

Ignition of *n*-propanol–air hydrothermal flames during supercritical water oxidation

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Abstract

A novel concept was applied for the in situ study of the ignition of *n*-propanol–air hydrothermal flames during supercritical water oxidation. An innovative experimental arrangement including NASA's supercritical water oxidation facility was used to study the effect of reactor and oxidant (air) temperature on the spontaneous ignition of hydrothermal flames. New data were obtained concerning time- and temperature-resolved profiles of *n*-propanol in hydrothermal flames. Transient temperature profiles were recorded and the sudden increase in the temperature was used to determine the onset of flame ignition. The optimal reactor temperatures for flame ignition (ignition delay ~ 1 s) were determined as 380 °C (at oxidant temperature of 450 °C) and 420 °C (at oxidant temperature of 400 °C) with 20.7 MPa pressure and 1.5 mL/s oxidant (air) flow rate. The influence of buoyancy on ignition is also discussed in terms of Froude number. Time delays associated with the flame ignition at near-critical and supercritical environments as well as possible routes towards their diminution are elaborated. The ignition mechanism of *n*-propanol–air hydrothermal flames together with the ignition maps have been proposed for the first time.

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1. Introduction

Supercritical water (SCW) is an ideal medium for a variety of hydrothermal reactions due to its flexibility in tuning the thermo-physical properties such as density, dielectric constant and specific heat with alterations in temperature (≥ 374 °C) and pressure (22.1 MPa) [1–3]. The enhanced

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solvent properties and high diffusivities at supercritical conditions provide an ideal medium for the reactions to occur in a homogeneous phase thereby eliminating the mass transfer limitations and increasing the reaction rates during oxidation processes [4]. High temperatures attained with the presence of flames in SCW result in near-complete conversion efficiencies with reactions completing in the order of milliseconds [5]. With the added advantages of controlled flames in SCW, recalcitrant molecules can be successfully treated accompanied by high energy recovery [6].

Various organic solvents, e.g. methane, ethane, methanol, heptane and toluene have been investigated for flame ignition in SCW [7]. Several applications (e.g., denaturation of wastes and thermal spallation of rocks) have been suggested by Cabeza et al. [8] and Stathopoulos et al. [9] for hydrothermal flames using isopropanol and ethanol, respectively. Methanol and isopropanol have received widespread attention compared to other organic solvents in hydrothermal flames research due to their heating values and miscibility at room temperature that eliminates the two-phase pumping of the mixture [10]. Schilling and Franck [11] indicated the occurrence of hydrothermal flames during supercritical water oxidation (SCWO) of highly concentrated methane solution (30 mol%).

The current work employs a SCWO test facility designed and developed by NASA at Glenn Research Center for evaluating the potential of SCWO in waste remediation and water reclamation during spaceflight missions or extra-terrestrial habitation. The SCWO test facility was initially used at NASA's Zero Gravity Facility to understand the effect of gravity on the heating rates of the hydrothermal flames using methanol as fuel and air as oxidant [12].

This research investigates the ignition parameters of an aqueous solution comprising of a primary alcohol (i.e., *n*-propanol) that has an auto-ignition temperature (371 °C) close to the critical temperature (374 °C) of water. Air was used as the oxidant for oxidation reactions performed at near-critical and supercritical conditions. All the experiments were carried out under normal gravitational conditions to study the hydrothermal flame characteristics (i.e., the impact of oxidant temperature, reactor temperature, pressure and fuel concentration) along with the ignition map. Our investigations allowed proposing, for the first time, the ignition maps for *n*-propanol–air hydrothermal flames.

2. Experimental

2.1. Materials and SCWO flame reactor

The fuel *n*-propanol (purity: > 99.9 wt%) used in this study was obtained from Sigma-Aldrich (Oakville, ON, Canada). Compressed air (indus-

trial grade, purity: > 99.9 vol%) was used as the oxidant, whereas nitrogen (ultrapure grade, purity: > 99.99 vol%) was used as the inert gas for cleaning the reactor prior to each experiment. The flame reactor was designed by NASA at Glenn Research Centre to investigate the role of gravity upon SCWO of human metabolic wastes generated during space-shuttle missions.

The SCWO flame reactor is described in details elsewhere including its schematics and operational procedures [13]. The main components of the experimental facility included a 480 mL reactor vessel (Hastelloy C276, up to 40.7 MPa), heating elements (1500 W band heater), piston accumulator (up to 34.5 MPa), feed injection system (e.g., air booster pump, gear pump and vacuum pump), sampling systems (gas-liquid separator), thermocouples, pressure gauges, and a LabVIEW-assisted temperature/pressure control system. The ignition events were determined from temperature readings recorded by Inconel-sheathed Type-K thermocouples T_B (base) and T_D (dome) located at 5.1 cm and 12.9 cm from the injection plane, respectively [13]. A high precision metering valve allowed the controlled fine flow of the oxidant and inert gas into the reactor. Three heat exchangers are available to transfer the heat from the product gases and cool them to room temperature. All the reactor tubings were made of stainless steel (SS 316) with pressure resistance of 50 MPa.

2.2. Experimental procedure

The experimental procedure consisted of the following six stages: (1) purging and vacuuming of flow lines, (2) injection of *n*-propanol solution, (3) pressurizing and heating of the reactor and flow lines, (4) oxidant injection and subsequent reaction/ignition, (5) reactor shut down, and (6) collection of products for analysis. The inert gas N_2 was purged through the tubing between the reservoir, reactor, heat exchanger and product collection chamber. The reactor lines were vacuumed to remove any leftover trace components for injecting the feed solution into the reactor.

The oxidant tubing was heated to the desired temperature prior to oxidant injection. A metering orifice valve was used to set the oxidant flow rate which ranged from 0.5 to 3 mL/s. The reactor was heated at 17 °C/min and the system attained steady state conditions within 25 min after the initiation of heating *via* band heaters. As the reactor system (*n*-propanol–water) reached desired near-critical or supercritical conditions, the oxidant was injected into the reactor. Upon introduction of the oxidant and commencement of reactions, the fuel concentration inside the system was depleted due to its rate of consumption. The rise in pressure was compensated by the piston accumulator. During the reaction period, the reactor outlet was closed to ensure that the reactants and products stayed inside the

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