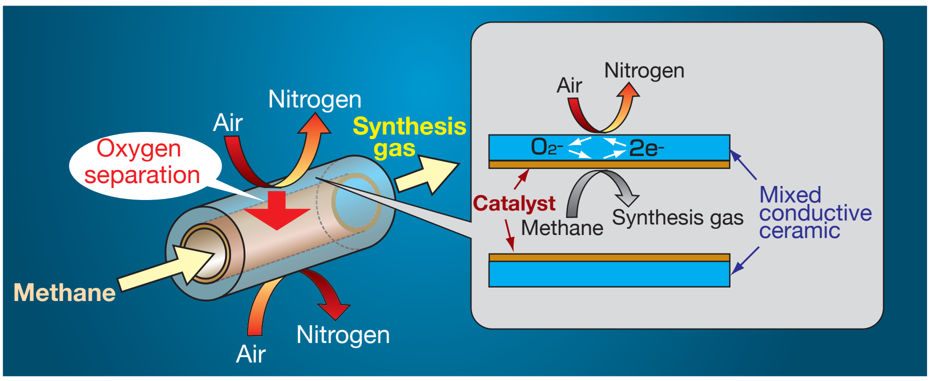


Fig. 2 : Ceramic membrane reactor for synthesis gas

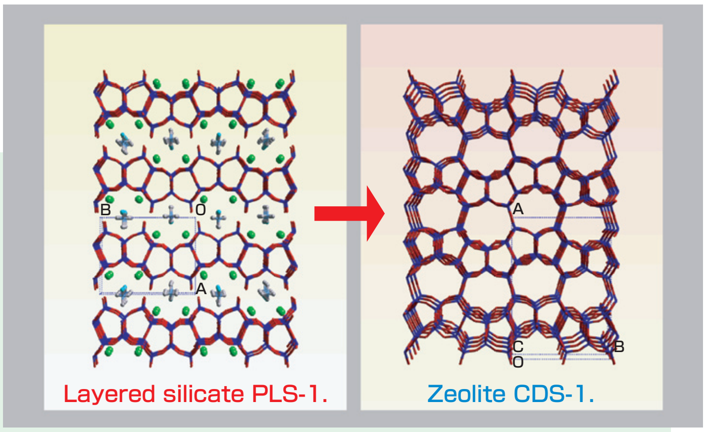
A synthesis gas production system without oxygen separation step

The partial oxidation process, in which molecular oxygen reacts directly with natural gas, is a desirable production method of synthesis gas, particularly suitable for natural gas from smaller gas fields. This process, however, requires pure oxygen instead of air as the oxygen source to keep the scale of the production plant within a reasonable limit, which means that an energy-consuming oxygen separation unit is indispensable for this process.

We have proposed a simple membrane-based synthetic gas process as shown in Fig. 2, which eliminates the oxygen separation process. The membrane consists of a mixed conductive ceramic and a catalyst for hydrocarbon reforming with a similar chemical composition. Oxygen in air brought into contact with the ceramic surface is ionized and moves to the catalyst on which the reaction takes place. Nitrogen in the air supplied is automatically excluded from the reaction process, which eliminates the oxygen purification plant. A simple and compact process is thus realized.

Industrial prospects

Selective production and separation of p-xylene is a very important process since the product serves as the starting material for synthetics including polyethylene terephthalate (PET) via terephthalic acid. AIST research groups have found that



Model of conversion of layered silicate PLS-1 to novel zeolite CDS-1

Novel Zeolite (CDS-1) Membranes from Layered Silicates

Yoshimichi Kiyozumi
Nano-porous Material Design Team,
Research Center for Compact Chemical Process

Zeolite is a general name given to a series of crystalline aluminosilicates. The unique framework structure is responsible for a number of functions of zeolites such as adsorption, ion-exchange property, solid acidity and molecular, sieving property which provide the minerals a wide range of industrial applications. An advantage of zeolites as an inorganic membrane material is resistance to heat and chemicals. However, synthetic zeolites have poor plasticity and are usually obtained only powdery form.

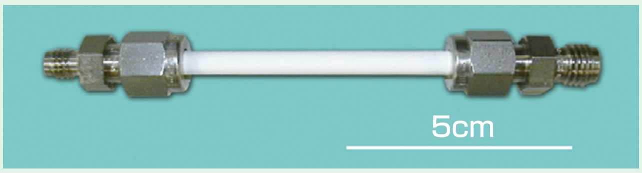
AIST has developed a novel process in which a layered silicate as a precursor is converted to a zeolite by dehydration condensation of the interlayer Si-OH groups, instead of normal hydrothermal synthesis method. Since the precursor silicates can easily synthesized at low costs, this is a promising, inexpensive method of synthesizing zeolites, even those with novel topologies inaccessible by hydrothermal synthesis.

A model shown in Figure illustrates conversion of PLS-1, a layered silicate with five-membered rings as the framework, obtained from an SiO_2-KOH-TMAOH (Tetramethylammonium)-1,4-dioxane-H_2O system, into a novel zeolite CDS-1 (Cylindrical Double Saw-edged-1). This structural transition proceeds by simply heating the precursor to 400°C or higher in vacuo or in air. CDS-1 obtained has a framework consisting only of Si and O_2, in an octagonal structure, which is responsible for relatively small pore structure, thermal stability up to 900°C, and resistance to acetic acid, hydrochloric acid and other chemicals.

The separation performance of a membrane made of the novel zeolite CDS-1 was studied. Seed crystals of the precursor PLS-1 were applied on the outer surface of a mullite or alumina porous tube, (10 mm in diameter and 80 mm in length). Secondary growth of the seed crystals gave a dense PLS-1 layer, which was then converted to a CDS-1 membrane by heating the assembly to 400°C or 600°C for 10 hours in air.

Water-ethanol separation performance of the CDS-1 membrane showed that a membrane obtained by calcined at 400°C permeates water selectively (water/ethanol separation factor = 30, permeation flux = 0.2 kg/m^2 h), while one by calcined at 600°C permeates alcohol selectivey (water/ethanol separation factor = 53, permeation flux = 0.5 kg/m^2 h). This indicates a possibility of modifying CDS-1 membrane properties via controlling the condensation conditions.

Studies are on the way on applications in gas separation processes and other processes which cannot be dealt with by other membranes, taking advantage of CDS-1's chemical resistant feature and stable structure.



CDS-1 membrane formed on a mullite tube

diffusion of a methanol-toluene mixture through a zeolite membrane produces p-xylene selectively. In a separate research using a porous membrane containing silver and strontium as a diaphragm between an air flow and a propylene flow, instead of using a palladium membrane described previously, AIST found that propylene oxide is produced at a selection rate of about 60%, with a propylene reaction rate of about 5%. Propylene oxide is currently produced in a two- or multistage reaction process.

While no inorganic membrane has yet been commercialized for chemical reaction processes, Mitsui Engineering & Shipbuilding Co., Ltd. uses zeolite membraned for removal of water from organic solvents on a commercial basis. The membrane process has realized a solvent dehydration plant 30 times as small as a distillation plant. This example clearly illustrates the great potential of inorganic membranes as a key element in simplified, compact chemical processes.

Development of Future Chemical Plants Based on Supercritical Atmosphere

Kunio Arai
Professor, Tohoku University, Graduate School of Evironmental Studies / Director, Research Center of Supercritical Fluid Technology, School of Engineering, Tohoku University

We regard the development of chemical plants based on supercritical atmosphere as a key factor for realization of sustainable society, and are engaging in energetic reseach. This article discusses a desirable route of change of chemical plants, and describes the significance of supercritical fluid for chemical plants of the future.

Failure of mass production system

Along with advances in technology, cost reduction by mass production is the most effective way to provide products to the public at low prices. Production cost is roughly proportional quantity of goods produced raised to the 0.6th power. According to this rule, cost of a ¥1,000/kg item at a certain production scale will become only ¥10/kg if the production scale is expanded by 100,000 times. This example clearly shows the large effect tthat mass production has on economics. However, mass production is only possible if a sufficiently large market exists and a sufficiently large amount of the raw materials can be supplied. Our finite resources will certainly run out someday, resulting in the collapse of our mass-production, mass-dumping economy. Moreover, our reckless use of natural resources will lead to changes in substance distribution on the Earth's surface, causing environmental problems such as global warming or chemical pollution.

Geographically distributed plants as plants in future

The Earth is a "closed system" in thermodynamic phraseology and this means that we are clearly restricted on how we should use our resources. For a process to be sustainable in a closed system, it should use the energy flow between the system and its environment -- for the Earth, flow from the incidence of sunlight to the infrared emission to space -- to restore matter transformed by the process. Photosynthesis by plant life is an example. Sustainable society in a closed system such as Earth is only possible on the basis of solar energy and with complete circulation of matter.

Fortunately, the incoming solar energy is far more than sufficient: even if ten billion people, which is an estimated upper limit of global population corresponding to the limit of food supply, were to consume as much energy as the Japanese person today, total energy consumption would account for only 0.05% of the solar energy received by the Earth. Fossil fuels could be completely replaced by 10% of the recyclable biomass that forms a part of the circulation of 95 billion tons of carbon. Solar energy, however, is highly dispersed and this can hardly be used in a concentrated form like fossil fuels. Using biomass in the same way as oil would also be extremely inefficient. Distributed energy can only be effectively utilized by distributed, autonomous and self-sufficient economic units, and such units must be supported by distributed chemical plants. This is why future chemical plants should be geographically distributed, small-scale, simple and environment-friendly.

Supercritical fluid technology in future chemical plants

Supercritical water and carbon dioxide technology are suitable for recycling oriented, and distributed production modes of operation since both water and carbon dioxide have a natural affinity for the environment. Reaction rates can be controlled through temperature and pressure adjustment conditions and high control is also possible of the solvent (see figure). These factors allow miniaturization and simplification of the reactors. If reaction time of some tens of minutes can be reduced to a few milliseconds, a reactor volume of 1 m^3 can be lown-sized to 1 cm^3. Studies on micro- and nano-scale reactors and components have started as a first step to control high-speed processes. Another prerequisite for microreactors is the numbering-up technique that is, (parallel accumulation versus conventional scale-up. This development will be greatly helped by nanofabrication and integration techniques. A workable numbering-up procedure will allow direct commercialization of laboratory experiments, eliminating conventional scale-up and pilot plant steps, thus greatly reducing time and cost for development. We expect that it is not too long before hydrogen production from biomass via a supercritical fluid process, can

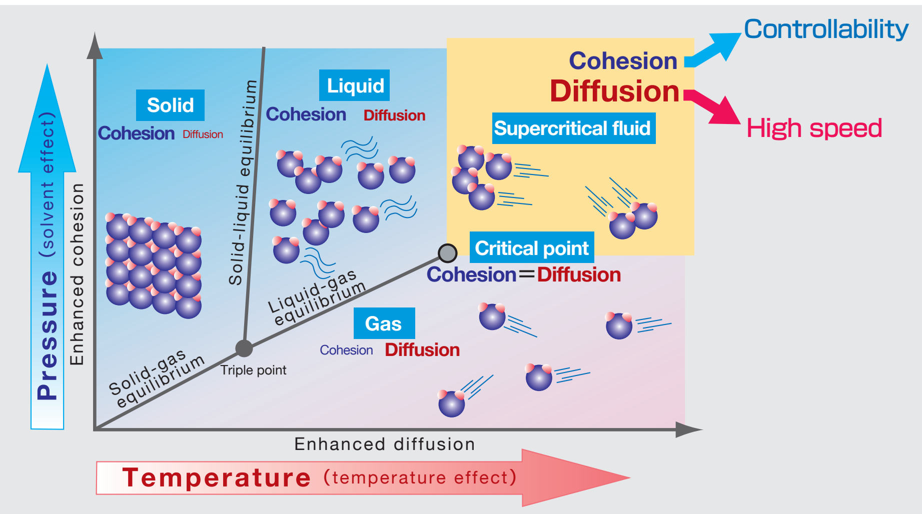


Figure : Range of existence and functions of supercritical atmospheres

be performed in apparatus that can be home appliances.

While replacement of the petrochemical industry by biomass-based chemistry and development of the supercritical fluid technology as an industrial infrastructure may need several decades, some aspects of the new engineering, e.g. precise control techniques of high-speed transportation processes in a micro- or nano-scale space, will find application immediately. Supercritical fluid technology is being commercialized and at the same time, the supercritical atmosphere can be viewed as a fundamental element of future sustainable society.

Building a Process for Maximizing the Potential of Supercritical Fluids

Akira Suzuki
Compact System Engineering Team,
Research Center for Compact Chemical Process

Supercritical water has been used as a medium to decompose organic compounds. Recently, supercritical water is attracting attention as a medium in which the Beckmann rearrangement and other organic reactions can be controlled. The yield of such reactions, however, was not of industrial interest before development of a process specifically designed for reactions in supercritical atmospheres: a continuous microreactor consisting of a single fine pressure-resistant metal tube. Unlike conventional batch processes, in which temperature changes so slowly that the reaction product is partially decomposed during the heat-up step and the cool-down step, the new continuous microreactor allows very quick recovery of the product with high yields and selectivities. Rapid heating has generally been realized by direct mixing of reactants with supercritical water. While this technique is very useful in continuous processes for small production scales, a method for rapid indirect heating is needed, as well as a method for numbering-up of reactors, for production of larger amounts of material is needed. We are now developing a general-purpose high-temperature, high-pressure microreactor and micro-scale heat exchanger that addresses these challenges. A specific target is a micro-scale reactor or heat exchanger consisting of an efficient aggregation of metal tubes and a high-efficiency heat exchange mechanism, e.g. direct application of electric current. This concept should allow reaction conditions unattainable even by the existing microreactors, high performance (rapid heating, highly responsive temperature control, resistance to heat and pressure, etc.), and miniaturized design.

Fig. 1 : High-temperature, high-pressure microreactor and heat exchanger operated at 600°C and 300 MPa

Fig. 2 : Experimental plant based on a continuous reactor using high-pressure supercritical water

Enhancing the flow and efficiency of chemical reacting species is important in achieving distributed chemical production systems that will replace energy-intensive conventional plants. Miniaturized reactor and heat exchanger systems are proving to be reliable and promising devices for chemical production with low environmental burden.

Supercritical Fluid Database
for practical applications of supercritical fluid technology

Yoshiaki Kurata
Supercritical Fluid Team, Research Center for Compact Chemical Process

A tool for promotion of supercritical fluid research

Supercritical fluids are non-condensing: its density may change in a wide range depending on the pressure. This and other exotic characteristics of supercritical fluids has long been known, but it began to attract attention as a new class of solvents only recently. Once brought to the supercritical state, the naturally abundant water or carbon dioxide acts as an innovative medium for energy-saving, safe and environment-friendly chemical processes for the future.

Japan Chemical Innovation Institute (JCII) has been leading a NEDO joint research program called “Development of Technology to Reduce the Burden on the Environment” since 2000. We attempted to make its accumulated results, ranging from fundamental to application research, open to public for widespread practical use of the technology and provide a basis for more systematic presentation of related data and methodology.

This task was achieved by the compilation of Supercritical Fluid Database (SCF-DB).

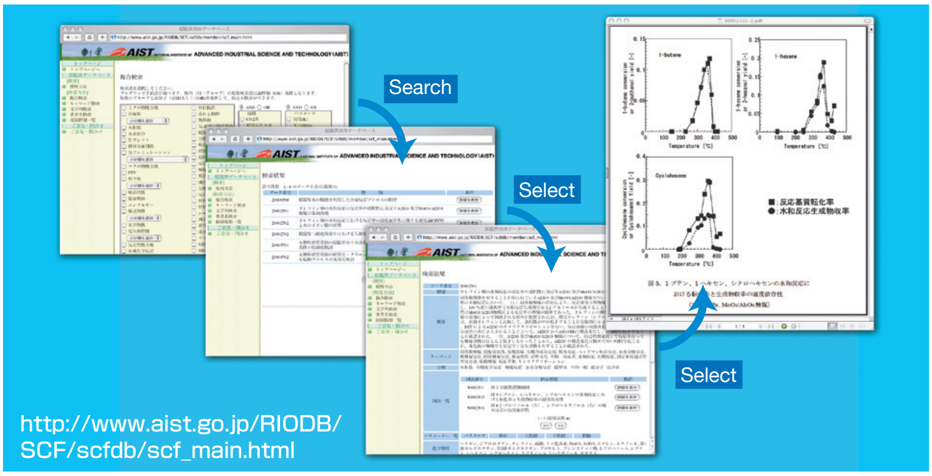


Fig. 1 : Procedure of searching the Supercritical Fluid Research Database and an example of results obtained (taken from AIST Web site, RIO-DB page)

The database is provided on the Internet as a convenient tool for R&D activities related to supercritical fluids.

Structure of SCF-DB

SCF-DB has two objectives: making research results accessible to the public and assisting practical application of the technology through systematic representation of relevant knowledge. In order to deal with research results including numerical values and graphic representations, and to integrate information needed for industrial use of the technology ranging from the basics of supercritical fluids to intellectual properties information, SCF-DB was constructed as an aggregate of six segments:

1. Supercritical Fluid Research Database, comprising abstracts, figures and citations of research reports.
2. Corrosion Database, designed for numerical search and two-dimensional representation of corrosion data.
3. High-Pressure Gas Database, a collection of safety data, accident reports, and related regulations.
4. Intellectual Property Database, providing recent information on intellectual properties related to supercritical fluids.
5. Simulations, providing examples of simulations using estimated data including those on properties of polymers.
6. Research Results of The Society of Chemical Engineers, Japan, a collection of bibliographic data compiled by the Division of Supercritical Fluids of the society.

Fig. 1 shows a procedure of searching the Supercritical Fluid Research Database to obtain graphics. In addition to research

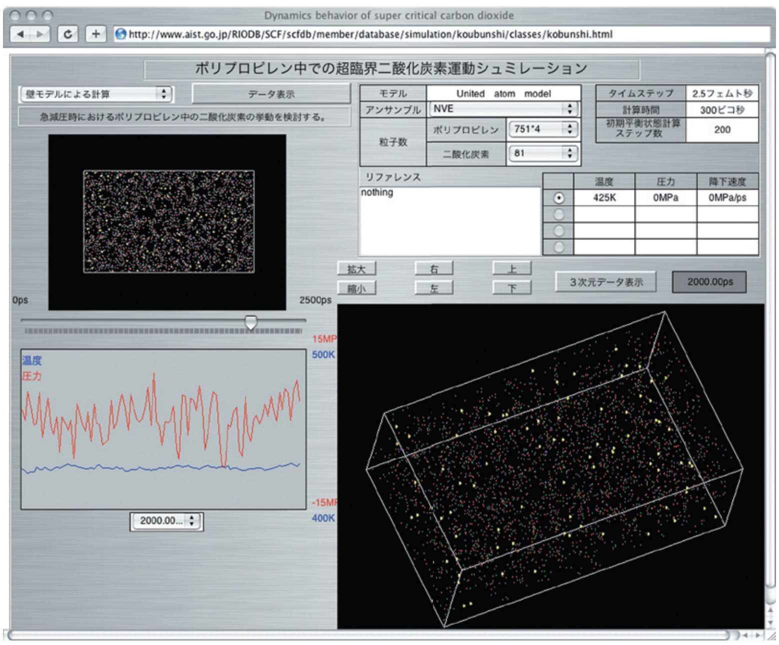
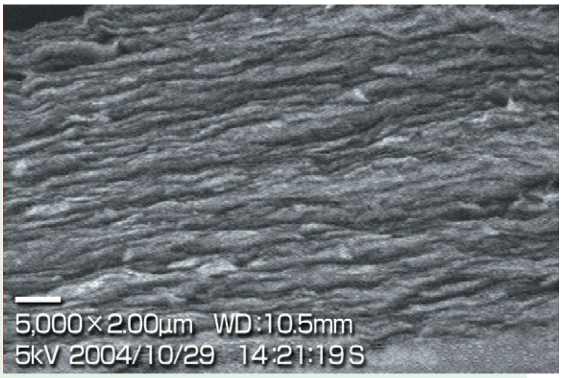


Fig. 2 : Two- and three-dimensional movie representations of simulation results

Development of "Claist", a Flexible, Heat-Resistant Inorganic Film

Takeo Ebina
Material Processing Team, Research Center for Compact Chemical Process



Electron micrograph of a section of Claist

"Claist" is a film under development in the Research Center for Compact Chemical Processes. It has both plastic-like flexiblilty, workability, and ceramic-like characteristics. The membrane is manufactured by densely laminating clay crystals about 1 nm (1 nm is one billionth of 1 m) in thickness, together with a small amount of additives, if necessary to improve flexibility and for other purposes. The total thickness of the membrane can be controlled in a range from 10 to 100 μm, maintaining flexibility comparable to that of a sheet of copying paper.

Permeability (cc/m^2/24 hr/atm) of dry Claist to inorganic gases such as helium, hydrogen, oxygen or nitrogen was under detection limit at room temperature. This is far lower than conventional engineering plastics (e.g. Nylon-6 with an oxygen permeability of 18 cc/m^2/24 h/atm) and comparable to that of aluminum foil (oxygen permeability = 0). Currently samples of standard type (usable at temperatures up to 350° C) and heat-resistant type (up to 600° C) are available from AIST innovations. Other possible modifications include reinforcement with heat-resistant fibers, lamination with heat-resistant cloth, surface treatment for higher thermal resistance, and water-proof surface treatment.

Claist is an environment-friendly product because it consists principally of clay and no byproduct is formed in its production process. Its production cost is comparable with that of general engineering plastics. Clasit, with its thermal resistance and gas barrier characteristics, is a promising material for gaskets in automotive engines, sealing of the piping in chemical plants, fuel cells and flexible circuit substrates. Developments are planned for expansion of applications by improving moisture resistance, surface flatness, and mechanical properties.

results, the database contains drawings of testing and measuring apparatuses, providing useful information for developing new research programs. The Society of Chemical Engineers data can also be utilized in the same way.

The main content of the Corrosion Database is numerical data on the corrosion behavior of metallic materials in supercritical water, collected from a variety of reports and articles published in and outside Japan. These data should be helpful for preliminary screening of materials for apparatuses.

The High-Pressure Gas Database aggregates safety data (such as ignition of materials in high-pressure oxygen), important standards in selected countries, and accident reports, thus providing useful information for safety design of experiments. The database also contains Japanese regulations and related legal procedures.

Fig. 2 shows an example of two- and three-dimensional movie representations of simulated dissolution and distribution of carbon dioxide in a polymer. The file can also be used for new analysis using retrieved data.

While data in SCF-DB are still behind the target to build a comprehensive information basis for the supercritical fluid technology, links to "SCF-related Web sites" are provided as supplements.

Supercritical Fluid Database in future

While being expected as an innovative way of chemical reactions, the supercritical fluid technology has suffered from lack of organized knowledge needed as the basis for research and development. SCF-DB aims at fill this gap by providing information that assists in appreciation of possibilities of the technology and promotion of its industrial applications in a new way.

Future plans for SCF-DB include addition of estimation formulas for properties of supercritical fluids and for behavior of polymers in supercritical atmospheres. Efforts will further be intensified to collect relevant data, material, news and related technical information.

Development of CO_2 Dry Cleaning System

Hiroshi Inomata
Research Center of Supercritical Fluid Technology, Graduate School of Engineering, Tohoku University

Current status of dry cleaning industry

Commercial cleaning service may be classified into laundry using water and dry cleaning using organic solvents. The latter has become predominant because of its adaptability to a wide variety of clothes. Statistics published by the Ministry of Health, Labour and Welfare shows that about 43,000 commercial dry cleaning machines were in operation in Japan in 2002. Consumption of chlorine-based solvents is decreasing because of its potential carcinogenicity and consequently more stringent regulations. Most of Japanese dry cleaning machines have thus adopted petroleum-based solvents. The process needs a drying step where energy corresponding to an estimated 1.23 million kiloliters of crude oil is consumed and an estimated 320 thousand kiloliters of the solvent is released to the air.

Environmental, energy-saving and resource-saving demands have urged the industry to seek alternative cleaning techniques. Particularly small and family businesses, accounting for the majority of consumer-oriented cleaning shops, have difficulties in responding to environmental problems and improving working environment.

Silicone-based solvents have been developed and commercialized as an alternative, but high prices have hindered their generalization. Another candidate solvent is liquid carbon dioxide, developed in the U.S. and Germany. A system using liquid CO_2 under a pressure of about 5 MPa (about 50 atomospheres) has been commercialized in some parts of the U.S., but it suffers from insufficient detergency and requires an auxiliary chemical detergent.

Development of the supercritical CO_2 dry cleaning in the Research Center of Supercritical Fluid Technology, Tohoku University was occasioned in an encounter at the Technology Licensing Organization of Tohoku University, which introduced the cleaning company Auto Laundry Takano Co., looking for effective solutions of the solvent problem, to the Center when it was conducting research on precision cleaning of machine parts and optical components with supercritical CO_2 as a Regional Consortium R&D Project (1997-1999). Results showed that CO_2 is a very powerful degreasing agent but high equipment costs would be a problem in commercialization. Thus the study of dry cleaning as a new application of supercritical CO_2 was launched on an industry-academia collaboration basis.

Principle of dry cleaning with supercritical CO_2

Carbon dioxide becomes supercritical at a temperature of 31° C and a pressure of 7.3 MPa, a condition mild enough to handle clothes. Supercritical CO_2 is capable of dissolving weakly or non-polar fats. Its diffusion characteristics are close to those of gases, enabling the substance to penetrate to every corner of the fabric. Even submicron-sized fine structure can be cleaned with this medium because the problem of capillary force is virtually absent. Furthermore, CO_2 is removed simply by evaporation after cleaning is finished, thus eliminating the drying process that may cause clothes shrink. Supercritical CO_2 can be thus an excellent dry cleaning solvent: in fact, experiments showed that no additional detergent is needed for satisfactory cleaning operation. Problems remained, however: how to handle the large quantity of clothes in the commercial-scale cleaning shops? How to achieve reasonable economic performance? How to prevent redeposition of dirt onto the clothes?

In order to study prevention of dirt redeposition and recovery of the solvent, an experimental supercritical dry cleaning apparatus (Fig. 1) was constructed which consists of a cleaning unit, a solvent recovery unit and a solvent recycling system. High thermal expansion coefficient of the supercritical CO_2 allows an increase of pressure by simple heating, which is used to supply the solvent at a high velocity. The low heat of evaporation of CO_2 at the subcritical region (i.e. at temperatures slightly lower than the critical point) is exploited for evaporation/condensation of the solvent for recovery and reuse. A simple closed-system dry cleaning apparatus without high-pressure pumps is thus realized. While the basics of the system had already been demonstrated, tests as a dry cleaning apparatus included minimization of cleaning time and energy consumption, optimization of the contact of the solvent with the clothes, improvement of cleaning performance by adding auxiliary detergents, and optimization/automation of operations. These studies were actually conducted by

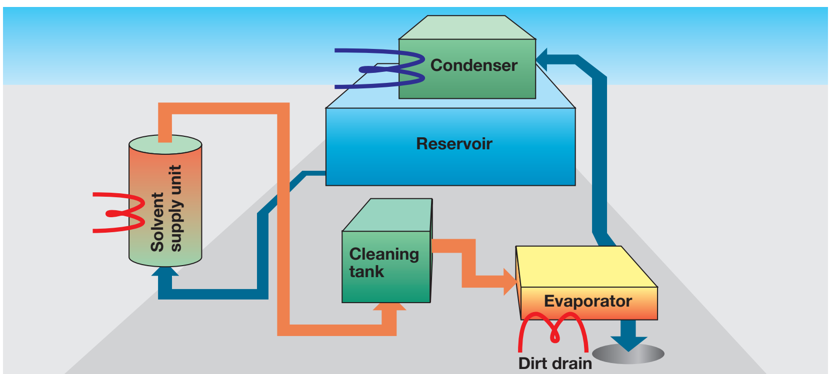


Fig. 1 : Concept of the dry cleaning system based on a supercritical solvent cycle

a consortium comprising Takano and other companies, AIST and Tohoku University. While demonstration operations were carried out by the industry members, the University performed analysis of heat flow in the solvent supplying unit (Fig. 2) and other scientific studies. AIST contributed assistance in engineering.

Results of cleaning tests with supercritical CO_2

The cycle time for a cleaning batch is about 30 to 40 minutes, which is considerably shorter than that in the current system including drying time. The system without a drying step gives less damage as well as better feeling of the fabric after cleaning. The cleaned items have, of course, no odor. The development targets have thus almost been achieved.

Commercial prospects

Following the experimental studies that have cleared basic problems, a prototype of the commercial cleaning apparatus has been completed. It is intended for monitor tests for market evaluation. The system will be improved further for a full-fledged commercial application. The new dry cleaning system is expected to spread in many countries, thus contributing to improved environment and health.

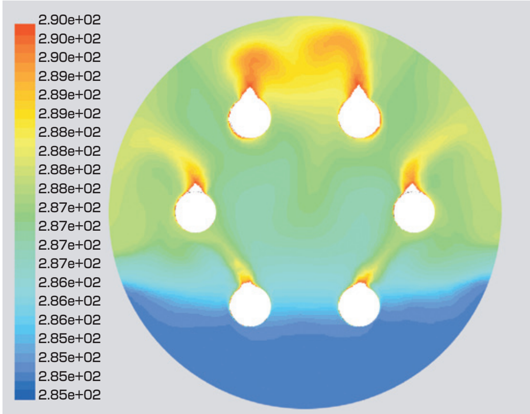


Fig. 2 Temperature distribution in the solvent supply unit immediately after a pressure of 10 MPa is reached


Fig. 1 : Experimental apparatus for supercritical CO_2 cleaning

Industry-Academia Collaboration Program for Commercialization of New Dry Cleaning System

Yoshinori Kato
Auto Laundry Takano Co.

The cleaning industry faces the problem of adverse effects of organic solvents on the natural and working environment. We started development of a novel dry cleaning process based on the patent (to Arai, Inomata and Smith, Tohoku University) on cleaning with supercritical CO_2, using a closed-system cleanig apparatus driven by heat.

Our developmental research was a part of Consortium R&D Project for Regional Revitalization (2002-04) led by Prof. Inomata, which is commissioned by the Ministry of Economy, Trade and Industry. The research was conducted on a cooperative basis between the industry, Supercritical Fluid Research Center of AIST and Professors Arai, Inomata and Smith.

Fig. 2 : Results of CO_2 cleaning of different fabrics stained with fluid paraffin and oil red dye under dual solvent supply

The research resulted in the supercritical CO_2 dry cleaning apparatus shown in Fig. 1, the world's first unit of this category. The apparatus comprises 30- and 40-liter cleaning tanks, four solvent supplying uinits, an evaporator, a condenser and a reservoir. Not only being favorable in terms of energy and environment, supercritical CO_2 permits finishing with less damage and better feeling, because the clothes need not be agitated during the cleaning operation owing to the high penetration of the medium.

Fig. 2 shows several samples of soiled fabric cleaned with supercritical CO_2 without an additional detergent. All the samples indicate performance of CO_2 comparable to or better than conventional solvents. Another merit of supercritical CO_2 cleaning is compatibility with chemical sensitivity, because no chemical substance is employed except CO_2.

Test cleaning using the experimental apparatus offered to patients throughout the country via the Internet have received favorable responses including those from serious cases.

The research project has been granted from the Ministry of Economy, Trade and Industry from 2004. Commercial service will start in 2006 for chemically sensitive people as well as high-quality cleaning of high-grade wears.

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New Material Circulation Systems for the Future

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