

Catalyst Enhanced Oxidation of VOCs and Methane in Cold-Plasma Reactors

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Volatile organic compounds, VOCs, are used in a number of industrial processes in a variety of roles, for instance as solvents, cleaners and liquid fuels. During these processes, some of the VOCs, which are known to be air toxins, inevitably escape into the surrounding air. It is the release of this VOC-laden air into the atmosphere that is partly responsible for stratospheric ozone depletion, tropospheric ozone increase, global warming (the Greenhouse Effect) and some health problems. Here, we briefly review some applications of platinum metals catalysts in the abatement of VOCs and in the conversion of methane using cold-plasma techniques. Some information on the abatement of acid gases, the generation and application of non-thermal plasmas is also presented.

Volatile organic compounds, VOCs, which are toxic by nature and which are used in industrial processes as solvents, cleaners, thinners, degreasers, monomers and liquid fuels, can be converted into water and less harmful carbon dioxide by thermal incineration at high temperatures ($> 700^{\circ}\text{C}$) (1). However, thermal incineration requires a lot of energy to heat up the ambient gas molecules. Some of this energy can be saved by incorporating a suitable catalyst into the incineration system, enabling the oxidation reaction to be performed at lower temperatures (~ 300 to 500°C) (2). Clearly, this still involves using elevated temperatures.

The use of cold plasmas can offer a promising alternative to the combustion of VOCs. By selectively channelling electrical energy into the desired plasma, chemical reactions occur at ambient temperature and pressure to break up the VOCs into smaller components (3, 4). Cold plasmas are produced by electrical discharge techniques (5–15), and have been utilised in a number of industrial applications (5–8, 16–23). In cold-plasma techniques, free electrons are accelerated under the influence of the strong electric field produced by electrical discharge.

Heavier ions and the bulk of the neutral molecules remain close to room temperature (6, 7). The high-energy electrons undergo inelastic collisions with the ambient gas molecules to produce reactive species, such as $\cdot\text{O}$, $\cdot\text{OH}$, $\cdot\text{HO}_2$, $\cdot\text{H}$, $\cdot\text{N}$, $\text{O}_2^{\cdot-}$, $\text{N}_2^{\cdot-}$, O_3 , $\text{O}_3^{\cdot-}$, O , O_2 , OH , N_2^+ , N^+ , O_2^+ and O^+ (6–8, 14, 24, 25). These free radicals, ions and excited-state molecules in turn oxidise the targeted VOCs.

Cold-plasma techniques are rapidly being developed for the destruction of toxic VOCs in air (3, 4, 26–29), soil (30) and water (31, 32), as discussed in reviews (15, 33–43). They are also being developed for the abatement of other air pollutants, such as sulfur oxides, nitrogen oxides and carbon dioxide (7–9, 34, 35, 38, 44, 45).

Cold Plasmas with Catalysts

Combining cold plasmas with catalysis is an emerging technique which can oxidise VOCs at a much faster rate, at a lower temperature and with lower input energy, when compared with cold plasma or thermal catalysis techniques alone (15, 20, 46–49).

For example, when acetone-containing air was passed at a flow rate of $400\text{ cm}^3\text{ min}^{-1}$ through

a wire-cylinder type pulsed corona discharge reactor, the acetone concentration was reduced from 40 to 30 $\mu\text{mol min}^{-1}$. When the discharge gap of this reactor was packed with alumina pellets (3–5 mm diameter), the acetone concentration was found to be further reduced from 40 to 6 $\mu\text{mol min}^{-1}$, the other operating conditions being identical (48). The operating conditions were: room temperature, atmospheric pressure, 9 W power from a DC source (as high voltage pulses of 45 kV (positive polarity), 4 μs width, 0.4 μs rising time and constant frequency) and a reactor volume of 100 cm^3 (48). There was, however, almost twice as much of the by-product ozone produced when alumina was present.

When the macroporous alumina was replaced by alumina having primarily meso- and micropores, the ozone was reduced to a negligible level, without affecting the efficiency of the VOC destruction (49). Alumina can therefore be used as the support for the active metal catalysts.

In a further example, palladium-coated spherical pellets of porous γ -alumina were shown to decompose methane in corona discharges (15, 50). The nitrogen oxides formed were significantly reduced when this palladium coated γ -alumina catalyst was employed.

By comparison with conventional catalytic reactors, excellent results have also been achieved using platinum, palladium and rhodium, for example, as catalysts in cold-plasma catalytic reactors (51), to convert Freon and methane into simpler molecules.

Electrode surfaces can also catalytically enhance the oxidation of VOCs, if a catalytically active material is incorporated into the electrode structure. When stainless steel electrodes were replaced by electrodes having catalytically active noble metal or noble metal alloy surfaces, the destruction of methane in cold plasma was improved (52).

The decomposition of air pollutants other than VOCs (sulfur oxides, nitrogen oxides and carbon dioxide) has also been improved by the addition of suitable catalysts in the cold-plasma environment (15, 53–55). As an example, sulfur dioxide could be removed with high

efficiency after a very short treatment time when palladium- and platinum-coated γ -alumina were used in a corona discharge reactor (15).

Partial Oxidation of Methane

The partial oxidation of methane (a major component of natural gas) in cold plasma yields far more valuable products than its complete oxidation (to carbon dioxide and water). C_2 products (ethane, ethene and acetylene) (56, 57), hydrogen (58), synthesis gas (59–61), methanol (62–66) and formic acid (66, 67) are just some of these products.

There are two major obstacles to overcome in methane activation:

- [1] the tetrahedrally arranged strong C-H bonds of methane do not allow space for oxidising agents to attack and
- [2] the $\cdot\text{CH}_3$ produced from methane is likely to undergo further oxidation, which may convert it to carbon dioxide and water.

Consequently, conventional thermal activation of methane requires high temperatures ($> 1000^\circ\text{C}$), gives low yields and is costly. The addition of suitable catalysts, particularly supported noble metal catalysts, such as platinum, palladium, rhodium and ruthenium, can activate methane at relatively lower temperatures, at faster rates and with better selectivity to the desired products (68, 69).

Effects of Microwave Irradiation

Microwave irradiation of suitable catalysts, such as proton conductive catalysts, oxygen ion conductive catalysts, activated carbon or charcoal, can convert methane at lower temperatures and give higher yields of the desired products than conventional heating (70–75). Under certain conditions, microwave irradiation can generate cold plasma which selectively converts methane to acetylene (72). Methane conversion in plasma takes place faster, at lower temperatures ($\sim 500^\circ\text{C}$) and needs lower input energy (about one half) than the thermally driven process (76, 77).

Indeed, combining the cold plasma with catalysis can further improve the energy efficiency as well as the selectivity of the desired products

(20, 78–81). In general, methane conversion in different reactors follows the order (80):

Plasma-catalytic > plasma > catalytic > thermal

High-energy electrons and oxidising species, such as $\cdot\text{O}$, O^{\cdot} and O_2^{\cdot} , present in the cold plasma environment, can produce $\cdot\text{CH}_3$ radicals from methane by hydrogen abstraction (56, 82, 83). The catalyst adsorbs the free radicals and their target molecules, allows the desired chemical reaction to take place on a solid surface and finally releases the products (79, 80).

When catalytic materials, such as platinum, nickel, iron, manganese dioxide and molybdenum trioxide, were placed next to the microwave plasma cavity (79, 81), platinum was found to give the highest methane conversion of 55 per cent. Product selectivities of 54 per cent for ethane, 27 per cent for ethylene, 7 per cent for C_3 hydrocarbons, and 6 per cent for formaldehyde/methanol were achieved with platinum as the catalyst.

The application of an electric field inductance (EFI), used to evolve a high density of microwave plasma to the cold-plasma-catalytic reactor, results in further increases in the methane conversion (80). The highest methane conversion to C_2 hydrocarbons, of 63.7 per cent, was achieved by employing a palladium-nickel bimetal catalyst in the cold-plasma-catalytic reactor in the presence of EFI.

Effects of Corona Discharges

A disadvantage of microwave plasma is that it usually operates at pressures below atmospheric, which makes it less suitable for large-scale applications. Cold plasmas established at ambient conditions, such as by corona discharges, have proved successful for methane conversion

(56–67). The addition of a suitable catalyst in the discharge gap of corona discharge reactors has achieved faster rates of methane conversion and better selectivity to the desired products, while requiring less input energy than when catalyst is absent (20, 84–86).

Replacing the stainless steel electrodes in the cold plasma reactor with ones containing some catalytically active metal can enhance methane conversion. Almost the same methane conversion (~ 6 per cent) and selectivity for C_2 products (~ 80 per cent) were achieved with about half the input power (35 kV, 4.6 W) by using copper instead of stainless steel electrodes (45 kV, 9 W) in a DC pulsed corona discharge reactor at a flow rate of $30 \text{ cm}^3 \text{ min}^{-1}$ for a methane:oxygen feed of 95:5 (87).

Noble metal electrodes have the best results for the conversion of methane to C_2 hydrocarbons in a pulsed corona discharge. Results for the electrodes were in the following order (88):

Platinum > palladium > copper

Conclusion

From the above discussion, it may be inferred that using catalysts, particularly catalytically active platinum group metals, in cold-plasma reactors has become an active area of research in the last few years. The growing demand for controlling VOC emissions, for better utilisation of natural gas reserves, together with the rapid pace of development in this area, suggests that there may well be future applications for such catalysts on an industrial scale in cold-plasma reactors.

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Encapsulation of Platinum in Dendrimers

The high surface:volume ratios of platinum nanoparticles enhances their catalytic properties, and makes their production and utilisation of great importance. A successful sonochemical preparation of platinum nanoparticles is reported on page 108 of this journal.

Conventional routes for nanoparticle preparation include evaporation and condensation, and metal salts reduction, both requiring stabilisers of polymers, ligands or surfactants to control particle size and prevent agglomeration, but which passivate the cluster surfaces. Clusters and colloids can also be prepared using templates of reverse micelles and porous membranes, but again passivation occurs unless the template is removed.

Now researchers from Texas A & M University have developed a method to produce a less passivated platinum catalyst by using a dendrimer as both stabiliser and template (M. Zhao and R. M. Crooks, *Adv. Mater.*, 1999, **11**, (3), 217–220). A fourth generation (G4) polyamido-

amine (PAMAM) Starburst dendrimer was loaded with a predetermined amount of platinum(II) ions followed by reduction with BH₃. From 12 to 60 Pt nanoparticles may be inside each dendrimer without agglomeration for up to 150 days. The dendrimers are spherical, monodispersed, highly functionalised and branched, with an open centre and sterically crowded exterior. They can entrap the Pt ions and stabilise the clusters without totally passivating the surface. This hydroxyl-terminated dendrimer provides stability and size control to the Pt particles (diameter ~ 1.5 nm) and permits substrates to penetrate the interior and access the cluster.

When attached to gold electrodes, in the presence of O₂, Au/G4-OH(Pt₆₀) gives an enhanced catalytic current and a shift in peak potential to 75 mV, compared to a Au/G4-OH modified electrode. In the absence of O₂ only a small current is observed, confirming that O₂ reduction is taking place, and making this of potential interest for fuel cell catalysts.