Supercritical water oxidation: An environmentally safe method for the disposal of organic wastes

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Supercritical water oxidation provides a powerful means to transform toxic organic materials into simple, relatively inert oxides. Over the past decade, our understanding of the fundamental chemistry of this process has increased markedly. Many fascinating research papers are appearing from laboratories around the world, on the use of the technique for the decomposition of a variety of organic wastes. This paper summarizes the important findings of few such studies, which are particularly relevant to the disposal of industrial waste water containing organic pollutants.

WATER, the most important and abundant natural solvent has fascinating properties as a reaction solvent in its supercritical conditions. Supercritical water (SCW) is the fluid that is over the critical point of vapour–liquid co-existence state. Properties such as density and dielectric constant of water can be continuously controlled between gas-like and liquid-like values by varying its pressure and temperature. Thus the polarity of water and hence its ability to dissolve various solids, liquids and gases, which are otherwise insoluble or sparingly soluble, can be enhanced significantly by transforming ordinary water into SCW. This phenomenon has been tested and used in recent years in the oxidation of toxic waste water. The process, known as 'supercritical water oxidation' (SCWO) is environmentally clean. It does not use any other chemical, thermal or photolytic method of decomposing the pollutants. The pollutants present in the waste water are totally destroyed in the supercritical oxidation process and the products are carbon dioxide, water and a limited amount of mineral acids based on the halogenated solvent content in the waste water.

What is a supercritical fluid?

A one-component fluid is loosely defined to be supercritical, when its temperature and pressure exceed its critical temperature and pressure, respectively, while it is not far from its critical state[1]. In Figure 1, the region in pressure (P)–temperature (T) phase space, where the fluid is supercritical, is the right upper quadrant (cross-hatched). In the P–T phase, there are two remarkable features: the vapour pressure curve indicating the conditions under which the vapour and liquid coexist, and the critical point at which the distinction between vapour and liquid disappears. In Figure 1, the critical isotherm is $T = T_c$ and the critical isobar $P = P_c$. If the liquid is heated at a constant pressure exceeding the critical pressure, it expands and reaches a vapour-like state without undergoing a phase transition. In short, a fluid is critical when the difference between coexisting liquid and vapour phase disappear. At this point, the isothermal compressibility of the one-phase fluid becomes infinite. In the supercritical region, a state of liquid-like density can
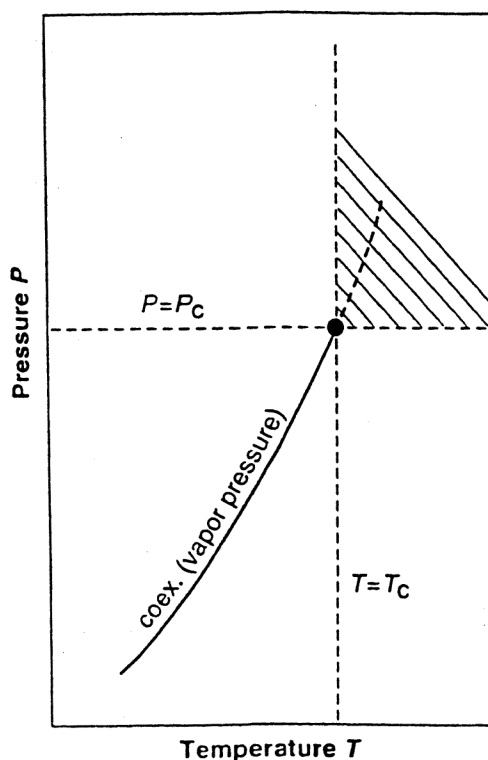

Figure 1. P–T phase diagram of a one-component fluid, with the vapour pressure curve and critical point.

transform into one of vapour-like density, by tuning the pressure or temperature, without the appearance of an interface. The farther from the critical point, the easier it is to gently manipulate the density by tuning the pressure or temperature. In the supercritical fluid, a range of intermediate density states can be reached which are not available at sub-critical temperatures and pressures.

The supercritical region is reached by pressurizing the fluid. As a result, often the term 'compressed fluid' is also used in place of 'supercritical fluid'. However, a compressed fluid can be a supercritical fluid, a near-critical fluid, an expanded liquid or a highly compressed gas depending on temperature, pressure and composition. Many excellent review articles and books are available on supercritical fluid technology[1–3].

Two supercritical fluids which attracted wide attention are carbon dioxide and water. Supercritical carbon dioxide is the subject of a number of investigations, with special focus on its use in extraction processes[4]. It has a mild critical temperature (31°C). It is non-flammable, non-toxic and environment-friendly. It may be a cure for the woes caused by industrial processes that rely on noxious industrial solvents such as acetone, methanol or toluene[5]. Ordinary carbon dioxide gas reaches its super-critical point at modest temperature (31°C) and high pressure (1070 psi) and becomes dense like a liquid, still retaining its ability to flow with almost no viscosity or surface tension. Moreover, it can be obtained from existing industrial processes, without further contribution to the greenhouse effect. It evaporates instantaneously once the pressure is released, leaving no trace behind. It is a small linear molecule, which can diffuse more quickly than conventional bulkier liquid solvents, especially in condensed phases such as polymers. These features make it an excellent solvent for food processing (e.g. coffee decaffeination). Scientists are also discovering that super-critical CO_2 is an intriguing medium in which new materials can be discovered and developed.

Critical parameters of some of the common super-critical fluids are given in Table 1. As seen from the

Table 1. Critical properties of common supercritical fluids[2]

Solvent	Critical temperature (°C)	Critical pressure (MPa)	Density (g/cm^3)
Ethylene	9.3	5.04	0.22
Xenon	16.6	5.84	0.12
Carbon dioxide	31.1	7.38	0.47
Ethane	32.2	4.88	0.20
Nitrous oxide	36.5	7.17	0.45
Propane	96.7	4.25	0.22
Ammonia	132.5	11.28	0.24
n-Pentane	196.5	3.37	0.24
Isopropanol	235.2	4.76	0.27
Methanol	239.5	8.10	0.27
Toluene	318.6	4.11	0.29
Water	374.2	22.05	0.32

table, water has an unusually high (374°C) critical temperature owing to its polarity. At supercritical conditions, water can dissolve gases such as O_2 and non-polar organic compounds. This is because of the possibility of varying the dielectric constant between 5 and 25, which corresponds to the dielectric properties of polar organic liquids under normal conditions.

Properties of supercritical water

Above the critical temperature and pressure, the properties of water are quite different from those of the normal liquid or steam at atmospheric pressure. For example, the solvent property of water changes significantly in this region. Under supercritical conditions, organic substances are completely soluble in water. The critical point occurs at a temperature 374°C and density 0.3 g/cm^3. Supercritical region lies above 374°C. Measurement of the density at different temperatures shows that, near the critical point, the density varies very rapidly with relatively small changes in temperature. The dielectric constant also varies with temperature. Normal liquid water has a dielectric constant (ε) of 80, largely as a result of strong hydrogen bonding. Dielectric constant of the liquid decreases rapidly with increase in temperature, even though the density falls slowly[6]. For example, at 130°C, (density 0.9 g/cm^3) the dielectric constant (ε) is around 50, which is near that of formic acid. At 200°C (density 0.8 g/cm^3), the dielectric constant of 25 is similar to that of ethanol. At the critical point, the dielectric constant is 5. Raman spectra of hydrogen deuterium oxide (HDO) in this region indicate that the residual hydrogen bonding is negligible[7]. The major contribution to ε at this stage is from molecular association due to dipole–dipole interaction, which gradually decreases with increase in void volume.

The dissolving power of water for organics varies with the dielectric constant, even though that is not the only parameter determining the solubility. Figure 2 represents a typical case of correlation between the solubility, dielectric constant and density of water[6,8–10]. As seen from Figure 2 at 25°C, benzene is sparingly soluble in water (0.07 weight%). Between 200 and 500°C, it is completely miscible with water. Other hydrocarbons also exhibit similar solubility behaviour.

As the solubility of organics in water increases with increase in temperature (and consequent decrease in dielectric constant), the behaviour is different, and even reverse, with respect to inorganic salts[6]. For example, the solubility of salts reaches a maximum at 300–400°C. Beyond the maximum, the solubility drops very rapidly with increase in temperature, e.g. the solubility of NaCl is 40 weight% at 300°C and 100 ppm at 450°C. $CaCl_2$ has a maximum solubility of 70 weight% at sub-critical temperatures, which drops to 10 ppm at 500°C. Given the

fact that the dielectric constant of water is only around 2 at 490°C and 25.3 MPa, it is not surprising that inorganics are practically insoluble in SCW. Thus SCW has practically no dissolving power for inorganic salts. Simultaneously, water also loses its ability to dissociate salts. For example, the dissociation constant of NaCl at temperatures of 400 to 500°C and density 0.35 g/cm^3 is of the order of 10^{-4}. Thus strong electrolytes become weak electrolytes in SCW[11].

Figure 2 also shows the solubility behaviour of salts in water under supercritical conditions.

Supercritical water oxidation

SCWO also known as 'supercritical wet oxidation' or 'supercritical wet-air oxidation' is the oxidation of organics with air or oxygen, in the presence of a high concentration of water, under temperatures and pressures above the critical point values of water; 374°C and 22 MPa (218 atmospheres). The process has been undergoing extensive development and testing as a means of disposal of organic wastes, since the ground-breaking observations by Modell et al.[12] that the tars formed during the decomposition of organics in water, below the critical temperature disappeared above it. Since then,

Figure 2. Properties of water at 25.3 MPa.

extensive work has been done in many parts of the world exploring the role of SCWO in the disposal of organic materials[13–15]. Robert Shaw et al.[15] have done pioneering work in this field, especially in the disposal of extremely toxic and hazardous materials such as chemical warfare agents.

As a waste destruction process, SCWO has several advantages over conventional processes and even some of the relatively modern processes such as wet-air oxidation and incineration. These advantages mainly arise from the properties of SCW itself. As a medium for chemical reactions, depending on its density, SCW has both gas-like and liquid-like properties. The gas-like low viscosity promotes mass transfer. The liquid-like density promotes solvation. The low dielectric constant promotes dissolution of non-polar organic materials. The high temperature increases thermal reaction rates. These properties provide a reactor medium in which mixing is fast, organic materials dissolve well and react quickly with oxygen, and the salts precipitate.

The oxidation reaction is complete: carbon goes to carbon dioxide, hydrogen to water, nitrogen to nitrogen gas or nitrous oxide. Heteroatoms form the corresponding oxyacids or salts if cations are present in the waste or added to the feed. Under supercritical conditions, the salt may remain dissolved in the SCW medium or condense as a concentrated brine solution or as a solid particulate. Heavy metals may form oxides or carbonates, which may or may not precipitate, depending on their volatility. Inert solids will largely be unaffected by the medium and remain as solids. Time required to complete the reactions is short. So reactor residence time ranging from a few seconds to a few minutes is sufficient for complete decomposition of most waste materials. Shorter reactor residence time means higher waste throughput. A comparative study of important operation parameters of three oxidation processes is given in Table 2.

The reactor in which SCWO is carried out is enclosed and operated under strict control. The system is not in contact with the outside and it can be quickly shutdown. The operating temperatures are much lower than those of an incinerator. So nitrogen oxides are not formed. SCWO systems can be made compact and portable[15]. They can be taken to toxic waste sites for the on-site disposal of small quantities of the waste. The process can be adapted to a wide range of feed mixtures. However, most of the applications to-date have been limited to laboratory and pilot-plant level, and no large-scale commercial application is reported yet. The challenges posed by the extremely reactive environment in SCWO, to the design and construction of suitable reactors, are yet to be overcome. Some of these problems are discussed later in this paper.

The advantages discussed above open the possibility of using SCWO as an environmentally attractive technology for the complete destruction of organic wastes. The
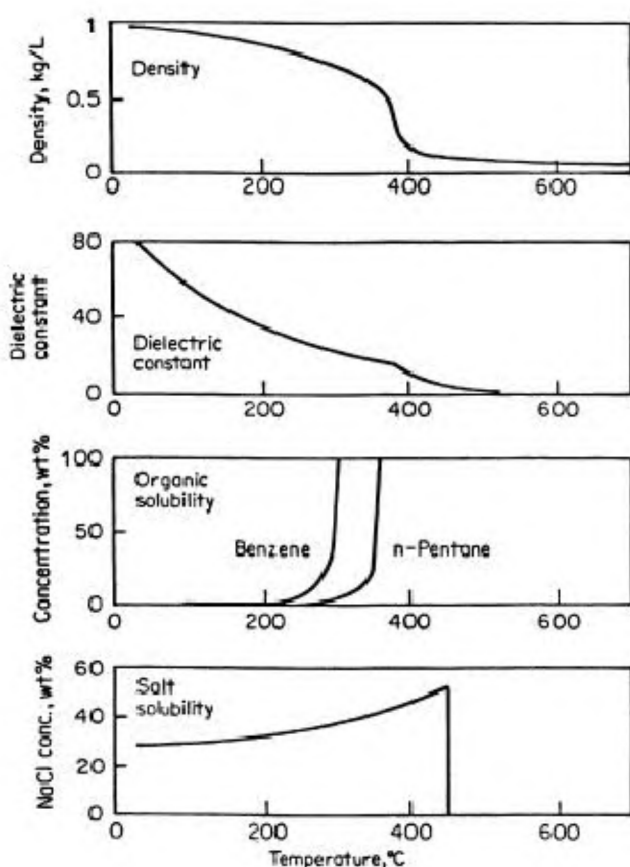
process has been the subject of extensive studies in recent years and research papers dealing with new reactor systems in near-critical and SCW continue to appear at an ever-increasing rate. In two excellent articles covering the field, Savage and co-workers[16,17] have reviewed the developments in the area until 1998. The first article[16] dealt with reactions at supercritical conditions in all fluids (not only in water), while the second article[17] covered three broad areas of research in this field, namely chemical synthesis, decomposition of organic materials and compounds, and complete oxidation. Of these, oxidation (SCWO) continues to be the most fascinating area of research for many workers, particularly in view of its potential to be exploited as an environmentally safe method for the disposal of chemical waste. This review deals with major developments in the field of SCWO, since the publication of the article by Savage[17]. The emphasis here is on the SCWO of organic pollutants, with special focus on hazardous and toxic chemicals. Hence, few papers which appeared prior to the above review but are relevant in the present context, are also referred to for the sake of clarity and continuity.

Supercritical water oxidation of organics in waste

One of the earliest publications on the use of SCWO in toxic waste management was by Thomason and Modell[18]. They have designed and built SCWO reactors at two companies, MODAR and MODEC, and reported on destruction of organic compounds, including polychlorinated biphenyls and DDT to 99.99% efficiency without the formation of dioxins. These reactors could also be used successfully for the disposal of municipal sewage sludge and paper mill waste[15]. The viscous, foul-smelling feed material which went in one end of the reactor came out in the form of clear water and gases. For the sewage sludge, a fine powder of iron oxide and aluminosilicates – soil present in the original feed material – was left behind.

An interesting study on the destruction of municipal excess sludge (and alcohol distillery waste water of molasses) was carried out by Goto et al.[19]. The municipal excess sludge used as the feed material was a slurry mixture of solid micro-organisms, dissolved organic and inorganic materials. The chemical composition of the

distillery waste water was also similar with comparable contents of C, N and H. The reaction was carried out in a batch reactor or a flow reactor with H_2O_2 as an oxidant, in the temperature range of 473–873 K. The reaction products were colourless and odourless, and the total organic carbon was completely removed when more than the stoichiometric amount of oxidant was used. Acetaldehyde, acetic acid and ammonia were the major intermediates in the reaction, which got mineralized in the process. Raman spectroscopic study of the oxidation of ethanol in SCW[20] also showed that aldehyde (formaldehyde) is the primary stable organic intermediate in the reaction involving alcohol or alcohol-containing wastes. Other products formed in the reaction at different stages were identified as CO, CO_2 and H_2O_2, which can serve as an oxidant, is an important product as well as reactant in these systems. Detailed kinetic studies[21,22] of methanol oxidation in SCW also showed that the reaction proceeded through typical 'oxidation-free radicals' HO, HO_2 and CH_2OH. However, in the absence of adequate oxygen in the system, the reaction changes to pyrolysis, in which H and CH_3 are the central free radicals in addition to CH_3O, and methane is one of the main products[22]. In these reactions water acts as an active reactant and reaction medium.

SCWO has been tested as an effective method for treating waste plastics. Vast amounts of non-degradable waste plastics pose significant environmental problems and various treatment or decomposition processes are under development. For polycondensation polymers such as polyethylene terephthalate (PET), polyurethane, nylon, etc. hydrolysis in SCW is reported to be a promising method for the recovery of monomers[23]. In this process, PET is decomposed to its monomer at around 400°C and 25 MPa pressure in SCW. The recovery yield of terephthalic acid (TPA) reaches almost 100% above 100°C. Similarly, addition polymerization plastics such as polyethylene, polypropylene and polystyrene are fully converted into shorter chain hydrocarbons in SCW at 400°C and 40 MPa in 30–120 min[24]. Pilot-plant studies of SCWO using the esterification waste water from PET industry (a major contaminant of which is ethylene glycol) showed that the 'total organic carbon' (TOC) removal efficiency was as high as 99.9% at 630°C and 23 MPa pressure[25]. The application of the process as an effective method for the disposal of plastic waste was reported by many other workers also[26,27].

Table 2. Comparison of three oxidative waste treatment processes[15]

Process	Temperature (°C)	Pressure (bar)	Phase	Residence time
Wet-air oxidation	150–300	10–200	Three phases – solid, liquid, gas	10–100 min
Supercritical water oxidation	500–650	200–1000	One phase – super critical fluid	10 s–1 min
Incineration	800–1100	1	Three phases – solid, liquid, gas	0 s

Many groups have reported that compounds containing heteroatoms hydrolyse rapidly in near-critical water or SCW, as in the hydrolysis of ethers[28], guaicol[29] and aniline[30]. Oxygen-bridged aromatics such as lignin react to give simple aromatic compounds such as phenols, which can undergo further oxidation under suitable super-critical conditions[21]. Compounds with many polar bonds such as carbohydrates are gasified in SCW to hydrogen, methane and carbon dioxide[31]. In all these cases, the reaction pathways are observed to be similar to semi-conductor-assisted photo-catalysed reactions in aqueous medium[32,33].

Detection of methyl tertiary butyl ether (MTBE) in groundwater has prompted many studies to understand the chemistry of MTBE in water and to investigate potential treatment options. MTBE is a common gasoline additive that was initially used to enhance the octane rating and to reduce the engine knock, replacing organo-lead additives used earlier. However, from underground storage tank leaks and other routes, MTBE is rapidly contaminating the groundwater, mainly due to its high solubility and mobility in water. The problem is further exacerbated by the fact that MTBE does not biodegrade under natural conditions and does not favourably adsorb onto carbon or soil particles. Studies on the hydrolysis of MTBE in sub- and supercritical water showed that it undergoes acid-catalysed hydrolysis in water under such conditions. The primary products of the reaction include methanol, isobutene and tertiary butyl alcohol[34].

Another compound, often encountered in many industrial-waste streams and which has been subjected to a number of studies for its oxidation in SCW, is phenol. Thornton and Savage[35] analysed the kinetics of the disappearance of phenol in SCW and showed that the reaction is of first-order in phenol and half-order in O_2 and is influenced by the total pressure. Later, Gopalan and Savage[36,37] simulated the reaction progress and reported that some kind of recombination process yielding dimeric compounds is a very important step in the overall reaction. Recent studies on the reaction at low concentrations of phenol ($1-3 \times 10^{-5}$ mol/dm^3) showed that the rate of phenol disappearance is proportional to the concentration of phenol and to the 0.4 power of O_2 concentration[38,39]. The decomposition is complete here. However, at higher concentrations (2%) the product distribution is found to be different, with the formation of tarry material, even though conversion follows the patterns predicted for much lower concentrations[40]. One possible explanation for this result is that the mechanism of the initiation of phenol decomposition is the same, regardless of its concentration, but that the succeeding radical reactions are different and influenced by radical concentration. The additive reaction between aromatic compounds resulting in the tarry material, was favoured by high phenol concentration. However, when the residence time is extended, the phenol gets completely oxidized[41].

The technique has been tested for the decomposition of heterocyclics such as quinoline and isoquinoline, which are frequently encountered in the environment because of their presence in fossil and synthetic fuels (oil shales, coal, oil sands and shale oils) and some pesticide mixtures, such as creosote[42]. These compounds are known to cause health risks, especially due to the presence of S and N in them. Efforts to reduce their presence by decomposition into non-toxic compounds have been receiving attention in recent years. Studies on the SCWO of quinoline and isoquinoline suggest that the process is effective for the complete degradation of these pollutants in water. Here water acts as a chemical reagent as well as a solvent. The decomposition was more effective when compared to pyrolysis. The products as well as their distribution were also different in the two processes, as shown in Table 3.

Carbon dioxide is a predominant gaseous product in these reactions. This suggests that isoquinoline or its reaction products undergo oxidation reaction. It is inferred that the dominant reactions during the SCWO are: hydrogenation, with the hydrogen produced from water and hydrocracking due to the relatively high reaction temperature.

The study also reported that addition of small amounts of Fe_2O_3 enhanced the decomposition of isoquinoline by SCW.

In an interesting study on the hydrolysis of cellulose in SCW[43], it was seen that valuable chemicals such as oligomers and glucose could be recovered from the reaction by suitably controlling the conditions. Under subcritical conditions, the hydrolysis rate of cellulose is slower than the conversion rate of the hydrolysis products; glucose or oligomers. However, under supercritical conditions, the hydrolysis rate jumps to more than an order of magnitude higher level and becomes faster than the glucose or oligomer decomposition rate. Thus a good yield of glucose and the oligomer can be obtained under supercritical conditions. Minowa and co-workers[44] reported that the efficiency of decomposition of cellulose in water under SC conditions is enhanced significantly in the presence of Ni or sodium carbonate catalyst.

SCWO is reported to be a promising alternative in the disposal of 'difficult-to-treat' wastes such as sulphonated

Table 3. Reaction products from pyrolysis and SCWO of isoquinoline (products as % of the composition)

Reaction product	Pyrolysis	SCWO
Toluene	–	Trace
o-Xylene	–	17.9
Ethylbenzene	–	18.3
Benzene,1,3-dimethyl	–	Trace
Benzene,1-ethyl-2-methyl	31.0	21.2
Benzene,1-ethyl-3-methyl	–	21.2
Naphthalene,2-methyl	69.0	–
Benzo F quinoline	–	20.9
5H-indo-1,2-b-b-pyridine	–	Trace

lignin wastes[45]. These are the primary wastes from polyester fibre and chemical pulping process industries. They also constitute a major component of the Kraft pulping process waste water. It is estimated that production of one tonne of pulp requires between 16,000 and 17,000 gallons of water[46] and hence it is of utmost importance that the waste water is free of any pollutants. Sulphonated lignins are polymers of phenyl propane with methoxyl groups that exhibit polyelectrolyte behaviour in an aqueous solution. These are very stable chemicals, which cannot be effectively deactivated by conventional waste water-treatment methods. The properties of sulphonated lignin vary depending on the molecular weight and molecular weight distribution (which is analogous to the degree of polymerization or chain length), the degree of sulphonation and the purity of the product. The chemical structure of sulphonated lignins is very complex and only partially understood. A typical sulphonated repeat unit of lignin can be represented as shown in Scheme 1.

Detailed kinetic study of the oxidation under supercritical conditions suggested that the technique can be used for the effective decomposition of sulphonated lignin in waste water and the overall rate-limiting step is the oxidation of the ring C–C bonds[45].

One of the eagerly awaited applications of SCWO is the destruction of halogenated hydrocarbons in waste streams. Michael Modell[47] of Modell Development Corporation has done pioneering work in this area. His studies have shown that halogenated aliphatics such as 1,1,1-trichloroethane, 1,2-ethylene dichloride, 1,2-dichloroethane, 1,1,2,2-tetrachloroethylene and hexachlorocyclohexane as well as halogenated aromatics such as o-chlorotoluene, hexachlorocyclopentadiene, 1,2,4-trichlorobenzene, 4,4-dichlorobiphenyl, DDT and PCB can be decomposed to 99.999% completion under supercritical conditions of water. Sako et al.[48] reported that perchlorinated dibenzo-p-dioxins and perchlorinated dibenzofurans in fly ash can be decomposed to less than 1% using SCWO at 673K. Similarly, 2,3,7,8-dibenzo-p-dioxines, tetra and octa chlorinated dibenzofuran and octachlorinated dibenzo-p-dioxin are decomposed with a destruction efficiency of higher than 99.9% at temperatures of 873–903 K and a residence time as short as 5 s (ref. 49). Many catalysts are also being examined for improving the efficiency of SCW-assisted decomposition of chlorinated organic wastes. SCWO has already been approved as a treatment technology to decompose polychlorinated biphenyl in Japan.
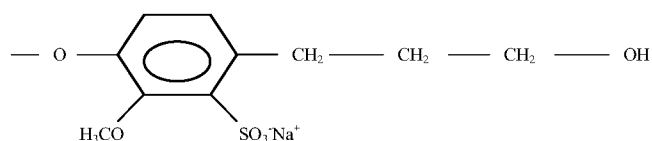
One of the major bottlenecks in the commercialization of the SCWO is the extreme condition of reaction. Most of the reaction products from hazardous chemicals decomposition are highly corrosive and no suitable commercially viable material which can withstand the corrosive nature of the reaction media on a large scale, has yet been found. To overcome this situation, a novel reactor concept was tested recently in which the reactor resists the attack of HCl under SCW conditions[50]. The technique, known as SUWOX, uses a floating-type acid-resistant reactor within a stainless steel pressure vessel and a process operation is conducted in a parallel-flow system. The system was tested for the decomposition of different polychlorinated hydrocarbons. The oxidation reaction and the formation of HCl are safely confined within the reactor housing made of alumina. For operations up to 1000 h involving the destruction of dichloromethane, no corrosion of the pressure vessel has been observed. The complete destruction of dichloromethane (99.99%) to CO_2 was achieved in less than 4 min at system pressures of 400 bar, reaction temperatures of 400°C and O_2 stoichiometry of 2. The performance of the reactor is promising in the case of sulphur containing compounds as well.

Catalysts in SCWO

Catalysts, like in many other processes, are found to enhance the efficiency of SCWO. However, investigations on the use of catalysts in SCWO systems have gained momentum only recently. Ding et al.[51] have summarized the relatively small number of SCWO studies on various catalysts reported until early 1996. Catalysed SCWO was reported[52] to yield higher conversion and higher selectivity (than uncatalysed SCWO) in the case of alcohols, acetic acid and phenol. It has also been reported[44] that by using a proper catalyst, the reaction temperature can be lowered to 400°C without sacrificing the destruction efficiency.

The use of Fe_2O_3 and Ni/Na_2CO_3 as catalysts in the SCWO of isoquinoline and cellulose respectively, was mentioned earlier in this article. Aki and Abraham[53,54] have investigated the SCWO of pyridine in the presence of several catalysts such as Pt/γ-Al_2O_3, MnO_2/γ-Al_2O_3 and MnO_2/CeO_2, and observed that the reaction rate is enhanced manyfold by the use of catalysts. Among these, platinum catalyst was the most effective and stable. The kinetics and mechanism of the reaction are different on different catalysts and so are the end-products. The oxidation reaction of pyridine to its ultimate products is as follows:

$$C_5H_5N + 25/4\ O_2 \rightarrow 5CO_2 + 1/2\ N_2 + 5/2\ H_2O.$$

Several partial oxidation or condensation products were also detected, though eventually they also got oxidized.

Scheme 1.

These included ammonia, dimethylamine and several carboxylic acids such as formic acid, acetic acid and oxalic acid[55].

Sodium carbonate is observed to be an effective catalyst in many SCWO reactions. Alkali materials as well as Ni are known as good catalysts for oil production from biomass and they inhibit the formation of char from oil[56]. Under supercritical conditions, the alkali and Ni even catalyse the steam-reforming reaction of aqueous products and methanation reaction[57]. Sodium carbonate is also found to assist the SCW oxidation of chlorinated wastes[58]. In this case, sodium carbonate acts as a catalyst as well as an HCl scavenger. Also, sodium carbonate plays a key role in reducing corrosion on the reactor walls by first neutralizing the acid and then providing a large surface area for adsorption of the precipitated corrosive compounds. Another catalyst, subjected to extensive studies for its effectiveness in SCWO of trace organics, is MnO_2/CeO_2 (refs. 59, 60). In these studies, the addition of the catalyst to the SCW system led to the complete destruction of the organics in the temperature range of 380 to 450°C.

Yu and Savage[61] studied the activity, selectivity and transformation of catalysts during oxidation of phenol in SCW, in a tubular reactor. Three different catalysts, bulk MnO_2, bulk TiO_2 and CuO/Al_2O_3 were used in these studies. All three catalysts largely maintained their activities throughout for more than 100 h of continuous use. However, CuO/Al_2O_3 was not stable, while the other two catalysts remained fairly stable. All three catalysts and the support underwent transformation: MnO_2 to Mn_2O_3, CuO to Cu_2O, the Al_2O_3 support to AlO(OH) and anatase TiO_2 to rutile TiO_2. Bulk MnO_2 appeared to be the best oxidation catalyst with good stability, high activity and good activity maintenance.

Identification of suitable catalysts is expected to help in reducing the extreme process conditions of SCWO. Extensive studies on a number of catalyst systems are in progress in many laboratories worldwide, to make the process more amenable to commercial applications.
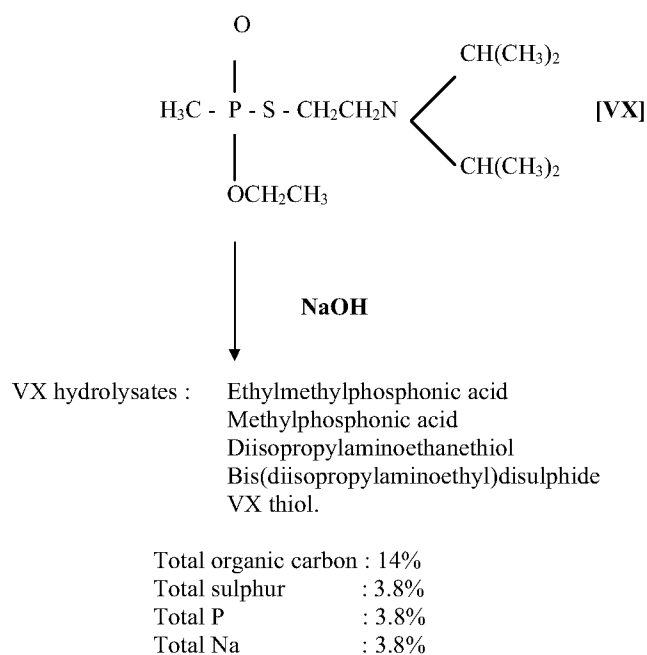
SCWO in destruction of chemical weapons

One of the most important potential applications of the technique is the environmentally safe destruction of chemical warfare agents. The technology currently used in the destruction of these extremely hazardous and toxic chemicals is incineration at high temperatures. Most of these agents are P, F and S containing molecules and their decomposition products consist of highly corrosive materials such as HF, H_3PO_4, P_2O_5, HCl, SO_2, N_2O_5, etc. These can eat away the refractory lining of the incinerator furnace and this results in expensive replacements, time loss and cumbersome operations. Ever since the possibility of using SCWO as a viable technology for the decomposition of hazardous chemicals was identified, it

was considered as a replacement for the incineration of chemical warfare agents as well. Shaw and co-workers[15,62] have done extensive studies on this possibility and have summarized the current status of research in this area in various laboratories. These studies have increased the knowledge on chemical reaction rates of CW simulants, properties of hot aqueous solutions, nucleation and precipitation of salts, equations of state, corrosion of reaction materials, etc. SCWO is already in use for destruction of military smokes, dyes and explosives. The challenges posed by the corrosive products generated from the SCWO of nerve and mustard agents is being solved by using suitable reactor material and special designs. The technique is currently applied in the destruction of small quantities of chemical warfare agents, organic wastes from ship operations and aircraft maintenance, wastes from semiconductor processing and pulp mills and municipal sewage.

One of the chemical warfare agents which has been successfully tested for its complete destruction by SCWO method is VX (o-ethyl-S-2-diisopropylaminoethyl methyl-phosphonothiolate). In this case, the agent is first treated with sodium hydroxide and the resulting products are completely detoxified into carbon dioxide and inorganic salts by SCWO. The chemistry of the process may be summarized as shown in Scheme 2 (ref. 63):

The hydrolysis products, known as VX hydrolysates, whose composition is approximately as shown in Scheme 2 are subjected to SCWO. The reaction products are reported to be carbon dioxide and inorganic salts. The liquid effluent contains mainly a 1 : 1 molar mixture of NaH_2PO_4, Na_2HPO_4 and Na_2SO_4.

VX hydrolysates : Ethylmethylphosphonic acid
Methylphosphonic acid
Diisopropylaminoethanethiol
Bis(diisopropylaminoethyl)disulphide
VX thiol.

Total organic carbon : 14%
Total sulphur   : 3.8%
Total P   : 3.8%
Total Na   : 3.8%

Scheme 2.

This may be considered as one of the best applications of the use of SCWO in the decomposition of extremely hazardous and toxic compounds into harmless products.

The process

SCWO can be applied to wastes with a wide range of organic concentration. A simplified flow chart for the process is presented in Figure 3. The process consists of the following steps[6]:

The waste, as either an aqueous solution or slurry is pressurized and delivered to the oxidizer inlet. Oxygen is supplied in the form of compressed air. Both oxygen and air have been used with equal success for SCWO and the choice is dictated by economics. Oxygen compression is considerably less expensive than air and for large-scale applications, it will be more economical. The recycled reactor effluent (which is mostly SCW) also joins the inlet stream, thereby making the reactor feed a homogeneous phase of air, organics and SCW. The organics get oxidized in a controlled but rapid reaction. The heat released by the combustion is sufficient to raise the temperatures to a level at which all organics are oxidized rapidly. The effluents from the oxidizer are then fed to a cyclone in which the solids (salts from the original feed as well as those formed in the SCWO reaction) are separated. The fluid effluent of the solid separator is a mixture of H_2O, N_2 and CO_2, a portion of which is recycled to provide supercritical conditions at the oxidizer inlet. The remainder of the effluent, which is a high-temperature high-pressure fluid, is cooled to a subcritical temperature in a heat exchanger, which serves to generate low and high-pressure steam. The outlet stream from the steam generator is now at subcritical temperature and is fed to a liquid–vapour separator. Here, the gas phase consisting of N_2 and most of the CO_2 is let-off as clean gas effluent. Alternately, the gas stream can be expanded

through a turbine to extract the available energy as power. A portion of this power can be used for compression of the inlet air. The liquid consists of water with an appreciable amount of CO_2. This liquid, if needed, may be depressurized and fed to a low-pressure separator. The vapour stream is primarily CO_2, which is vented with the gas turbine effluent. The liquid stream is clean water. Many of the SCWO systems in use today are very small and power recovery cannot be justified on economic grounds. Hence the heat of combustion and the energy input for air compression are recovered as steam.

The efficiency of the reaction (for the complete decomposition of the target chemical) was found to be a function of reaction temperature and residence time. Modell[64] has made a series of studies on this and has reported that reactor effluent temperature of 600 to 650°C and a residence time of 5 s are sufficient for the complete oxidation (99.999%) of many organics. Higher temperature could be used for reducing the residence time.

Results of some typical bench-scale studies

Some of the laboratory results from various research studies have been tested at the bench-scale using synthetic mixtures of pure chemicals. Most of these reactor units consisted of continuos flow systems with a throughput of 0.06 m^3/day of water containing 5% organics. Operating temperatures ranged from 400 to 700°C at 250 atm. pressure. The residence time varied from 0.5 to 7–10 min, depending on the reactor length and SCW flow rate. Selected results relevant for most industrial wastes are summarized in Table 4.

While numerous pilot-plant studies have been conducted on widely varying feedstock, SCWO is still in its developing phase for commercial operations. Currently, two SCWO plants are reported to be undergoing commercial testing and operation[18]. These plants incorporate special wall reactor design to reduce the corrosion. One of these plants is designed to destroy navy excess hazardous materials (EHM), which consists of organic materials such as chlorinated solvents and lube oils, found aboard navy ships. The plant is reporting destruction removal efficiency of 99.99%. A second plant designed for the destruction of obsolete-coloured smokes/ dyes and pyrotechnic munitions is also fully operational.

Principal challenges

The properties that make SCW a good reaction medium are also disadvantageous to the process today. The very reactive chemistry, which is the advantage of SCW systems, can attack the reactor, especially if the compounds being treated contain heteroatoms (atoms other than H, C, N or O). Halogens are especially reactive and
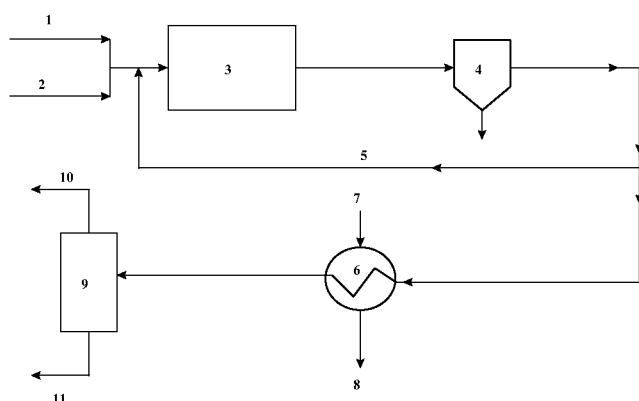
Figure 3. Process flow diagram for the SCWO of a typical organic waste. 1, Organic waste; 2, Compressed air; 3, SCWO reactor; 4, Solids separator; 5, Recycling; 6, Steam generator; 7, Water; 8, Steam; 9, Liquid–vapour separator; 10, Clean gas effluent; 11, Clean water effluent.

treatment of any halogen-containing compound will require special reactor material and/or design to reduce corrosion. Heteroatoms generally form their corresponding inorganic acids during SCWO (e.g. Cl forms HCl), and often a strong base is added to the feed to neutralize these acids and reduce corrosion. Design of appropriate systems to facilitate the feed, reaction and subsequent operations with minimum contact with the corrosive materials as well as the invention and use of special corrosion-resistant alloys which can extend the life of the SCWO systems, is a major challenge in the large-scale use of the technology. While Ni and Ni-based alloys are generally considered important for severe service applications, results from laboratory and pilot-scale SCWO systems presently in operation indicate that they will not withstand some aggressive feeds[65]. Materials such as Ti and its alloys, refractory metals such as Mo, Nb and Ta, noble metals such as Pt, Rh and Ir have been tested in this context for corrosion resistance[1]. However, these materials are too costly to be used in large commercial operations. Innovative reactor design and judicious feed modification are being tested as alternatives in place of exotic reactor materials, to control corrosion[18,50,66]. Extensive research is in progress in the area.

Another challenge is from the low solubility of salts in the SCW. The low dielectric constant of SCW, which makes it a good solvent for non-polar organic substances, also makes it a poor solvent for inorganic salts. The addition of strong bases to neutralize the acids formed during the SCWO, generates salts that may precipitate onto the reactor surfaces and change flow pattern and even block the reactors. Heat exchanger fouling or scale build-up is another factor limiting the kind and concentration of salts that can be processed. The salt and corrosion problems are linked; they are associated with the presence of heteroatoms. Likewise, in the absence of oxidant, certain organic materials are prone to polymerization to form insoluble tar or char which may also lead to scaling of heat-transfer surfaces or plugging. Feedstock with high

levels of heteroatoms which form higher concentration of salts must be managed using special cleaning techniques, reactor designs or by running at very high pressures to keep the salts in solution. The order and temperature/pressure conditions under which the various inputs are introduced into the SCWO system are also important. Hong[63] has critically evaluated various salt-water systems relevant to SCWO and suggested that studies on the phase behaviour and solubility information for systems containing carbonate, phosphate and fluoride salts as well as salt-water systems containing O_2, N_2 and/or CO_2 would be of particular value in this context. Another complication arises from the surface charge, existing on oxidized metal surfaces, which leads to adsorption of ions from solution (depending on pH, temperature and the nature of metal surface). This may also affect the rate of dissolution of the underlying metal[67]. Non-condensable gases such as N_2, O_2 and CO_2 present in the SCWO feeds may also cause problems in the near-critical region owing to the effects of two-phase flow on fluid dynamics (heat and mass transfer). Basic research in areas such as corrosion, thermodynamics, thermochemistry, chemical kinetics, modelling, physical transport, mixing, phase separations, reactor modelling and process simulation in relation to SCWO is important in identifying solutions to these problems.

Evaluation of SCWO as an energetic source is also just at the beginning and more research is needed in this field as well. Unfortunately, little work has been done in this field to find the optimum reactor concept and the optimum process parameters. For a better understanding and evaluation of the 'energetic' SCWO, the results obtained from different reactor concepts of the 'waste treatment' SCWO will be immensely helpful[68]. Finding the optimum process parameters and the optimum reactor concepts is vital in achieving high efficiency and thus lower cost.

Kritzer and Dinjus[68] have analysed existing problems, possible solutions and new reactor concepts in the field and concluded that SCWO as an 'end-of-pipe' techno-

Table 4. Results of bench-scale evaluation

Compound	Temperature (°C)	Residence time (min)	Efficiency (%)	Reference
Cyclohexane	445	7	99.97	6
Biphenyl	450	7	99.97	6
o-Xylene	495	3.6	99.93	59
1,1,1-Trichloroethane	495	3.6	99.99	59
1,2-Ethylene dichloride	495	3.6	99.99	59
1,1,2,2-Tetrachloroethylene	495	3.6	99.99	59
o-Chlorotoluene	495	3.6	99.99	59
Hexachlorocyclopentadiene	488	3.5	99.99	59
1,2,4-Trichlorobenzene	495	3.6	99.99	59
4,4-Dichlorobiphenyl	500	4.4	99.993	59
DDT	505	3.7	99.997	59
PCB 1234	510	3.7	99.99	59
PCB 1254	510	3.7	99.99	59
Methylethyl ketone	505	3.7	99.993	59
2,4-Dinitro toluene	574	0.5	99.9998	59

logy has some disadvantages and this has hindered the industrial application of the process. These disadvantages include not only the technical problems due to reactor corrosion and salt precipitation, but also the problem of high cost, which needs to be looked into. Most cost-intensive are the investment costs on the plant itself, the staff costs and those for the oxidant. The oxygen costs were considered as one of the major ones based on many pilot-plant studies. However, these studies were run with 200% or more of the stoichiometric oxygen demand, and recent studies[69] have proven that even 5% may be sufficient for total oxidation. Thus, high excess of oxygen may not be realistic for prospective industrial application. Similarly, when used as a waste-treatment technology, SCWO need not be operated with all the precision as for the synthesis of a well-defined pure product starting from pure raw materials. Thus there is more flexibility with respect to process and equipment. Hence it is worth exploring different reactor designs and material concepts in this context. For example, salt-free waste streams can be easily oxidized in simple tube reactors and organics containing only C, H, O and N which do not cause any severe corrosion problems[70,71]. Development of suitable catalytic SCWO systems, in which the organics are oxidized over a heterogeneous catalyst, can also simplify the stringent reactor requirements[72].

Conclusion

SCWO is a powerful technology for the transformation of hazardous and toxic organic wastes into relatively inert compounds. Two principal challenges to the widespread use of this technology are: extreme corrosion-resistant material requirement for the reactor as well as heat-up and cool-down sections and incomplete understanding of the phase behaviour of SCW solutions (and consequent inability to predict the same). Development of equations of state for systems containing salts, water and gases and determination of the static dielectric constant and viscosity of water–nonpolar mixtures at all compositions are key to the development of appropriate disposal technologies for different kinds of waste. The process is especially recommended for the disposal of such hazardous wastes as explosives and propellants, halogenated organics, chemical weapons and miscellaneous organic wastes.

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MEETINGS/SYMPOSIA/SEMINARS

International Workshop on Biotechnological Interventions for Dryland Agriculture – Opportunities and Constraints

Date: 18–20 July 2002
Place: Hyderabad, India

The objectives of the workshop are: (i) To review the state-of-art of technologies on genetic engineering and molecular markers for crop improvement at national and global level; and (ii) To identify specific areas for further interventions by APNLBP. As a part of this workshop, it is also proposed to organize a public debate on 'Agricultural biotechnology: Promise or peril' on 19 July 2002 between 5 and 6:30 p.m.

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The 3rd Federation of Asian-Oceanian Neuroscience Societies Congress (FAONS Congress)

Date: 28 September–1 October 2002
Place: Seoul, Korea

Topics include: Neuronal intracellular signaling, Apoptosis in the nervous system, Stem cell neurobiology, Parkinson's disease, Molecular pathology of Alzheimer's disease, Stroke, Drug abuse, Signaling by ginsenoside, Retinal neurobiology, Hypothalamic integration, Learning and memory, Protein therapy in neuroscience, Alcohol and their neuronal targets, Roles of zinc in the nervous system, Modulation of pain, Calcium channels in neural activity; Neuronal development, degeneration and regeneration.

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