Supercritical Water Oxidation – Current Status of Full-scale Commercial Activity for Waste Destruction

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ABSTRACT

After more than three decades since its potential was first recognized, supercritical water oxidation (SCWO) remains an innovative and viable treatment technology for destruction of aqueous based organic wastes. An extensive data base of destruction efficiencies, corrosion data, and salt phase behavior has been developed over the years through the combined efforts of many investigators at both the fundamental research and commercial level. As a result, SCWO technology has been increasingly utilized in a variety of full-scale designs and applications, handling feeds as diverse as polychlorinated biphenyls (PCBs), sewage sludge, spent catalysts, and chemical weapons. This paper reviews the status of current full-scale commercial SCWO facilities around the world, focusing on the unique challenges and design strategies employed by different companies for corrosion and salt precipitation control in each application. A summary of past commercial SCWO activity as well as future plans among the current active SCWO companies is also included.

INTRODUCTION

Supercritical water is a unique medium that has been studied for a growing and increasingly diverse number of applications. Above its thermodynamic critical point (374°C, 221 bar), water, like all supercritical fluids, exists as a single dense phase with transport properties comparable to those of a gas and solvent properties comparable to those of a liquid. Unlike other supercritical fluids, however, water undergoes a significant change in its solvent behavior between ambient and supercritical conditions. As water is heated under pressure, it loses a sufficient amount of its hydrogen bonding to transition from a highly polar solvent to nonpolar solvent [1]. This phenomenon is evidenced by the dramatic drop in water's dielectric constant and dissociation constant near the critical point and beyond. The result is that supercritical water becomes a very good solvent for nonpolar materials such as hydrocarbons and oxygen gas and a poor solvent for polar materials such as salts and other ionic compounds, which is opposite behavior to that exhibited at ambient conditions. With a fuel source and oxidant fully miscible at high temperature in a dense, single phase environment, oxidation reactions are rapid and complete in supercritical water. This process, referred to as supercritical water oxidation (SCWO), has been developed as a means of waste destruction for aqueous organic wastes. While the unique properties of near-critical and supercritical water have been utilized in an increasing number of applications ranging from power cycles to synthesis reactions to biofuels formation [2, 3], SCWO is the application which has been studied the longest and is furthest along in commercial development. Several recent review articles provide more details on the properties of supercritical water and on SCWO [4-6].

Since its potential was first recognized more than three decades ago, a substantial amount of research on supercritical water and SCWO has been carried out by many distinguished groups in many areas including reaction kinetics, salt nucleation and growth, materials compatibility and corrosion, physical property measurements, and reaction modeling. In parallel with this collection of data, there have been a number of companies over the years that have been established or become involved in the business of commercializing the SCWO process (Table 1). The first commercial SCWO company, MODAR, was established in 1980 (it was bought by General Atomics in 1996). As of January 2012, there are six companies that are still active in commercializing SCWO technology: General Atomics (the oldest among active companies), SRI International, Hanwha Chemical, SuperWater Solutions, SuperCritical Fluids International (SCFI), and Innoveox. By active, it is meant that a company is currently marketing SCWO technology and has at least one full-scale SCWO facility in operation, in construction, or in design. Three of the six companies that are still active today (SuperWater Solutions, SCFI, and Innoveox) were established within the past five years. Thus, while none of the initial companies started in the 1980s are still in business and many subsequent companies have come and gone, new SCWO companies are still being established even today.

Table 1. Past and Present Companies in SCWO Commercialization.

Company (currently active ones in bold)	**Year of Establishment or First Involvement**	**Licensees or Partners**
MODAR, Inc.	1980	Organo Corp.
MODEC (Modell Environmental Corp.)	1986	Organo Corp., Hitachi Plant Engineering & Construction, Ltd., NGK Insulators, Ltd., NORAM Engineering and Constructors, Ltd.
Oxidyne Corp.	1986	-
EcoWaste Technologies, Inc.	1990	Chematur Engineering AB, Shinko Pantec (Kobelco)
Abitibi-Price, Inc.	1991	General Atomics
General Atomics (GA)	1991	Komatsu Ltd., Kurita Water Industries, Ltd.
Turbosystems Engineering	1992	-
Foster Wheeler Development Corp.	1993	Aerojet Gencorp Corp., Sandia National Laboratory
SRI International	1993	Mitsubishi Heavy Industries, Ltd.
KemShredder, Ltd	1993	-
Hanwha Chemical	1994	-
Chematur Engineering AB	1995	Johnson Matthey, WS Atkins, Stora-Enso, Feralco AB
HydroProcessing, L.L.C.	1996	-
Hydrothermale Oxydation Option (HOO)	2000	-
SuperWater Solutions	2006	-
SuperCritical Fluids International (SCFI)	2007	-
Innoveox	2008	-

SCWO PROCESSES

The SCWO process provides rapid destruction of a wide variety of organic species normally considered to be refractory or difficult to breakdown under conventional means such as incineration. Most hydrocarbons and oxygenated hydrocarbons are converted to CO_2 and H_2O. Nitrogen in the feed is converted to N_2 or N_2O. Heteroatoms in the feed such as chlorine, sulfur, or phosphorus are converted to their corresponding mineral acids (HCl, H_2SO_4, or H_3PO_4) or salts if pre-neutralized with base. Typical operating conditions are usually well above the critical point in the range of 500-650°C and 250-300 bar, with reactor residence times under one minute for complete destruction. Under these conditions, dioxins, furans, NO_x and other noxious by-products that plague incineration-based processes do not form in SCWO. Most SCWO reactors tend to be either of the vessel type or tubular (pipe) type, the difference being the magnitude of the diameter. Vessel reactors typically have an internal diameter of at least 10 centimeters and tend to be relatively short, while tubular reactors tend to have a diameter of only 2-5 centimeters or less but are significantly longer.

The targeted niche for most commercial SCWO applications are aqueous organic wastes in the range of 1-20 wt% organic; too concentrated for activated carbon to remove economically but too dilute for incineration to handle efficiently. The easiest wastes to process are those that contain only the elements C, H, O, and N. Heteroatom-containing wastes are more difficult to process, since the associated acids and/or salts that form lead to the two biggest challenges for SCWO processes: corrosion and salt precipitation/accumulation. Salt precipitation is a consequence of the poor solubility exhibited by ionic species in supercritical water. If not prevented or controlled, it can lead to rapid plugging of the reactor in a relatively short period of time depending on the nature of the feed material. Corrosion in SCWO systems is most severe in the hot, subcritical regions before (preheater) and after (cooldown heat exchanger) the reactor, but can also occur in the microenvironment formed under salt layers in the reactor [7]. Depending on the particular feed composition and materials of construction involved, corrosion rates in SCWO can be as high as several mils/hr (tens of μm/hr) [8]. If not controlled, corrosion and salt precipitation can lead to rapid shutdown and/or failure of expensive process equipment, as will be shown later in this paper. Out of this necessity, a number of methods or approaches for handling corrosion and salt precipitation in SCWO systems have been developed over the years by many of the commercial SCWO firms. These methods are listed in Tables 2 and 3 and discussed in more detail in the literature [9, 10]. The philosophy behind these methods for corrosion and salt precipitation control range from actively preventing their occurrence, to managing their occurrence, to limiting operation to feeds where these phenomena cannot occur. The latter approach is one that has been favored by many companies, but is risky if used as the sole means of control, as it requires complete knowledge of feed composition at all times (especially during upsets or slow variations) to avoid exposure of the unprotected system to excessive corrosion or salt plugging. In general, the particular method or combination of methods utilized by a commercial SCWO company is often what distinguishes one company's SCWO process design and operation from another's.

Table 2. Commercial Approaches to SCWO Corrosion Control

Category	Approach
Prevent corrosive species from reaching a solid surface	Transpiring wall reactor or Film-cooled wall reactor
	Adsorption/reaction on fluidized solid phase (Assisted Hydrothermal Oxidation)
	Vortex/circulating flow reactor (conceptual)
Form a corrosion resistant barrier	Use of high corrosion resistance materials (long term applications)
	Liners (corrosion resistant material)
	Coatings
Manage/minimize corrosion	Liners (sacrificial material)
	Use of adequate corrosion resistance materials (short term applications)
Adjust process conditions to avoid or minimize corrosion	Pre-neutralization of feed
	Cold (ambient temperature) feed injection
	Feed dilution with non-corrosive wastes
	Effluent dilution/cooling (quench water addition)
	Optimization of process operating conditions
	Avoidance of corrosive feeds
	Pretreatment to remove corrosive species

FULL-SCALE COMMERCIAL SCWO PLANTS

Table 4 summarizes the status of full-scale commercial SCWO plants associated with each of the six currently active SCWO companies in the world. While the term "full-scale" is somewhat subjective, here it is used not necessarily to denote a certain size as it is to indicate a system that is 1) commercially available on the open market, 2) designed for eliminating a specific waste for a customer (i.e., not for conducting research and not a demonstration unit), and 3) located permanently at a customer's site (not the SCWO company's facility). Most plants that meet this definition are at least on the order of tens of kg/hr with respect to feed capacity. The term "commercial" as used here refers to the SCWO company not the customer, so commercial SCWO plants can be built for either corporate or government customers. For each company, Table 4 shows the number of full-scale plants that are in operation, built but not in operation (either not commissioned yet or shutdown), and in the planning stages (i.e., under design/construction). The latter category represents plants that are under contract to be built, not simply a paper objective or intended goal.

As of January 2012, there are two full-scale commercial SCWO plants in operation as well as one near-critical hydrolysis plant. This is down from as many as seven plants that were in operation ten years ago. However, there are eight SCWO plants that are on the horizon, with seven of these scheduled to come on line within the next one to two years.

The oldest plant currently in operation was built in Tokyo by Mitsubishi Heavy Industries utilizing the Advanced Hydrothermal Oxidation (AHO) variation of SCWO that was developed by SRI International [11]. It was built for and is operated by the Japan Environmental Safety Corporation (JESCO), an agency of the Japanese government,

Table 3. Commercial Approaches to SCWO Salt Precipitation/Accumulation Control

Category	Approach
Avoid salt precipitation	Extreme pressure operation
	Restrict feed type/composition
Allow salts to precipitate but prevent wall surface contact and/or accumulation	Reverse flow, tank reactor with brine pool
	Transpiring wall reactor
	Adsorption/reaction on fluidized solid phase
	Centrifuge reactor
	High velocity flow
	Additives (those which provide surface for nucleation)
	Low turbulence, homogeneous precipitation
	Crossflow filtration
	Density separation
Remove salts after accumulating on surfaces	Reversible flow, tubular reactor
	Mechanical brushing
	Rotating scraper
	Reactor flushing
	Additives (those which alter properties of salt mixture)

Table 4. Summary of Existing SCWO Plants

Active Companies	Full-scale SCWO Plants		
	Operational	**Built**	**Planned**
GA	0	2	2
Hanwha	1[*]	3	0
Innoveox	1	0	4
SCFI	0	0	1
SuperWater Solutions	0	0	1
SRI / Mitsubishi	1	0	0
Total	**3**	**5**	**8**

[*] Near-critical Hydrolysis

for destroying polychlorinated biphenyls (PCBs) [12]. The plant is one of five regional facilities set up by the Japanese government for destroying the country's stockpile of PCBs, but the only one that is based on SCWO technology [13-14]. The plant has a capacity of 2000 kg/day of PCBs and 100,000 kg/day of water. AHO utilizes a solid phase (typically sodium carbonate) that both assists in oxidation and adsorbs the resulting corrosive species (such as chloride in this case). The reactor thus resembles a fluidized bed vessel. Operating conditions are typically in the range of 370-400°C (just hot enough to keep the fluidizing salt in the solid phase) and 265 bar. The Tokyo plant has a second tubular reactor to ensure adequate destruction [15]. This plant has been in operation since November 2005.

The second commercial SCWO plant currently in operation was built by Innoveox and is located in Arthez-de-Béarn in southwestern France [16]. Innoveox is a relatively new company that, like its predecessor HOO [17], has a license to the SCWO technology and patents developed by the Center National de la Recherche Scientifique (CNRS) of France [18, 19]. The system was built for a private customer to process hazardous industrial waste at a relatively low capacity of 100 kg/hr. The system utilizes a tubular reactor with oxygen injected at multiple points along its length. Operating conditions range from 250-550°C (increasing down the length of the reactor due to the multiple oxygen injections) and 265 bar. Feed composition is limited to < 1 g/L chloride and < 10 g/L salt [20]. This system has been in operation since June 2011.

One additional commercial plant in operation that is not SCWO-based but worth mentioning is a Near-Critical Hydrolysis (NCH) facility built by Hanwha Chemical Corp. for Korea Fine Chemical Co. The process is based on a hydrolysis reaction of toluene diisocyanate (TDI) residue carried out in hot water near the critical point [21]. The residue is generated from the manufacture of TDI, a precursor chemical in the manufacture of polyurethane. The hydrolysis reaction takes advantage of the properties of hot subcritical water to convert the otherwise waste material back to its toluene diamine intermediate, which can then be recycled back into the TDI manufacture process. The plant has a capacity of 20,000 kg/day and utilizes a tubular reactor [22]. It has been operating since December 2008. This is a good example of the increasing use of subcritical and supercritical water for applications other than SCWO.

Table 5 includes a list of full-scale commercial SCWO plants that are no longer in operation. As can be seen, there have been a number of SCWO plants over a period of almost two decades that were built for a variety of feeds in many locations around the world. The reasons for shutdown have varied, from technical to business-related in nature. Of concern, however, is the number of plants that have shutdown due to corrosion-related issues. As best as can be determined, all of the plants that shutdown due to equipment corrosion did not have a mechanism for handling corrosion other than limiting operation to non-corrosive feeds such as hydrocarbons and sewage sludge. While this is a valid and cost reducing way to operate, it requires an accurate knowledge of feed composition and continuous monitoring of the feed to detect any sudden variations in corrosive or salt-forming species. The result of not understanding feed composition and its variations or not incorporating corrosion and salt control methods in the design can lead to plant shutdown and/or extensive litigation, as has unfortunately occurred on more than one occasion.

Table 5. Inactive Full-scale SCWO Plants

Company (Process)	Location	Feed	Dates	Reason for Shutdown
EcoWaste Technologies	Huntsman Chemical, TX	Oxygenated hydro-carbons, amines	1994-2000	Corrosion from out-of spec feed
Foster Wheeler	Pinebluff Arsenal, AR	Smokes and dyes	1998-2002	Liner mechanical problems
Organo (MODAR)	Nittetsu Semiconductor, Japan	Semiconductor manufacture wastes	1998-2002	Stopped operation when Nittetsu was sold
Shinko Pantec (EcoWaste)	Japan	Municipal waste-water sludge	2000-2004	Corrosion
Hanwha Chemical	Huchems DNT/MNT plant, Korea	DNT process waste water	2000-2005	Off-spec feed; heat exchanger corrosion
HydroProcessing	Harlingen, TX	Wastewater sludge	2001-2002	Heat exchanger corrosion
Chematur	Johnson Matthey (JM), Brimsdown, UK	Spent catalyst	2002-2007	Loss of contract by JM for Rh catalyst recovery
Organo (MODAR)	University of Tokyo, Japan	Laboratory organic waste water	2002-2010	Several problems (plan to remodel plant with change in reactor type)
HOO	Southwest France	Food industry waste water	2004-2006	HOO went out of business
Hanwha Chemical	Samnam Petrochemical, Korea	TPA process waste water	2006-2007	Oxygen feed compressor problems

ACTIVE SCWO COMPANIES

Despite the limited number of full-scale SCWO facilities currently in operation, most of the companies that are involved in commercializing SCWO technology today remain very active in this business field. A summary of SCWO technology and recent activity at each of these companies (in alphabetical order) is provided below:

General Atomics (GA) – Headquartered in San Diego, CA, GA has the longest tenure in SCWO of all active SCWO companies (since 1991). They acquired the first SCWO company (MODAR) and its knowledge assets in 1996. GA has typically utilized a vessel type reactor design and has had extensive experience with several different methods for controlling corrosion and salt precipitation/accumulation, such as the use of liners, coatings, feed additives, and mechanical scrapers [9, 10]. This has allowed them to design their systems to handle a wide variety of aggressive feeds such as chemical agent hydrolysates. Most of GA's work has been for government/ military entities but they have also worked with industrial clients as well. GA has recently unveiled a simpler, more cost effective version of SCWO for commercial applications referred to as industrial SCWO (iSCWO) [23]. To date, they have built two full-scale SCWO systems for the US government that are waiting to be installed and commissioned: an 11.4 L/min (3 gpm) system for the Tooele Army Depot for destroying obsolete explosive cartridge components and a 4.5 L/min (1.2 gpm) system for processing pink water (wastewater from explosives manufacturing) in Korea [24]. As part of the Bechtel-Parsons Bluegrass Team, GA is currently fabricating three SCWO systems, each with a capacity of 450 kg/hr (1000 lb/hr), for the US Army's Assembled Chemical Weapons Alternatives program [8, 25]. These units will be installed at the Blue Grass Chemical Agent-Destruction Pilot Plant (BGCAPP) facility being built in Richmond, KY to destroy the stockpile of chemical weapons located there. The SCWO system will process chemical agent and explosive hydrolysates generated during the demilitarization process. In a separate project, GA is constructing a 38 L/min (10 gpm) iSCWO system for the Blue Grass Army Depot for destruction of obsolete conventional munitions.

Hanwha Chemical Corp. – Hanwha is one of the most versatile companies involved with hydrothermal technologies, with its SCWO Business Group having interest in SCWO, NCH, and various hydrothermal synthesis processes such as growth of metal oxides and carbon nanotubes [26]. They have utilized both vessel and tubular reactor types in their systems and have several bench and pilot scale systems on which they have performed testing of various feeds by hydrothermal treatment and research on different types of synthesis reactions [27]. Headquartered in Seoul, South Korea, they have been working with these supercritical water-based technologies since 1994. Most of their customers for SCWO and NCH plants appear to be chemical manufacturing companies, as they have targeted industrial waste treatment applications. To date, they have built two full-scale SCWO plants; a 2000 kg/hr system for dinitrotoluene (DNT) wastewater and a 5500 kg/hr system for terephthalic acid (TPA) wastewater [22]. Both of these plants are no longer operating. They have also built two full-scale NCH plants of 35,000 kg/hr (melamine wastewater) and 20,000 kg/day (TDI residue) capacity, with only the latter being operational at the present time.

Innoveox – Paris-based Innoveox has exclusive global rights to the version of SCWO developed and patented by Cansell's group at CNRS in France. Innoveox is the youngest of the active SCWO companies (starting in 2008), but has built and improved on the developments and progress made by an earlier similar company, HOO (though there is no official connection between the two) [28]. The unique aspect of Innoveox's SCWO design is the use of multiple injection points for oxidant along the length of its tubular reactor (instead of all at once at the beginning like most designs). This is done to control temperature for greatest reaction efficiency and to avoid thermal runaway, and results in a temperature rise down the length of the reactor. Corrosion and salt precipitation are managed by limiting the concentration of key species for these phenomena in the feed. Innoveox's focus and business model is based on providing waste treatment as a service at a customer's site rather than just designing and selling SCWO equipment [29]. In addition to the SCWO system already in operation at Arthez-de-Béarn, Innoveox is building a larger capacity system of 1000 kg/hr for an industrial client that is expected to come on-line in 2012, and has contracts to build three more systems of comparable size [20].

SRI International – The scientific research institute SRI International, located in Menlo Park, CA, developed the AHO version of SCWO. This is the technology that is being utilized in the full-scale facility built for JESCO and currently in operation in Tokyo Japan for destruction of PCBs, as described earlier. While AHO technology is still available, there does not appear to be any recent activity or developments in AHO at SRI International and no further plans for additional full-scale plants at the present time.

SuperCritical Fluids International (SCFI) – Based in Cork, Ireland, SCFI is a relatively new company with a long history. Their SCWO technology began with Eco Waste Technologies (EWT) of Austin, TX, one of the original SCWO commercial companies. The Swedish firm Chematur AB first bought a license for the EWT SCWO process in Europe in 1995 and then bought the worldwide rights to EWT SCWO in 1999. With further development work, Chematur marketed their version of SCWO under the name Aquacritox®. They also developed and named different customized versions of the Aquacritox® process in collaboration with various clients. In 2007, Chematur sold their supercritical fluids division and equipment to SCFI. SCFI has continued to improve on the Chematur SCWO design, though they have consolidated Chematur's many versions of SCWO under the single Aquacritox® brand name for ease of marketing. SCFI utilizes a tubular reactor design and has chosen to focus primarily on sewage sludge and digestate feed applications [30]. While they have a sacrificial mixing pipe configuration that can be used at the entrance and exit to the reactor for dealing with corrosive feeds [31], SCFI prefers to limit applications to feeds that are relatively low in corrosion and salt formation potential. As such, they typically restrict salt levels in the feed to a few percent and do not process feeds with chlorinated materials. SCFI has partnered with Parsons to provide internal engineering support and marketing in North America [32], and with Rockwell Automation to provide control systems and construction support. SCFI has designed four different models of the Aquacritox® process based on nominal feed rate: 600, 2500, 10,000, and 20,000 kg/hr [33]. They are currently building their first commercial system (2500 kg/hr) for the waste treatment and recycling firm Eras Eco in Youghal, Ireland [34]. This system will include the option of power generation from the process effluent heat via a waste heat boiler and turbine.

SuperWater Solutions LLC – This is the latest company that was co-founded by Dr. Michael Modell, whose experiments at MIT in the 1970s formed the basis of SCWO technology and who subsequently founded MODAR. SuperWater Solutions was started in 2006 and is based in Wellington, FL. Its main focus has been on processing non-corrosive wastewater sludge. The SuperWater Solutions SCWO design is similar to that of Modell's previous company, MODEC. It features a tubular reactor system, and utilizes a high velocity flow and mechanical brushes for control/removal of salts/solids accumulation [35]. Since 2007, SuperWater Solutions has worked closely with the city of Orlando, FL, with the city funding development of their system [36]. As a return for this investment, Orlando has a unique deal in which it will receive a royalty of $2.50 for every ton of sludge treated at any future SCWO facility built by SuperWater Solutions for other customers. From 2009-2011, they installed and successfully tested a 4536 kg/day (5 tons/day) SCWO system at one of the city's wastewater treatment facilities. Since that time, the city has continued to lease space to SuperWater Solutions for further development work of their system design. A full-scale 9072 kg/day (10 tons/day) SCWO system is expected to be built for the city in 2013 [37].

NOTABLE PILOT PLANTS

Most of the active SCWO companies have at least one pilot scale SCWO facility and smaller scale systems used for research and demonstration purposes. In addition, there are several non-commercial pilot or demonstration-scale SCWO systems that are worth noting. These systems are owned mostly by groups in academia using them primarily for research and development work. While many research groups formerly active in SCWO have transitioned to other applications of subcritical and supercritical water (e.g., biofuels generation), there are many that are still actively involved with SCWO research. SCWO systems of 20 kg/hr capacity or higher are currently in operation at the University of Valladolid [38] and University of Cádiz [39] in Spain, the University of British Columbia [40] in Canada (used primarily for heat transfer and fouling research), and the Boreskov Institute of Catalysis [41] in Russia (Table 6).

Table 6. Active Non-commercial Large-scale SCWO Pilot Plants

Group	Country	Capacity (kg/hr)	Reactor Type
University of Valladolid	Spain	40	Transpiring Wall
		200	Transpiring Wall and Film Cooled
University of Cádiz	Spain	23	Tubular
University of British Columbia	Canada	120	Tubular
Boreskov Institute of Catalysis	Russia	40 - 60	Tubular

SUMMARY

Commercialization of SCWO technology has been in progress for over three decades since its potential for destruction of aqueous organic wastes was first realized. As of early 2012, there are six commercial firms around the world that are working in SCWO. While this is less than the number of SCWO companies in the past, new companies continue to enter the field as other ones leave. There are two currently operating full-scale SCWO plants in existence along with one plant using the related NCH technology. However, each currently active company has at least one plant either in operation or in the design or construction stages at the present time. There are eight SCWO plants currently in the planning stages with seven of these slated to start operation within the next 1-2 years. Each SCWO company has one or more unique features to their system design (for operation and control of corrosion and salt buildup) and/or business plan, and each one has targeted a specific feed niche. While not without its challenges, SCWO technology commercialization remains an area of great interest and activity.

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